DEVELOPMENT OF WASTE TYRE RUBBER BASED CONCRETE FOR STRUCTURAL APPLICATIONS

Kuruwita Arachchige Don Venuka Lashan

218086R

Degree of Master of Science

Department of Civil Engineering

University of Moratuwa

Sri Lanka

May 2023

DEVELOPMENT OF WASTE TYRE RUBBER BASED CONCRETE FOR STRUCTURAL APPLICATIONS

Kuruwita Arachchige Don Venuka Lashan

218086R

A thesis submitted in partial fulfillment of the requirement for the degree Master of Science in Civil Engineering

Department of Civil Engineering

University of Moratuwa
Sri Lanka

May 2023

DECLARATION

I declare that this is my own work and this thesis does not incorporate without acknowledgement any materials previously submitted for a Degree or Diploma in any other University or Institute of higher learning and to the best of my knowledge and belief it does not contain any material previously published or written by another person except where the acknowledgment is made in the text.

Also, I hereby grant to University of Moratuwa the non-exclusive right to reproduce and distribute my thesis, in whole or in part in print, electronic or other medium. I retain the right to use this content in whole or part in future works (such as articles or books).

Signature: Date: 29.05.2023

The above candidate has carried our research for the Masters under my supervision

Name of Supervisor: Prof. (Mrs.) J.C.P.H. Gamage

Signature of supervisor: Date: 31.05.2023

ABSTRACT

Development of Waste Tyre Rubber Based Concrete for Structural Applications

The drastic expansion in the construction industry has created a scarcity of construction materials. Specifically, the use of natural minerals in concrete production has created sever environmental problems. On the other hand, non-recyclable waste production causes hazardous problems to the environment. Waste rubber tires are one such phenomenon. Hence, the use of waste rubber tires as a partial replacement for aggregate in concrete has been a real concern over the past decade. It is understood that rubberized concrete (RuC) concrete enhances properties of better energy absorption, damping ratio, impact resistance, thermal resistivity, sound resistivity, freeze-thaw resistance, decrease in acid penetrations and chloride penetrations. However, mechanical properties were identified to be poor compared to conventional concrete. Implementation of RuC in structural elements could expand the real benefit of using RuC. Hence, it is crucial to identify and develop the required parameters to improve the mechanical properties of RuC. Investigations were carried out with numerous modifications to the concrete matrix. Despite this, the enhancement in mechanical properties was marginal and inconsistent. The study focused on pretreating the rubber with high reactive graphene oxide (GO) and investigating the micro and macro-level material behavior. The research was conducted in three phases. The optimum mix design was developed, and the concrete properties were investigated in the first phase. Secondly, the material variation of crumb rubber (CR) with respect to GO treatment was investigated. Investigating the optimum CR rubber percentages and their variation with respect to different GO treatment types and identifying the high-strength concrete properties concluded in the third phase. Non-homogenous waste rubber tire aggregate replacement enhances the packing density and thereby improves the mechanical properties. The maximum pretreatment time of 2 hours was identified. The precipitation of GO around CR means to improve the bonding with the cement matrix and a significant strength recovery of 88.18 % resulted. With reference to the three pretreatment methods used, fully air-dried GO-treated CR shows better strength recovery, as a result of the higher percentage of GO precipitation. Yet, 2 g/l was identified to be the optimum GO concentration. With reference to the durability properties, the chloride ion penetration of GO-treated RuC was identified to be very low (100 – 1000 coulombs). However, the water penetration of GO-treated RuC is 35 % greater than the control sample.

Key words: Sustainable construction, Rubberized concrete, Pretreatment, Graphene oxide

ACKNOWLEDGEMENT

The research project investigates the incorporation of waste tire rubber as a partial replacement for mineral aggregates in the concrete matrix. The knowledge I pursued through my undergraduate work and post-graduate studies has been a great influence in completing the project as a success.

There are many individuals to whom I owe this success. Foremost, I would like to gratitude my research supervisor, Prof. (Mrs.) J.C.P.H. Gamage, professor of Civil Engineering and director of the career guidance unit, University of Moratuwa. Without her guidance, none of this would have been possible.

Next, Ceylon Graphene Technologies (Pvt) Ltd. and Siam City Lanka Ltd (INSEE) for providing the required material for the project continuously and arranging the possible lab facilities for the project. Without the industrial corporation, this project would have been a success as this.

I would like to acknowledge the laboratory staff of the Building Material laboratory, Structural Engineering laboratory, and Environmental Engineering laboratory, who provided continuous help in conducting experiments.

Moreover, the progress review panel, which highlighted the shortcomings, and improvements and commented on the research project pushed me to achieve higher goals.

Finally, I would like to thank my parents and family for believing in me and supporting me throughout these years.

LIST OF PUBLICATIONS

International Journals

1. ***V.L. Kuruwita Arachchi***, J.C.P.H. Gamage 2022 'Waste Rubber Tire for Producing Concrete: A Review on Materials, Methods, Application and Challengers' – European Journal of Environmental and Civil Engineering – Under review.
2. ***V.L. Kuruwita Arachchi***, J.C.P.H. Gamage, Kajanan Selveranjan 2023 'Effect of Graphene Oxide Treatment for Crumb Rubber in Producing Rubberized Concrete with Improved Compressive Strength' – Construction and Building Materials – Submitted.

International Conferences

1. ***V.L. Kuruwita Arachchi***, J.C.P.H. Gamage, Kajanan Selveranjan 2022 'Investigation of Replacing Aggregate with Non-homogeneous Waste Tire Rubber in Concrete', presented in MERcon 2022 International Research Conference, Moratuwa, Sri Lanka. https://doi.org/10.1109/MERCon55799.2022.9906163

Table of Contents

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

Abbreviation	Description
GO	graphene oxide
FA	Fine aggregate
CA	Coarse aggregate
RuC	Rubberized concrete
NaOH	Sodium hydroxide
PFA	Pulverized fuel ash
SD	Standard deviation
SEM	Scanning electron microscopy
ITZ	Interfacial transition zone
LWC	Light weight concrete
NC	Normal concrete
HWC	Heavy weight concrete
UV	Ultraviolet
SCA	Silicon coupling agent
CR	Crumb rubber
OH	Hydroxyl groups
LTP	Low-temperature plasma
SP^2	The mix of one S and two P atomic orbitals
XRD	X-ray powder diffraction
FTIR	Fourier transformed infrared spectroscopy
XPS	X-ray photoelectron spectroscopic
CR-GO	Treated crumb rubber with graphene oxide
IC	Ion chemometric
PPM	Parts per million
OPC	Ordinary Portland cement

LIST OF APPENDICES

CHAPTER 1 INTRODUCTION

1.1 Research Background

The global expansion of the construction industry in the past decade made concrete the most widely used construction material. As a result of that, the constituents required in making concrete were identified to be in extreme use. Most importantly, the natural materials used in concrete identified to be scarce and cause sever environmental problems with the mass-scale use of it. Further, river sand was identified to be the second most used natural resource after water as a result of the expeditious use of concrete [1]. Hence the industry has been diverging from using natural resources and investigating replacements for concrete constituents.

In the last two decades, the construction industry has focused on enhancing sustainable construction in numerous ways. One of the prominent investigations is to replace concrete aggregate with waste materials. Researchers investigate using recycled plastic, glass, construction demolishers, waste tire rubber, natural fibers, etc. With the evolvement of the investigations, it is found that some of the concrete properties were enhanced while several concrete properties were diminished with respect to each replacement. However, the development of research studies identified waste tire rubber as an outstanding candidate in this regard.

The concrete properties such as ductility, energy dissipation, sound absorption, damping ratio, abrasion resistance, freeze-thaw effect, and thermal resistance are enhanced [2]–[4] with the rubber aggregate replacement. However, poor mechanical properties were observed in RuC, such as diminished compressive strength [5]–[7], tensile strength [8], [9], and flexural strength [9], [10]. Moreover, higher shrinkage [11]–[13], porosity [14], [15], and water absorption [16], [17] were identified to be negative outcomes. As a result of diminished mechanical properties, RuC has not been utilized in structural elements. Yet non-structural applications similar to pavement concrete, roadside barriers, running tracks, bunkers, shooting ranges, and lightweight concrete are constructed using RuC [18]–[21].

The poor mechanical behavior of RuC is caused due to high roughness of rubber particles, a high percentage of voids, poor boding between rubber aggregate and cement, the hydrophobic nature of rubber, non-uniform dispersion of rubber [14], [22]–[24]. Hence, the aforementioned reasons should be addressed by applying modifications to the concrete matrix. However, greeting all the possible details of RuC is a wide research area and not efficient.

The study focused on investigating the dominant cause of poor mechanical properties, the poor bonding behavior of rubber aggregate and cement paste. Even though there are numerous studies investigating to overcome the issue, there is no specific explanation or procedure. Yet, optimized concrete matrix with modification to rubber surface identified to be viable. Hence, the study investigated identifying the optimized concrete matrix and a pretreatment method to enhance the rubber-cement bonding, which could lead to better mechanical properties of RuC.

1.2 Objectives

The objective of the study is to develop an optimized concrete mix design with graphene oxide-treated crumb rubber which is sustainable and enhanced with mechanical properties for structural concrete elements.

- To conduct a detailed literature review to identify the current status of knowledge on Rubberized concrete.
- To identify the variation of mechanical properties of concrete mix with replacement of aggregate by non-homogenous recycled tire rubber.
- To investigate the short-term and long-term properties of developed rubberized concrete mix.
- To provide recommendations for practical applications of rubberized concrete in structural elements.

1.3 Methodology

The research methodology was comprehensively divided into three phases as represented in Figure 1.1. The study was initiated with an extensive literature review to understand the major drawbacks of RuC and the possible modification for those. After a comparison was made between homogenous and non-homogenous rubber aggregate replacement to identify the better performance in mechanical properties. Pretreatment was essential to improve the concrete properties, hence the martial properties of rubber aggregate with suitable pretreatment methods were investigated at micro and macro levels. Afterward, the best-suited pre-treatment technique was selected to further develop the rubber-based concrete mix. Finally, the mix with optimum performance will be selected and short-term and long-term properties will experimentally be determined.

1.4 Research significance

The poor mechanical properties have been identified as the major drawback of RuC in the application process. Hence, research investigations have taken place to enhance mechanical properties in numerous ways. However, the investigations were found to be less effective, sustainable, or consistent.

The major drawback of rubberized concrete is the weak interface between rubber and the cement phase. Hence, a detailed literature review was conducted to identify the possible materials or techniques that are capable of enhancing the interface between rubber aggregate and cement paste in concrete. Among those materials, graphene oxide (GO), containing moieties with high percentage oxygen (hydroxyl, carboxyl, and epoxy, etc.), outstanding mechanical, electrical, thermal, and chemical properties, high reactivity and strong adhesion with polymer membranes being prominent of candidates to enhance the concrete properties.

Thereby, the research study was focused on enhancing the mechanical properties of RuC by incorporating GO as a pretreatment to the crumb rubber involves in making concrete matrix. Hence, no such studies of using GO as a pretreatment to crumb rubber in developing RuC as per authors' knowledge to date.

1.5 Arrangement of the thesis

In this thesis, the research project has been conferred by nine chapters. The **first chapter** represents the research background, objectives, and overall methodology followed.

In the **second chapter**, the literature review on waste rubber tires, recycling, and properties was reviewed. In addition, the RuC production, mix proportions, and concrete properties were discussed comprehensively.

The investigation on replacing non-homogenous rubber aggregates was discussed in the **third chapter**. In the **fourth chapter**, a review was conducted on pretreatment methods and the significance of using graphene oxide.

The fifth chapter investigates the possible pretreatment methods of GO to CR and changes that occurred to the material chemically and physically.

The sixth, seventh, and eighth chapters discuss the mechanical properties, bonding structure, morphology, and durability properties of GO-treated RuC respectively. By the **ninth chapter**, the thesis is concluded by representing conclusions and recommendation of the research project.

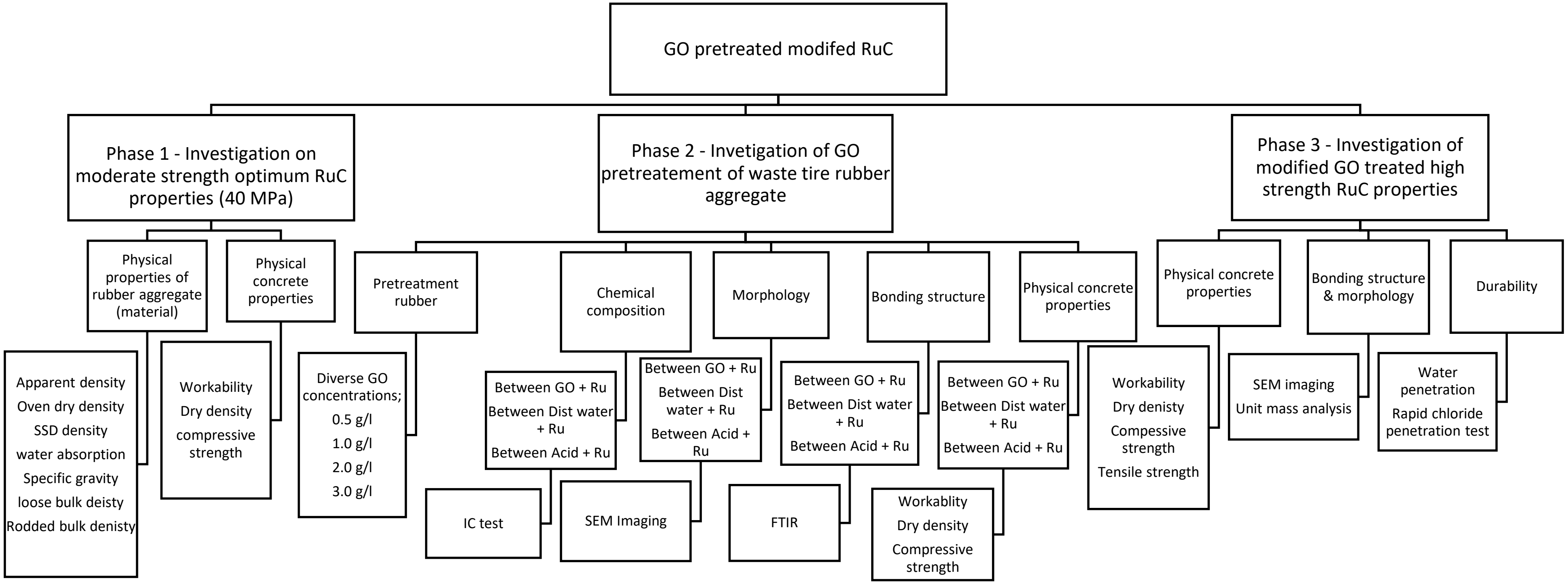


Figure 1.1. Overview of the test series

CHAPTER 2 LITERATURE REVIEW

2.1 Introduction

The development of innovative construction materials emerges as a main field in the construction industry. The concept of sustainable construction focusses on environmentally friendly construction material. Waste rubber is identified as one such innovative and sustainable material in the construction industry. With the rapid expansion of the transport industry and rubber-based industries, a mountainous amount of rubber waste is produced. The main source of rubber waste is tire waste. Raffoul et al. [25] noted that the majority of waste rubber from waste tires and proportional to tire production in the world yearly. The yearly tire production of 2017 is more than 2.9 billion and the International Rubber Group of Companies forecasts 2.8% growth compounded yearly from 2017 to 2025 [26]. The heap amount of rubber waste causes environmental hazards with a non-biodegradable property of rubber waste. As a result of the massive negative impact on the environment, reusing and recycling rubber waste was considered without stockpiling. Forrest [26] mentioned that 30% of rubber waste was stockpiled while 15% was recycled, 23% reused and 60% was recovered as energy. However, U.S. Tire Manufacturers Association and ETRMA [27], [28] concluded that only 6% to 8% of scrap tire rubber waste is used for civil engineering purposes in USA and EU countries while Australia used only 0.4%.

Expansion in the construction industry prevailed on focusing on environmentally friendly construction materials. As a result, the industry is keen on finding innovative construction materials to replace non-ecofriendly construction materials. Concrete is the most used construction material in the world because of the extensive expansion of the construction industry strained concrete to the extreme use at the present day. The aggregate used in concrete such as river sand and manufactured sand causes damage to the environment as a result of excessive use. River sand is identified as the second most used raw material on earth with 80% of 50 billion of tons material mined every year. The continuous demand for river sand caused severe damage to the environment. Excessive mining of river sand caused, intrusion of sea water into the river, the collapse of riverbank and loss of riparian lands, etc. Manufacture sand (M Sand) is identified as a major replacement for river sand but exploiting M-sand makes ecological imbalance causing environmental problems. Therefore, the identification of an aggregate that is eco-friendly while enhancing the mechanical and durability properties of concrete with the sustainable concept is a timely need. Waste rubber was identified as a pioneer in the list to replace as fine aggregate (FA), coarse aggregate (CA), and binder in the concrete mix. The studies [29], [30] revealed that incorporating crumb rubber into cementitious composite can reduce CO2 emission.

For the past couple of decades, studies were conducted to identify the properties of rubberized concrete (RuC) in different aspects. As a result of the evaluation of RuC, research studies have been conducted to identify the properties of RuC as lightweight concrete, resistive concrete, and structural concrete. Hence RuC shows a 14-28% of reduction in the unit weight of concrete by replacing 10-30% of FA and CA, RuC is advantageous as lightweight concrete [31]. Studies revealed that mechanical properties of RuC have a negative effect where the reduction in compressive strength and workability with increment in water absorption and shrinkage. However, literature shows that enhancing concrete properties such as ductility, energy dissipation, flexural strength, and sound absorption are pros points on RuC [2], [3], [32]. An optimization of RuC was discussed by Raffoul et al. [33] concluded that 100% of rubber replacement shows a 92% reduction in strength. Thomas et al. [34] revealed that better resistance to water absorption and carbonation can be achieved with 12.5% of FA replacement. Senin et al. [35] concluded that exceeding 20% rubber replacement cause an adverse effect on desirable concrete properties. However, literature shows that RuC can be applied in structural elements with required moderation [19], [31]. The enhancement in the damping ratio of RuC expands the use of RuC concrete in the industrial floor, road pavements, retaining structures, bridge sidewalks, and decks [18]–[20], [36]. Liu et al. [21] concluded that RuC has a lower dynamic increase factor than conventional concrete. As RuC exhibits 10 % of rubber incorporation to enhance the energy absorption capacity. As a result of enhanced behavior in energy absorption, RuC is implemented in running tracks and roadside barriers [37]. Ibrahim et al. [38] concluded that abrasion resistance increases as the rubber content increases. Fine rubber aggregate, possesses higher abrasion resistance than coarse rubber aggregate. Such resistance to abrasion in RuC can implement in concrete highways, industrial floors, pavements, hydraulic structures, and other surfaces subjected to abrasive forces [16]. Richardson et al. [39] concluded that the most efficient size of CR is 0.5 mm which accompanies the best freeze-thaw protection in RuC. Therefore, constructions in cold climate conditions can be optimized by using RuC as a result of the positive freeze-thaw effect [40]. Thermal resistivity is identified as an enhanced property in RuC with an increment of 29.4%. In contrast, thermal diffusivity and thermal effusivity were reduced by 65.1% and 37.6% respectively [41]. Therefore, in application in which high thermal resistance and acoustic resistance is needed, RuC can be used [42].

This study aims to identify the evaluation of RuC over the past couple of decades. The initiative of using rubber as an aggregate in concrete to provide a balance between the environment and construction is upheld in the study. The performances of RuC in different conditions and the behavioral pattern is presented in this study. Providing a fundamental background of rubber as a waste material and as a construction material is discussed in the study. As well as to reuse of rubber environmentally wise to reduce environmental pollution and its impact on the construction industry is critical to discuss. Mechanical behavior, dynamic properties, and durability of RuC in different conditions are presented in this study. However, the paper presented the moderations applied to RuC to enhance the performance in different conditions. This review also aims to provide compressive insight into a range of applications of RuC used in industry and the impact on the betterment of the sustainable environment.

2.2 Waste tire rubber source

The studies revealed that a total of 300 million tires are disposed of in the USA with an estimation that each person discards one car tire per year [43]–[45]. The disposal of rubber tires in landfill caused damage to the environment, hence tires restoring to the surface with time making a hazardous condition to the environment. This cause not only environmental problems but also damages to public health and aesthetic value [46]. To minimize the damage caused by the tires, several attempts were taken nevertheless using waste rubber tires as a construction material can uplift the concept of sustainability in the construction industry.

Tires can be categorized into two: automobile tires and truck tires. It is important to identify the source of waste tire hence the shape, weight, size, and importantly the ratio of the components of the base mixture is different [47]. On average 70-80% of tire contains highly durable vulcanized rubber which cannot be easily recycled [48]. Siddika et al. [49] mentioned that any type of tire provides an approximate amount of

14% to 55% rubber depending upon the actual composition and thread and sidewall parts of the tire contain most of the rubber of the extraction. Figure 2.1 represents the raw materials of rubber tires.

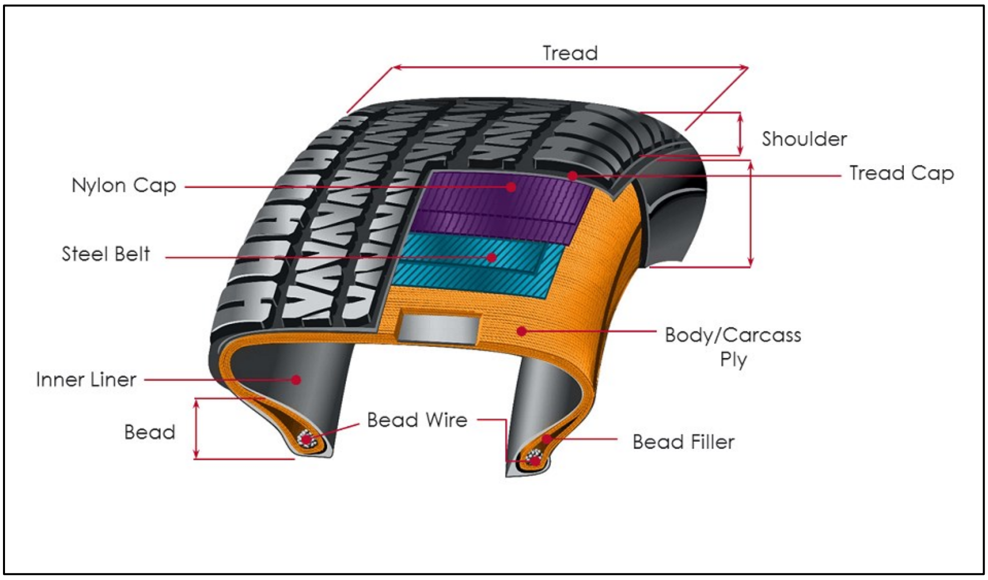


Figure 2.1: Raw material in rubber tire [50]

The composition of tire rubber is critical in the rubber extraction process. Natural rubber, synthetic rubber, carbon black, steel, ash, fabric, textile, and fillers are compositions consisting of different proportions. It is important to show in the literature that the amount of rubber and other compositions can be extracted from a tire. Table 2.1 revealed the percentages of composition in tire rubber.

Table 2.1. Composition percentage in tire rubber

Composition	Percentages %							
References	[51]		[52]	[53]		[54]	[42]	[55]
Type of tire	Car	Truck	Car	Car	Truck	Not specified	Not specified	Not specified
Natural rubber	41-48	41-45	21-42	14	27	-	23.1	-
Synthetic rubber	-	-	40-55	27	14	-	17.9	-
Carbon black	22-28	20-28	30-38	28	28	20-25	28	29

Streel	13-16	20-27	-	14-15	14-15	-	14.5	-
Ash	-	-	3-7	-	-	-	5.1	5
Plasticizer	-	-	-	-	-	-	-	10
Polymer	-	-	-	-	-	40-55	-	50
Other (fabric, filler, textile)	4-6	0-10	-	-	-	20-40	16.5	-

2.3 The general process of rubber waste recycling from waste tire

Waste tire management is critical in social, economic, and environmental aspects. There are several practices used in managing waste rubber tires: recycling, reusing, recovery as energy and stockpiling are some of the methods. The estimated percentages of waste tire managing methods [26] are provided in Table 2.2.

Table 2.2. Estimated percentages of waste tire managing method

Method	**Estimated amount**
Recycle	15%
Reuse	23%
Recovery	60%
Stockpiled	30%

The high number of stockpiling in lands consumes lands with no benefits. The high percentage %), hence the damping cost extensive areas and creates hazardous environmental problems [56]. Deriving energy from the waste tire is a major concern in the industry 43% of the scrap tire produced in the USA is derived as fuel [57]. Germany is the highest tire manufacturer in Europe union treats scrap tires as the first country with the highest scrap treatment for energetic recovery [58]. Rather than creating a problem, the issue of the waste tire can be transformed into an opportunity. New tires after some periods become part-worn tires. Those tires are reusable after regrooving or after reconditioning (retreading). Figure 2.2 illustrated the material recovery from waste tires in the EU.

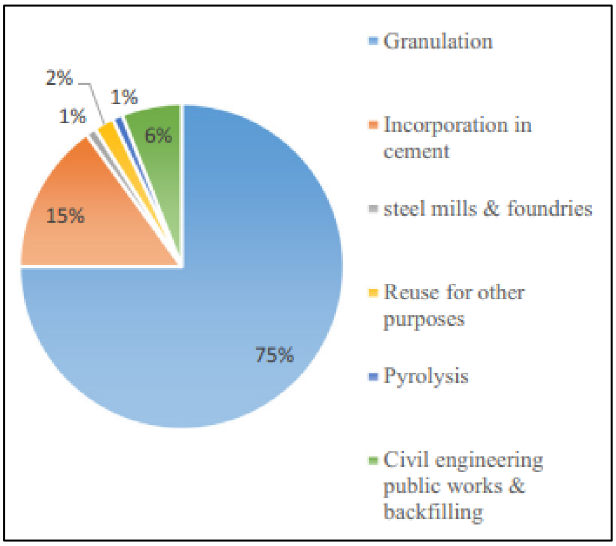


Figure 2.2: Material recovery from waste tire in EU [59]

The recycling process that scrap tires should undergo is illustrated in the following.

Collection of waste tires: As the first step of recycling, collection of waste tires should be done by the means of individual businesses and local authorities.

Shredding: Starts with reducing the rubber tire size by cutting them into small pieces for easy handling and shredding the tires by using a rotating shaft. At the end of this stage, 2-inch length rubber can be found, which can derive for fuel. Shredding can be done in two ways. In a mechanical system, tires are scraps into smaller chips at ambient temperature. In a cryogenic system, tires are turned into tiny particles by freezing the rubber with the help of liquid nitrogen.

Steel liberation: This step includes the elimination and separation of tire wire from rubber. Also, this includes the fiber and screening as well. The removed steel wire can be used in manufacturing new wheels or as a material in concrete to improve strength [60].

To produce crumb rubber, the grinding process is specified. Rubber must pass through the grinding process in a dry environment including the environment temperature. There are four types of grinding specified by Elshazly et al. [61].

In the first type, griding at surrounding temperature using mills. The second type is to grind at the surrounding temperature in wet conditions to induce water to minimize the high temperature. The third type is to grind at a high temperature of 130°C to produce grains of 1 mm to 6 mm. The fourth type is to grind after freezing rubber in a glass case by using an impact-type mill.

2.4 Tire Rubber Aggregate Properties

Studies have been done on tire rubber aggregate to place in the concrete mix in different formats. Chipped rubber, crumb rubber, and powdered rubber are the three types of rubber forms identified by using RuC. Chipped rubber is mostly replaced as the coarse aggregate of the concrete mix as higher dimensions possess in chipped rubber. Due to its high irregularity character, crumb rubber was replaced as the fine aggregate of concrete mix with lesser dimensions than the chipped rubber. Powdered rubber is extremely small in dimensions, less than 1 mm, as collected the powdered rubber in the crunch process of the plant as the waste rubber. This form of rubber can be used as filler in concrete due to its small dimensions possessed [62]. The physical properties of a rubber tire in the literature are summarized in Table 2.3.

Table 2.3. Physical properties of rubber tire

Reference	Form of rubber	Size (cm)	Water absorption	Specific gravity	Density (kg/m3)	Flakiness index	Tension strength MPa
[62]	Chipped	2.5-3.0	-	-	-	-	-
	Crumb	0.3-1	-	-	-	-	-
	Powdered	<0.1	-	-	-	-	-
[63]	Crumb rubber	-	-	-	640-720	-	-
[61]	Crumb rubber	1.3-7.6	-	-	-	-	-
		0.0425-0.475	-	-		-	-
	Powdered rubber	0.0075-0.0475	-	-	-	-	-
[54]	Crumb rubber	1-2	0.80-1.30	1.10	480	10.4-17.5	-
		0.5-1	5.30-8.90	1.10	450	6.6-8.3	-

	Powdered rubber	0-0.5	-	-	400-460	N/A	-
[64]	Crumb rubber	1-2	0.80-1.30	1.10	0.48	-	-
	Crumb rubber	0.5-1	5.30-8.90	1.10	0.45	-	-
	Powdered rubber	0-0.5	-	-	0.40-0.46	-	-
[65]	Well graded Crumb rubber	0.015-0.236	-	0.83	0.530	-	-
[66]	Crumb rubber	0.2-0.6	0.65	1.12	0.489	-	-
	Crumb rubber to replace coarse aggregate	0.1-0.3	85%	0.54	-	-	28.1
	Crumb rubber to replace fine aggregate	0.5-1			-	-	

The composition contains in tire rubber influences the concrete mix chemically [67]. The general composition includes natural and synthetic rubber, polymer, carbon black, zinc, silicon, sulfur, magnesium, aluminum, nitrogen, hydrogen, and organic compounds. Percentages of chemical composition in rubber tire is summarized in Table 2.4

Table 2.4. Percentages of chemical composition in rubber tire

Composition %	Reference						
	[54]	[68]	[69]	[22]	[70]	[23]	[71]
Polymer	40-55	40-55	38.3	45	-	-	-

Ash		3-7	5.43	-	-	-	-
Carbon black	20-25	30-38	31.3	40	91.5	87.51	81.2-85.2
Zinc	-	-	-	-	3.5	1.76	-
Silicon	-	-	-	-	-	0.20	-
Sulfur	-	0-5	3.23	-	1.2	1.08	1.52-1.64
Magnesium	-	-	-	-	-	0.14	-
Oxygen	-				3.3	9.23	1.72-2.07
Aluminum	-	-	-	-	-	0.08	-
Nitrogen	-	-	-	-	-	-	0.31-0.47
Hydrogen	-	-	-	-	-	-	7.22-7.42
Other (fillers, softeners, organic compounds)	20-40	-	-	15	-	-	-

2.5 Pretreatment of Tire Rubber

One of the major drawbacks of RuC is the lack of compressive strength. This has been explained as a result of a weak bond between the rubber-cement interface. To improve the interface bonding, absorb a certain amount of water, and avoid the floatation of rubber particles in the concrete matrix, several pretreatment methods were considered. A summary of different pretreatment methods on the rubber is represented in Table 2.5.

Table 2.5. Pretreatment methods for rubber aggregate

Reference (taken from an experimental investigation)	Pretreatment type
[72]–[75]	Washing with water
[76]	Polyvinyl alcohol

[77]–[81]	NaOH
[82]	Ca (OH)$_2$
[83]	silane coupling agents
[84]	Organic Sulphur compounds
[85]	Acid
[86]	Partial oxidation of rubber surface
[87]	Expose to UV radiation
[88]	Pre-coating with cement
[89]	Pre-coating with mortar
[90]	Pre-coating with silica fume
[91]	Using limestone
[80]	Pre-coating with sand

Studies [78], [88], [89], [91], [92] revealed that the strength of RuC increased by 3-40% as a success in rubber pretreatments. However, these measurements are not conclusive hence, no comparison was done between pre-treated rubber and received rubber [73], [74]. However, pretreatments are identified costly and time-consuming, while no conclusive results to identify the enhancement of concrete properties.

Most of the studies were entertained on pretreating with water and NaOH. Treatment with sodium hydroxide solution (NaOH) explained to soaked rubber aggregate in a 10% NaOH solution, whilst the process allows rubber particles to ingest a particular water amount. After the treatment, the rubber aggregate needs to drain, then water soaking and rinsing a minimum of three times in clean water is required to neutralize the pH of the material [37]. Pham et al. [75] mentioned that to enhance the mechanical properties of RuC by improving the bonding between rubber-cement surfaces, washing of rubber aggregate was done and soaked for 24 h to reduce the rubber hydrophobicity. Furthermore, the study revealed that soaking in water was chosen over NaOH treatment, because of the simplicity of washing with water. Thus, the study [81] concluded that specimens pre-treated with NaOH resulted in comparatively high compressive strength than those specimens pre-treated with water. The study by Chaou et al. [93] concluded that a 13% of strength increment was observed with rubber particles treated with NaOH and Guo et al. [94] mentioned that a 22.9% strength increment was experienced using only NaOH while a 14.2% increase experienced using the solution mixed with blended cement paste coating.

2.6 Mix Proportions

Previous studies elaborate on the fact that achieving suitable mix proportions of RuC is difficult. Most of the literature conducted studies on considering low percentages of rubber aggregate up to 25% [77][95][96][97][98]. However, the studies done by previous researchers [99][100][54] mentioned that practicing small percentages of rubber aggregate hinders the actual benefit of using waste tire rubber. The study by Raffoul et al. [54] elaborated that not using waste rubber on large scale is a waste, whilst waste rubber can contribute to having a positive environmental impact as well a contribution to sustainable construction.

The difficulty of achieving suitable concrete mixes has been idolized with the concrete properties of RuC. The high percentage of voids and porous nature of RuC were identified as the main reasons for the poor behavior of fresh performance of RuC [101]. This was explained by the low specific gravity possessed with rubber compared to mineral aggregate and cement, the deficient mix proportioning, hydrophobicity of rubber, uneven shapes, rough texture, contamination interlink among the particles, enhanced friction with cement paste, consolidation, handling or casting can create floating of rubber at the top upon vibration [5], [102], [103][72]. Fresh concrete properties such as workability are defined by the ease of mixing, placing, and consolidating while maintaining adequate concrete homogeneity depending on the overall stability (i.e., segregation and bleeding) which is proportionate to fresh RuC mix [104].

The poor compressive strength of RuC has been explained by the high porosity of the composite, the relatively high Poisson’s ratio of the rubber aggregates, and the poor bonding behavior of the rubber-cement matrix [89][66]. The influence of rubber percentage, size, shape, and properties with better constituent parameters and proportions was identified as the parameters which lower the compressive strength of RuC [95][105][102][10][106]. Therefore, the significance of achieving an improved packing density of the concrete matrix, its rheology, durability, and mechanical properties are identified to be highlighted in the literature [107]. Therefore, the investigation of achieving an optimized RuC mix with high content of rubber is a timely need. The study [54] did an experimental study to examine the parameters influencing the performance of RuC under an optimized RuC mix. Table 2.6 illustrates the mixed proportions used in the literature.

Table 2.6. Reference mix proportions

Reference	Rubber percentage	Cement (kg/m^3)	Water	Sand (kg/m^3)	Aggregate (kg/m^3)	Rubber (kg/m^3)	Pulverized Fuel ash (PFA) (kg/m^3)	Silica fume (kg/m^3)	Plasticiser (l/m^3)	Superplasticiser (l/m^3)
[54]	Optimized mix	340	150(l/m^3)	-	0-5mm = 820		42.5	42.5	2.5	5.1
					5-10mm = 364					
					10-20mm = 637					
[37]	0%	426	213(kg/m^3)	843	<4mm= 130 4-7mm= 750	-	-	-	-	-
	15%			717	<4mm= 111 4-7mm= 638	5-7mm= 49	-	-	-	-

						1-5mm= 63				
	30%			590	<4mm= 91 4-7mm= 525	5-7mm 98 1-5mm= 127	-	-	-	-
[108]	0%	426	205(kg/m^3)	843	≤5mm = 130 ≤7mm = 306 ≤10mm = 444	-	-	-	-	-
	15%			717	≤5mm = 111 ≤7mm = 260 ≤10mm = 377	1-5mm= 45 5-10mm= 58	-	-	-	-

	30%			500	≤5mm = 91 ≤7mm = 214 ≤10mm = 311	1-5mm= 89 5-10mm= 116	-	-	-	-
[62]	5%	38.71	16.43		Gravel – 87.49	2.31	-	-	-	-
	10%			41.57		4.62	-	-	-	-
	15%			39.26		6.93	-	-	-	-
	20%			36.95		9.24	-	-	-	-

Based on the literature, insufficient studies on RuC performances and the impact created by the incorporation of rubber on numerous concrete properties have hindered the development and application of RuC in structural elements. Mainly RuC is applied on non-structural applications such as shooting ranges, road barriers [109], thin overlays [55], road pavements [106], [110], concrete panels [111], and as thermal and acoustic insulation [42], [112]. Further, RuC is used as lightweight concrete with reduced weight but improved ductility, vibration resistive, impact, and cyclic loads [112]–[115]. Therefore, the identification of significant RuC mix proportion can expand the value in terms of structural and environmental perspectives.

2.7 Fresh Concrete Properties of RuC

2.7.1 Density and Workability

Rubber aggregate contains a relatively low density compared to mineral aggregates, therefore replacing it with rubber aggregate reduces the density of concrete. Voids created in the concrete matrix as a result of very low adhesion between rubber-cement matrices can be explained as the result of low density [116] Further, voids are created to increase the porosity resulting in low unit weight [14][15].

The literature elaborates that density reduced with the rubber percentage in the concrete mixture, but no specific pattern was identified by the studies for respective rubber percentages. Noaman et al. [117] concluded that a 3% density reduction resulted in 15% sand incorporation proportionate to coarse rubber while, a 6.9% density reduction was observed with 50% of sand replacement by fine rubber [118] and a 38% reduction in density was concluded with 10% replacement of rubber aggregate [119]. Further, studies revealed that larger density reduction was experienced by replacing rubber aggregate with smaller particles. Replacing 50% of fine and coarse rubber aggregates resulted in a variation of up to 75 % dry density reduction compared to the range of 10-30% reduction for fine rubber replacement only [120]–[123][97][124]. The study carried out by using tire chips as partial replacement of coarse aggregate, using crumb rubber as partial replacement of fine aggregate, and using a combination of tire chips and crumb rubber as partial replacement showed 45%, 34%, and 33% reduction in density, respectively [125]. Thereby combination of non-homogenous replacement of CR aggregate shows improved properties in density. However, the study shows a contradiction with the literature that replacing fine particles resulted in a lesser reduction in density.

Workability was identified as the main concerned fresh property of RuC as lower workability compared to conventional concrete. Literature concluded that the slump value of RuC diminishes proportionately to the percentage of rubber [119][126][127]. The variation in slump values is represented in Table 2.7.

Table 2.7. Slump reduction with respect to the rubber content replacement

Reference	Rubber replacement	Slump reduction
[10]	20%-100%	19%-93%
[123]	50% of the total aggregate	90-100%
[128]	40% of the total aggregate	90-100%
[97]	15% of coarse aggregate	60%
[54]	0-10% of fine aggregate	0%
	45% total aggregate	30%

The studies have proven that there is a significant change in slump value as the form of the rubber varies. The study by [97] elaborated with fine rubber replacement up to 15% shows an increment in slump value, coarse rubber shows the maximum decrease in a slump at 15% replacement while the combination of fine and coarse rubber replacement shows a small deviation up to 25% replacement. Similarly, a study [38] concluded a maximum of 58% and 75% of reduction in a slump for 30% of fine and coarse aggregate replacement respectively. Figure 2.3 illustrates the variation of the slump with respect to rubber percentage and rubber form.

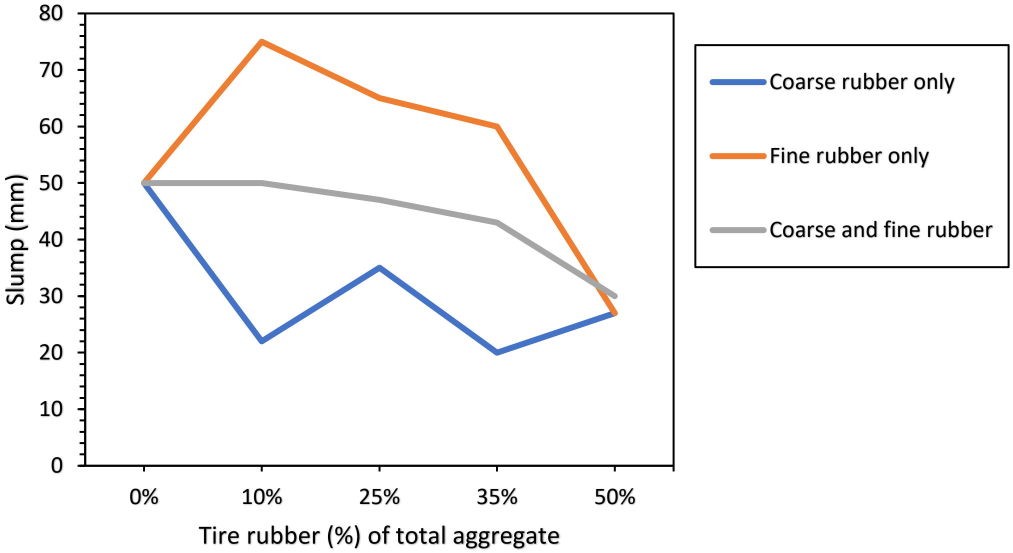


Figure 2.3. Variation in slump respect to rubber content (%)

The studies [38][97] concluded that replacing with fine rubber aggregate shows less tendency in decreasing slump while it can increase the slump by 15%. The reduction in workability has been identified mainly because of the greater water absorption capacity of rubber particles [129]. Further, a study [130] mentioned that the workability is directly proportionate to the specific surface area of concrete constituents, hence, coarser rubber aggregate shows a high possibility to have lower workability than fine rubber particles. This condition can be explained by the high roughness of rubber aggregate possess, the friction between the particles and concrete increases as it requires higher energy to flow [131]. Therefore, the requirement of water in RuC is comparatively higher than conventional concrete's water requirement. To improve the workability of the mixture, several experiments were done, and recommendations were decided. Using freeze ground rubber other than ground rubber can avoid the high roughness of ground rubber, rubber to use as a partial replacement to avoid similar rubber grain sizes [130][129][105][56], and use suitable superplasticizers and sufficient water to limit segregation and bleeding, to hydrate the cement [54]

2.7.2 Rheological Properties

Rheological properties of concrete mix are defined by the flow curves, dynamic yield stress, plastic viscosity, shear thinning behavior, and thixotropic behavior [132]. The defined properties have a great impact on the water percentage, aggregate properties, gradation of aggregate, mixing time, mixing system, temperature, shape, and texture of aggregate [49]. The investigations conducted on the variation of shear stress with respect to shear rate curves of mortar mixed rubber fiber were studied, as the highest shear stress resulted in a maximum rubber fiber percentage of 25% which shows an increment with rubber content [132], further studies incorporating various industrial fibers shows the trend of increase in shear stress with the incorporation of higher fiber content [133][134]. This was explained by the mechanical interlocking of the fiber with surrounding particles and amplified interlocking with higher fiber content. Dynamic yield stress is defined as the minimum stress required to assist the flow of cement-based mixes. Use of 0%, 10%, 15%, 20% and 25% rubber fiber mortar mixes, the dynamic yield stress resulted in 21.61 N/mm^2, 29.27 N/mm^2, 46.41 N/mm^2, 73.28 N/mm^2, and 55.91 N/mm$^{2\ \mathrm{With}}$. The pattern was to increase dynamic yield stress with the increment of rubber content as a result of a compact concrete matrix possess with incorporated rubber fiber as it provides ease of movement due to the anoint nature of rubber fiber [132][135]. The viscosity of rubber-contained mixtures was observed to decrease with higher rubber content with a change in the shear rate. This was explained by the breakdown of the inter-particle structures and the aligning of fibers along the flow direction which resist the flow by rubber particles [136][137][134]. Further, a study [138] concluded that replacing fine aggregate with coarse aggregate will increase the viscosity but a workable concrete matrix is only prepared with a high shear rate mixing system. To minimize this negative effect of RuC, the addition of fly ash was recommended.

The resistance within the flowing mortar quantified the plastic viscosity which may attribute to the forces among the particles, size distribution, percentage concentration, and particle shape [139][118]. The literature shows that as the rubber content increases the plastic viscosity increases, as the particle shape influences the flow behavior. Further, the incorporation of rubber constituents reduces the spatial distance of particles and causes overlapping, which creates a network structure that increased the resistance to flow [118][139][24]. Conversely, the study [132] concluded that the incorporation of rubber fiber can be advantageous as the increase in plastic viscosity may improve the uniform fiber dispersion [24] and may provide dynamic stability to the mortar mix [140]. In the study by [132] for all mortar mixes, the rheological behavior has been observed less than zero, which represents a shear thinning behavior of mixes. Further, study revealed that an increase in shear thinning intensity up to 15% of rubber fiber and decrease as the rubber content increased. This can be explained because of increment in non-hydrodynamic interaction [141]. However, further clarifications are required to identify the behavior change.

2.7.3 Shrinkage properties

The literature revealed the factors of early-age tensile strength, high deformation capacity, and resistance against fracture results series of minor cracks exposed concrete surfaces. Therefore, increasing the ductility capacity of concrete is identified as the task to improve the concrete cracking resistance. Rubber particles with low elastic modulus, and high flexibility and which can undergo high deformation have been identified as the construction material to use in the concrete mix [11][12]. However, the introduction of rubber aggregate to concrete has both positive and negative impacts. The reduction in tensile strength can experience with rubber indulgence while the concrete ductility increases. The degree of effect bank on the properties and rubber incorporated percentage [13].

Shrinkage cracking is not only depending on the concrete constituents but also on the concrete element size and geometry [142]. However, there is no test developed for measuring the toughness of RuC, if toughness is to measure energy measuring based, the maximum rubber content is to be identified by maximizing the area under the load-deflection curve [143]. It was concluded that higher toughness resulted in higher rubber content. In [13], the plastic shrinkage gradually increases after exceeding the 20–25% replacement level as represented in Figure 2.4. By contrast, previous studies reveal that the addition of RA can increase the drying shrinkage in concrete. The study [144] concluded that shrinkage may increase by 43% with 15% of fine aggregate replacement. Moreover, rubber aggregate has a huge impact on concrete shrinkage, but rubber aggregate does not provide a noticeable effect on shrinkage after a full drying shrinkage point. The study by Yung et al. [145] concluded that powdered rubber increases the shrinkage length by about 35% to 95% with 5% to 20% rubber replacement.

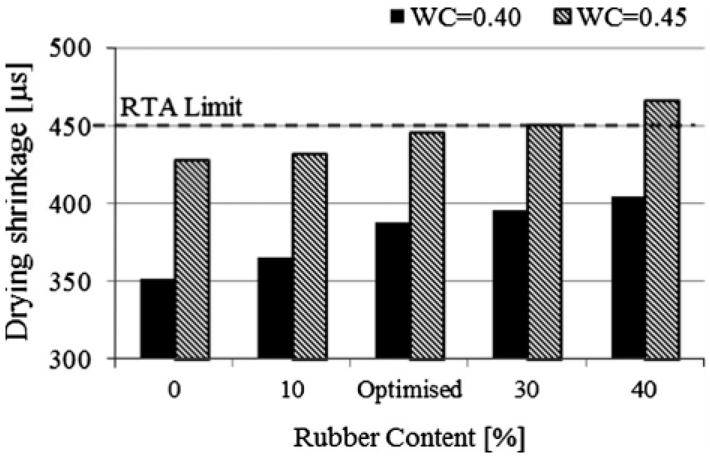


Figure 2.4. Drying shrinkage respect to rubber content [13]

The replacement of aggregate percentage with highly elastic rubber is to reduce the internal restraint of concrete and lower the elastic modulus of concrete. It revealed that lower elastic modulus led to lower thermal and shrinkage stress. Further, crumb rubber in concrete mix trap cracks and avoid the propagation of cracks [146]. Table 2.8 represent the improvements that were identified with experimental studies.

Table 2.8. Experimental identification to improve RuC shrinkage

Reference	Remark
[11]	The cracking time was reduced whilst the shrinkage properties were enhanced with less than 20% crumb rubber.
	The crack resistance of the mortar contained crumb rubber with a size of 1.5 mm improved
[147]	Drying shrinkage increased with increasing rubber content
[130][148]	Increasing the fine rubber content over coarse rubber, increase the drying shrinkage.
[82]	The crack area can be reduced by using rubber-filled mortar

2.8 Mechanical properties of rubber mixed concrete

2.8.1 Compressive strength

The literature concluded that the compressive strength of RuC is comparatively lower than that of conventional concrete [15][99][149][150]. A saturated number of studies have been conducted to identify the effect of rubber aggregate on the compressive strength of concrete. Further, specifications and improvements were also investigated to enhance the compressive strength of RuC. However, being the most critical property of concrete from a structural perspective, the compressive strength of RuC needs to investigate and recommended before implementation. Studies revealed that several factors such as the rubber aggregate size, shape, mechanical properties, rubber content, and rubber influence the compressive strength of RuC [14]. Table 2.9 illustrates the effect of using rubber aggregate in the concrete mix.

Table 2.9. Variation of compressive strength with respect to a rubber type

Reference	Rubber type	Particle size	Rubber percentage	Variation in compressive strength	Remarks
[66][150]	Crumb rubber	0.075-6mm	5-50%	Reduced by 4-70% compared to CS strength of 54 MPa	
[75]	Crumb rubber	1-10mm	0-30%	Reduced up to 71% from 56.33 MPa (SD = 1.65)	A combination of fine and coarse rubber aggregate used
[25]	Crumb rubber- fine	0-5mm	10-100%	Reduced by 13-84.4% compared to CS of 40 MPa	
	Crumb rubber- coarse	5-20mm	10-100%	Reduced by 25.6-86% compared to CS of 40 MPa	

	Crumb rubber- a combination	0-20mm	40% fine-60% coarse	Reduced by 83% compared to CS of 40 MPa	
[102]	Chipped rubber-Coarse	19-38mm	25-100%	Reduced up to 85% compared to CS of 35 MPa	200 cylinders with 150mm in diameter and 300mm in height were experimented with.
[38]	Crumb rubber- fine	0.5-5mm-	10-30%	Reduced by 14.4-28.6% compared to CS strength of 44 MPa	
	Crumb rubber-Coarse	7-15mm	10-30%	Reduced by 35.6-58.1% compared to CS strength of 44 MPa	
[47]	Powdered rubber	0-1mm	5-20%	Reduced by 30-63 % compared to CS strength of 44 MPa	The average value taken from 4 mixed proportions
[81]	Crumb rubber	1-7mm	15-30%	Reduced by 47-69% for NaOH pretreated, 51-69% for water	NaoH pretreated specimens show a comparatively

				pretreated compared to CS strength of 48 MPa (SD = 0.8)	lesser reduction
[151]	Crumb rubber	1.18mm average	6-18%	Reduced by 10.9-30.9 % compared to CS strength of 55 MPa	Fine rubber aggregate was replaced
[99]	Crumb rubber		25-100%	Reduced by 15-60% compared to CS strength of 26 MPa	Crumb rubbers enhance the strength more than chipped rubber
	Chipped rubber		25-100%	Reduced by 40-75% compared to CS strength of 26 MPa	

Note; CS – Control sample, SD – Standard deviation

Investigating the studies, specifications on rubber percentage, rubber type, pretreatment, and aggregate properties can be identified. Studies revealed that exceeding 30% of rubber content in concrete cause a huge reduction in compressive strength [75][25][47]. However, [54] concluded an optimized mix design that deducts 49% of compressive strength with 40% rubber replacement. Further, studies revealed that replacing fine rubber aggregate instead of coarse rubber can enhance the strength of RuC. But implementing a significant mix proportion of fine and coarse rubber aggregate can improve the mechanical properties by 2.6 times [25]. The reduction in compressive strength has been discussed by concerning different experimental studies. One such major is the poor binding between rubber-cement paste, in which rubber particles acts as voids by creating lower density in a concrete matrix [14][15]. This was proven by the study [16] by a scanning electron microscopic (SEM) test and confirming the presence of voids and cracks, possessing a weak bonding behavior. As a result of the softness of rubber particles, premature cracks are created along the rubber and cement paste [99]. Another factor is the very wide and porous weak interfacial transition zone (ITZ) possessed in RuC. This is explained because of the hydrophobic nature of RA, which tends to repel cement paste [17].

To enhance the weak bonding behavior in RuC, silica fume, and pretreatments were recommended [16][54]. As rubber aggregates contain a low specific gravity, rubber particles rise to the upper surface of the mold. To avoid such behavior non-homogenous matrix to use in concrete was recommended [14]. Minerals fillers such as siliceous, limestone with the use of silica fumes, and rubber fiber are used to enhance the strength of concrete [152][6] as ideal compaction can be achieved. Moreover, the incorporation of required solvents, fillers, and modifiers such as emulsion, resin, or other treatment methods on constituents are proven helpful for enhancing the bonding of the rubber-cement matrix [68][65][54][7][153].

2.8.2 Tensile Strength

The literature shows that the tensile strength of RuC is comparatively low with respect to conventional concrete. As rubber aggregates act as cavities due to micro-cracks resulting on the surface of rubber and cement, tensile strength dropped in RuC [14]. The rapid failure under tensile stress is also a result of weak ITZ and stress propagates along the ITZ. However, Akinyele et al. [154] revealed a 41% decrease in tensile strength when 4% coarse rubber was added to the concrete as a replacement for fine aggregate and a 58% decrease when 16% coarse rubber was used. Therefore, higher rubber constituents resulted in lower strength. In [155] revealed that powdered rubber shows enhancement in tensile strength than that of chipped rubber replacement in RuC. Further, Aslani et al. [22] reported a minimum reduction in tensile strength when 5mm sized rubber aggregate was used instead of the 2mm and 10mm sized aggregate. This behavior was explained by Gesoglu et al. [156], the smaller size rubber aggregates isolated from one another and can produce weaker bonding with cement paste. Conversely, the spitting failure occurs in rubber-incorporated concrete along the aggregates or paste, rather than at ITZ.

To improve the tensile strength of RuC, studies revealed that strengthening the bond between rubber and cement paste is to concern. Thereby, using admixtures and pretreating the rubber particles with NaOH were identified as enhancement methods [154]. A hybrid construction technique was introduced in term to improve tensile strength. This is to apply with two layers, the top layer with RuC and the bottom layer with conventional concrete. Thereby, RuC can be used on the top layer, whilst conventional concrete is on the bottom layer, which implies the hybrid concrete [157]

2.8.3 Flexural strength

The flexural strength of RuC does not show a specific pattern as other mechanical properties. Literature shows a contradiction in concluding the pattern. A three-point loading test was performed on identifying the flexural toughness [38]. The results indicate an increase in flexural toughness by 17.6% and 39.36% for 10% fine rubber replacement and 10% coarse rubber replacement respectively when compared to the control mix. But, with 30% replacement, flexural toughness decreased by 21.4%. Figure 2.5 represent the variation of absorbed energy with coarse and fine rubber aggregate.

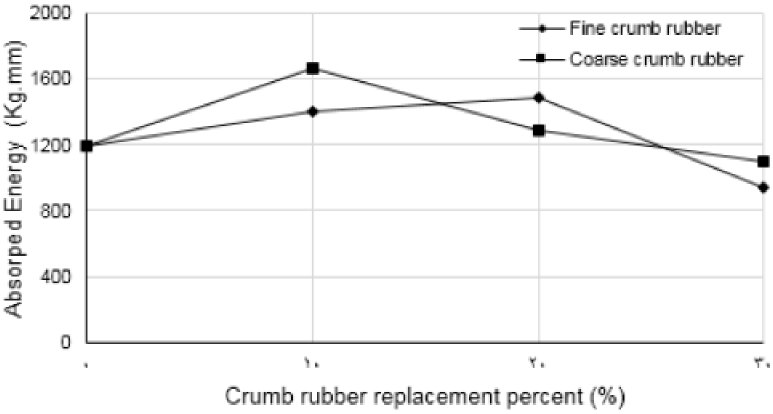


Figure 2.5. Abrasion lengths versus percent replacement by fine and coarse crumb rubber [38]

Conversely, decreasing flexural strength was concluded with rubber replacement [10]. 20% of coarse rubber replacement resulted in a 25-27 % reduction in flexural tensile strength [9]. However, sudden failure was not resulted in RuC, specifically under bending [100], as the incorporation of rubber increases the ductile nature and diminishes the brittle nature of concrete, where high deformation was observed in RuC [16][155]. The parameters identified by the studies to enhance flexural strength are represented in Table 2.10.

Table 2.10. Enhancing parameters of flexural strength

Reference	Specification
[158]	Replacing coarse rubber aggregate with fine rubber aggregate
[113]	Enhancement with decreased water/cement ratio and adding silica fume to enhance the bonding
[159]	Replacing with rubber fiber enhances the flexural strength
[160]	Adding filling material increases flexural strength by 20%
[56]	Adding silica fume decreases the flexural strength reduction
[161]	Inclusion of steel or synthetic fibers in RuC to improve the flexural strength and cracking resistance of it

2.8.4 Abrasion resistance

The abrasion resistance of RuC is comparatively enhanced than the conventional concrete [16][155][162]. Denser concrete matrix, identified to have better abrasion resistance, where the density of concrete increases with the addition of fine rubber particles enhances the abrasion resistance. Using a variety of sizes of rubber particles improves the abrasion resistance rather than including one rubber particle size [106]. Further, using rubber fiber instead of rubber powder improve the abrasion resistance [17] which exhibits lower wear depth. The study [163] mentioned that using crumb rubber enhances abrasion resistance, but adversely affects compressive strength. Further, the study concluded to use of silica fume with rubber replacement to improve the strength as well as the abrasion resistance. The study by [38] revealed that coarse crumb rubber enhances the abrasion resistance more than the finer rubber particles where a slight reduction in abrasion length resulted in 10 % rubber replacement, but 20% and 30% replacement of rubber shows a significant flop in abrasion length as represented in Figure 2.6.

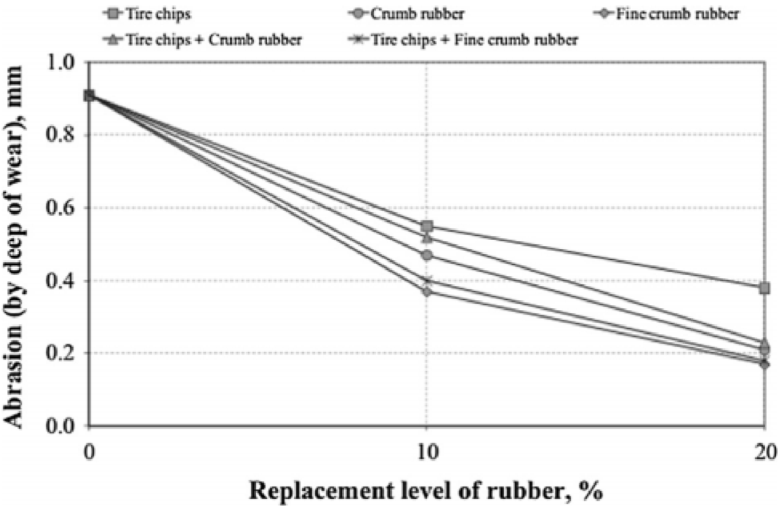


Figure 2.6. Variation of the abrasion depth of RuC with rubber content [163]

The increment in abrasion resistance has explained by the soft nature of rubber, which acts like a brush [162]. Also, rubber aggregate at the surface has more contact area for the abrasion test rotating disc, which causes more wear on the soft surface of rubber aggregate, hence rubber particles move to the top surface as it possesses low specific gravity with respect to mineral aggregates in the mix [150].

2.8.5 Impact resistance

Higher impact resistance values possessed in RuC are identified as one of the main enhanced properties of RuC. RuC has improved performance under impact loading than static loading [157]. The study [118] concluded that resistance to impact increased by 1.55 and 3.52 times with 10% and 50% coarse rubber replacement. Also, an 11.8% increment was concluded with 18% fine rubber replacement [4]. Further, 20% of CR inclusion resulted in a significant improvement of 279 % in fracture energy under impact loading [157]. Investigating the dynamic compressive strength, Study [164] concluded the great resistance under a high loading rate. Further, it explained that RuC significantly slowed down crack propagation and progressive failure as compared to conventional concrete. Similarly, RuC columns with 15% and 30% resulted in 58 % and 63 % enhancement in impact energy absorption compared to the reference columns, respectively [37]. In [38] revealed that 25%, 70.8%, and 150% increment in impact resistance with 10%, 20%, and 30% fine crumb replacement. This concluded as a result of absorbing more energy rubber particles. Further, the study compares the failure impact energy of fine and coarse rubber with different rubber contents as represented in Figure 2.7.

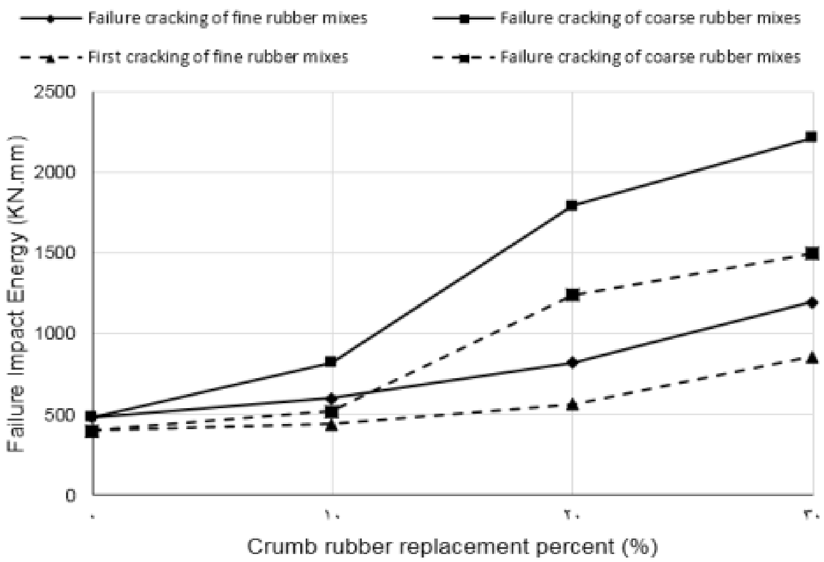


Figure 2.7. Initial and failure cracking impact energy versus percent replacement by fine and coarse crumb rubber [38]

The ability to absorb sudden shock by rubber aggregates over natural aggregate is explained by the non-brittle nature of rubber particles [68]. As rubber possesses enhance ductility, and provides enhanced resistance to crack propagations [19]. The study [165] heightens the requirement of having small-size CR aggregates, which mitigates the crack initiation under impact load. Further, the increment in the damping ratio is significant. Literature concluded that crumb rubber of 180 and 400 micro led to the best improvement in comparison with normal concrete [166]. However, excessive rubber is not recommended, as excessive rubber replacement causes porous nature, hence resulting from lower impact load carrying capacity [109]

2.8.6 Resistance to Fatigue

According to ACI concrete terminology, the weakening of material by the repeated load is defined as the fatigue of material. In the concrete matrix, fatigue depends on material composition, loading forms, and environmental conditions. Bridges and road pavements can be identified as crucial structures that should concern fatigue resistance [167]. The study [168] mentioned that the number of load cycles in RuC increased by 14.39% and 16.23% from conventional concrete for stress levels 0.9 and 0.8

respectively for 20% replacement of rubber ash. Further, the highest increment of 52.33% resulted when 10% rubber ash with 25% rubber fiber was replaced as fine aggregate. Therefore, higher rubber content enhances the fatigue life of RuC. The study [169] concluded that the replacement of 10% CR with an average size of 10 mm resists the maximum number of load cycles with constant deformation. Warm mix asphalt concrete with coarse rubber replacement has been identified to enhance the fatigue life under repeating load [170][171], hence coarse rubber improves the toughness, elasticity, viscosity, and aging resistance [171][67]. However, a minimum change in pavement slab thickness was observed by increasing the rubber content [172] (see Figure 2.8). Further studies are recommended on incorporating non-homogenous CR in the concrete matrix and its behavior with respect to hydration products at the interphase between cement paste and rubber particles. Also, no studies have been conducted to investigate on fatigue performance of the material under higher strain loads [169].

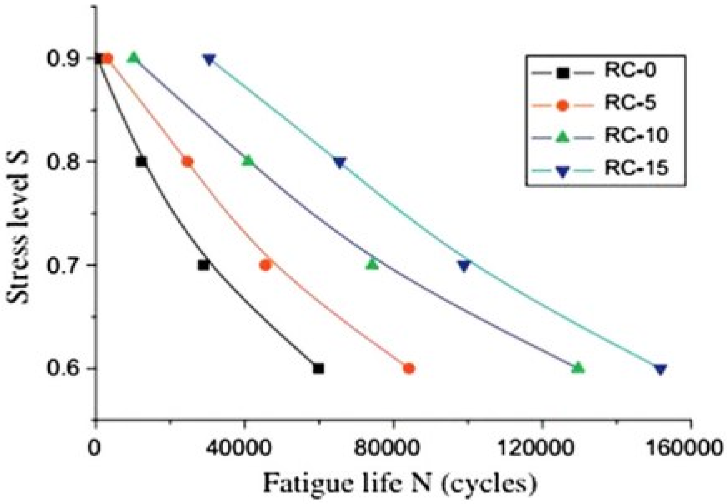


Figure 2.8. Fatigue life variation of RuC with varying rubber content [173]

2.9 Dynamic Properties

2.9.1 Dynamic modulus of elasticity

The modulus of elasticity of RuC shows a comparatively lesser value than conventional concrete. Further, with the increment of rubber replacement, the modulus of elasticity shows a decrease in pattern [122]. As illustrated in Figure 2.9 ground rubber shows a higher modulus in elasticity than crushed rubber. Hence, the rubber replacement improved from 15% to 45%, the dynamic modulus was reduced from

5.7% to 28.6% with respect to conventional concrete [122]. Thereby, the study revealed that the crushed rubber effect is superior to ground rubber impact in both dynamic and static modulus of elasticity of rubberized concrete. It is important to mention that the dynamic modulus of elasticity was found by both the beam element method and the elastic wave method. The study concluded that results obtained by the two methods have a very small deviation which is less than 10%.

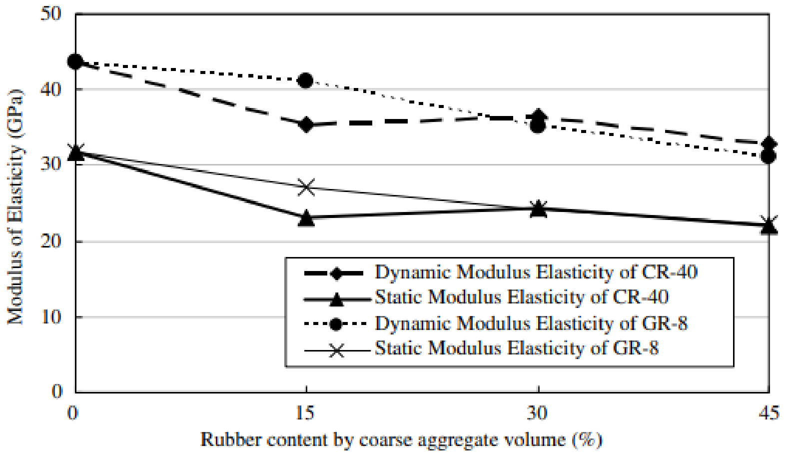


Figure 2.9. Comparison of dynamic and static modulus of elasticity [122]

2.9.2 Damping ratio

The damping ratio was identified as one of the enhanced dynamic properties of RuC [66][126][173]. As the damping ratio improved with rubber content, RuC can absorb more vibrational energy than conventional concrete [174]. The study [175] mentioned that the increment in the damping ratio is signed up to the early stage of the load cycle. Thereby, delaying crack initiation and rebar fracture under seismic loading can experience and will lead to lower demand for rebar [175]. The studies revealed that a 13% higher hysteretic damping ratio has resulted and a significant 150% energy dissipation but lesser viscous damping than conventional concrete [176] [177]. Further, the damage controlling, delaying, and reducing in RuC columns under seismic loading ate highlighted even though a 91.5% drift level has resulted in the study [176]. The study by [122] highlighted the comparison of ground rubber and crushed rubber with different rubber content and concluded the following points (see Figure 2.10).

- The ground rubber and crushed rubber concrete resulted to have 75.3% to 144 % increment in damping ratio at the first 40 cycles with reference to conventional concrete.

- Crushed rubber concrete enhances the damping ratio more significantly than ground rubber concrete.
- The damping properties of rubberized concrete are more sensitive to vibration response amplitude than plain concrete and enhancing the maximum response amplitude improves the damping ratio.
- Even though the damping ratio increased considerably with higher rubber content, the relationship is not linear.
- The optimal content of rubber concluded as 30% to get the standard static and dynamic properties hence, improving the rubber percentage can drastically reduce the modulus of elasticity of RuC.

To overcome the negative effect of high rubber content in the damping ratio, the inclusion of RuC in steel tubes was discussed hence the rubber possesses enhanced absorption capacity and ductile behavior [149]. However, the literature pointed out that, the damping ratio of RuC needs further experimental investigation for a better understanding of the variation of properties with high rubber inclusions.

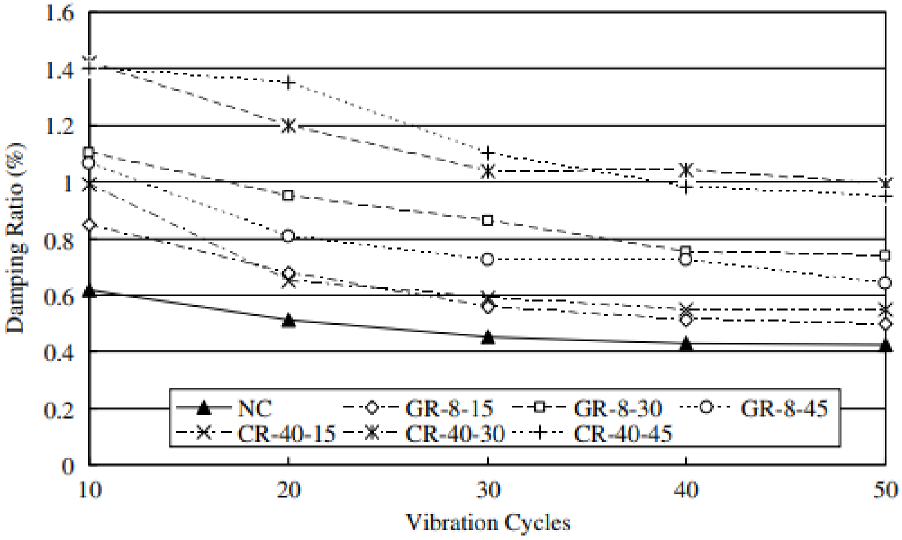


Figure 2.10. Decrease in damping ratio with the vibration of rubberized concrete [122]

2.10 Durability Properties

2.10.1 Resistance to carbonation

Reinforced RuC is identified to be a broad topic with the effect of corrosion, and is defined by carbonation. The RuC shows high carbonation with respect to plain concrete as higher depth in carbonation has resulted in increasing proportions of rubber aggregate [130]. The study [130] mentioned that the replacement of 15% of coarse rubber increases the carbonation depth by 56%. The study [81] did a comparative study on the carbonation of RuC based on two different pretreatment methods. Results concluded that at 28 days, an average depth of carbonation of 2.8 mm and 6.0 mm was obtained for 15% and 30% NaOH pre-treated rubberized specimens, respectively. The 15% and 30% water pre-treated specimens exhibited a carbonation depth of 3.9 mm and 7.2 mm, respectively. Further concluded that results show a linear relationship between the depth of carbonation and rubber content with high correlation factors as represented in Figure 2.11.

Several factors are identified as causing higher carbonation depth. The increment in void volume increased as mineral aggregate was replaced with rubber particles are different in both size and shape. This causes an increment in porosity, resulting in an increment in carbonation depth [81]. However, it is mentioned that ideal packing and denser mix can improve resistance to carbonation [16]. This explains by avoiding CO2 to infiltrate the concrete matrix resistance to carbonation can be increased [81]. A study [17] also confirmed the fact that rubber aggregate repels cement and thereby forms a porous matrix with weak ITZ in concrete. To improve the resistance to carbonation, a study [81] strongly concluded that pretreat rubber aggregates using NaOH. The results are heavily favored in inhibiting the penetration of CO_2 with non-treated RuC. However, carbonation resistance has a direct influence on strength, which explains that higher-strength concrete possesses high resistance to carbonation[178].

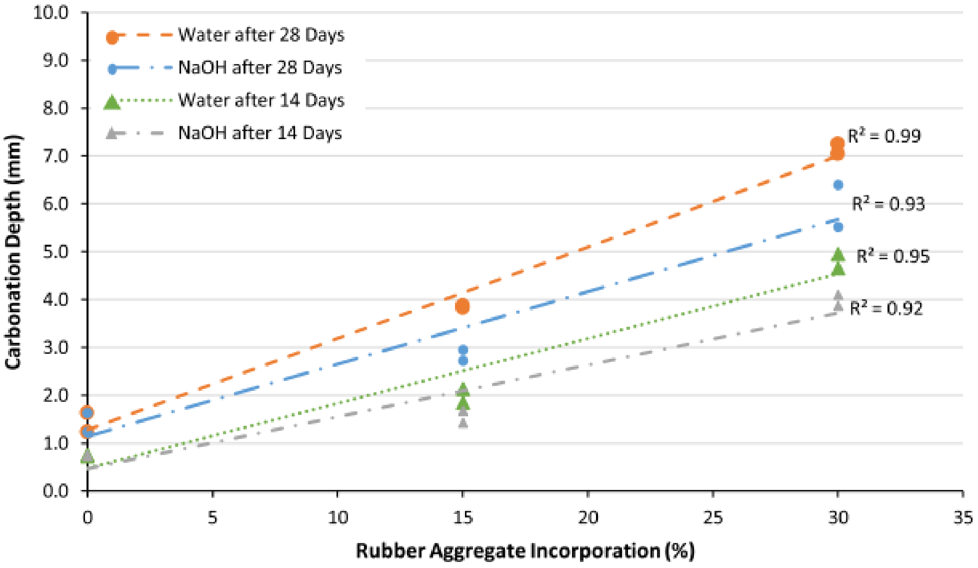


Figure 2.11. Relationship between the depth of carbonation and rubber content [81]

2.10.2 Water absorption

RuC possesses higher water absorption value with respect to plain concrete [16][17][155]. The comparative water absorption value is identified as a marginal increment, as a maximum of 1% difference was identified [81]. However, a diversity of conclusions can identify regarding water absorption, while both reduction and increment concluded in the literature. Table 2.11 represent the concluded studies on water absorption.

Table 2.11. Variation of water absorption with the variation of rubber replacement

Reference	Rubber replacement	Rubber aggregate size (mm)	Water absorption	
			Increased by	Reduced by
[130]	5–15% FA	0–4	3–14%	
[179]	0–12% FA			5–23%
[180]	0–7.5% FA	0–4		0–1.7%
	10–20% FA		0–2.5%	
[14]	5–10% CA	2–10	2.75–3.95%	
[181]	0-30% FA		5.9-7.2%	
[81]	0-30%	1-7	6.69% -7.72%	

It is concluded that water absorption of RuC increases with rubber content [182][81]. As a result of voids created in concrete and more voids within the concrete cause greater water weight in the concrete specimen. The study [183] revealed a correlation between water absorption and chloride attack for plain concrete. Further, the study mentioned that the chloride ion diffusion coefficient increases the water absorption, and exponential variation was noted among the two parameters. As the water absorption capacity of RuC depends on the rubber aggregate content, size, and water-cement ratio of the mixture, it is important to identify the significant proportions of the mixture. [184]. Further, the pretreatment of rubber particles with NaOH is considered to be more resistant to water penetration as represented in Figure 2.12 and recommended to use over other pretreatment methods.

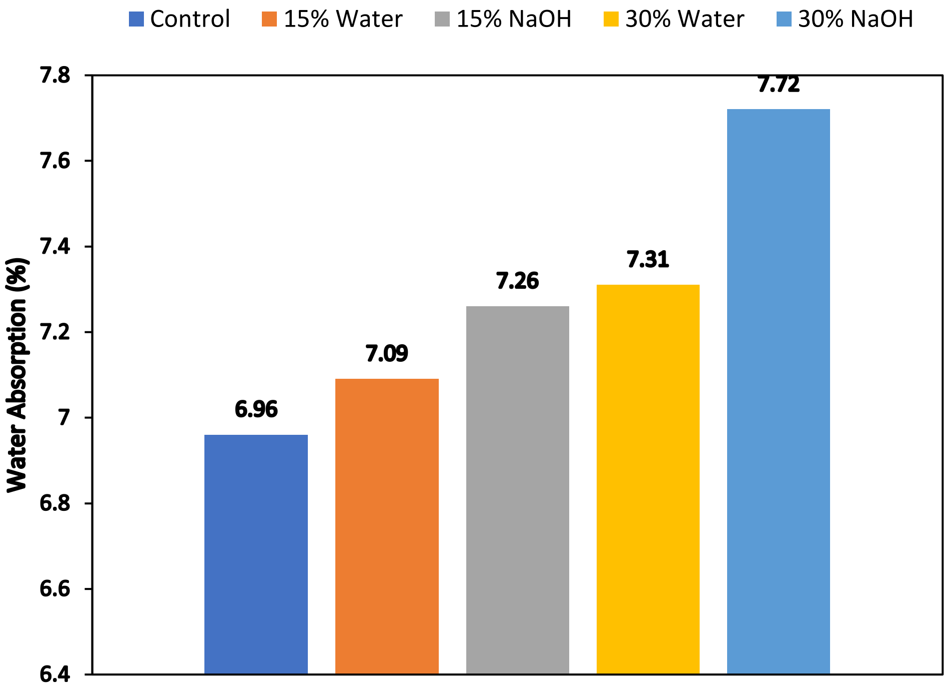


Figure 2.12. Water absorption of all the specimens [81]

2.10.3 Chloride ion penetration and Acid/sulphate properties

Corrosion of steel reinforcement is one of the major which influences on chloride ion penetration. The literature shows contradicting conclusions regarding the chloride ion penetration of RuC. The study [185] concluded an increment in chloride ion penetration with rubber replacement. The experiment was concluded for 0%,15%, and 25% rubber replacement and found maximum chloride ion penetration at 25% replacement with a 57% increment with respect to plain concrete. Similar results were reported in [130] showing an increment in chloride diffusion coefficient for 15% rubber replacement. Conversely, the studies by [180] and [186] concluded that mixing rubber aggregate up to 7.5% and 15% showed a reduction in chloride ion penetration by 5% and 35.85% respectively. Figure 2.13 illustrate the variation of chloride ion penetration with respect to different rubber contents [186] developed an electrical resistance method and recorded the chloride ion penetration. One of the factors revealed by [187] for chloride penetration was the total volume of permeability voids in RuC. The percentage of voids created in the specimen results the chloride ion penetration. This was confirmed by the studies [130][188] that ideal internal packing density will enhance the resistance to chloride ion penetration. Further, the finer size of rubber aggregate results in a closely packed matrix because of the filler effects of the rubber content. Conversely, increasing the size of the aggregates can increase porosity and subsequently increase chemical and water absorption. Similarly, study [16] concluded that CO_2 ingress through voids created on the RuC path, hence a dense concrete mixture is essential to mitigate the chloride penetration. To overcome the higher chloride penetration, short curing period was recommended and addition of silica fume with concrete was also recommended [185][189].

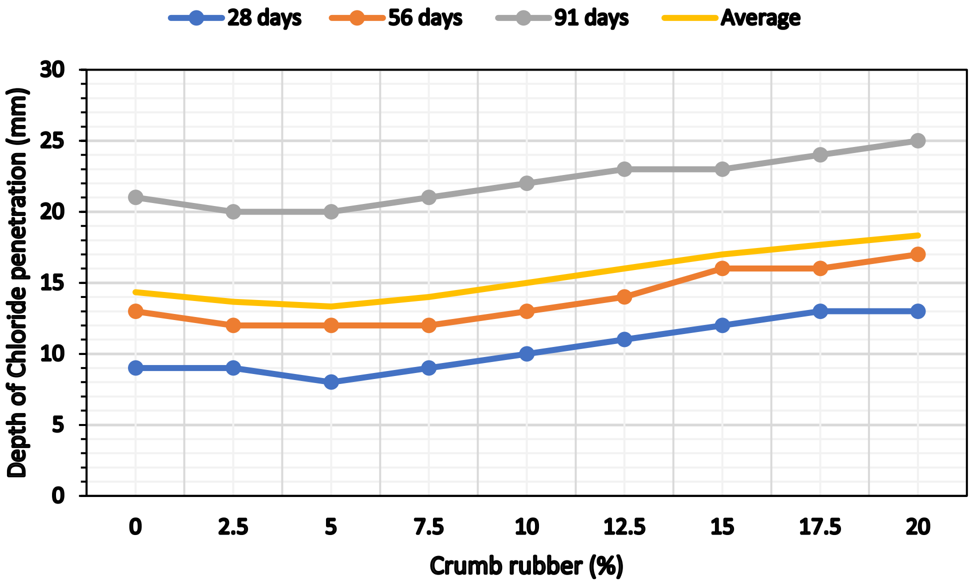


Figure 2.13. Variation in chloride penetration in RuC [182]

The other factor affecting the corrosion of reinforcement is the acid and Sulphate attack. This phenomenon creates negative impacts on cement aggregate with deterioration, cracking, and expansion. The study [9] concluded that RuC is enhance the resistance to acid and sulphate attack. This is supported by the study [145], which explained that the incorporation of 5% tire rubber as fine aggregates led to a 15% increased resistance to sulfate corrosion. Moreover, a sulphuric acid attack was identified as more disastrous than a sulphate attack, as the RuC retained 12% higher strength and 14% more weight compared to that of traditional concrete [180]. The removal of the top layer has occurred as a result of sulphuric acid action. However, the top layer wasn't affected by 20% of crumb rubber [188]. The ideal rubber replacement was concluded as 5% which passed through the number 30 sieve and had the best sulfate media resistance [145].

2.10.4 Sound absorption

RuC improves the sound absorption property with rubber content. As per the literature, sound absorption depends on the percentage of rubber content and the rubber type [18][116][190][42]. However, the sound absorption is resulted as marginal at 500Hz over pain concrete, but significantly greater at frequencies above 1000Hz and similar to that of plain concrete at 125-250 Hz [191]. The study [42] confirmed that the improvement of the sound absorption capacity of RuC is noticeable in the range of 500 Hz to 1000 Hz compared with conventional concrete. Further, a 33-48.6% improvement in sound absorption has resulted in the frequency range 800-1000 Hz with 80-100% replacement of fiber and coarse aggregate [192]. The study [193] revealed that mortar containing 25% CR enhances sound resistivity compared to conventional concrete. The reason explained for high sound absorption is given as the enhanced damping coefficient observed in RuC. The vibration produced from the sound wave was rapidly dampened and the sound was absorbed shortly [194]. In most practical structures, the frequency level is above the critical region, which is between 250-500 Hz. In that case, the dominant transmission mechanism is identified as the resonant vibration. Therefore, a reduction in resonant behavior has resulted in an increased damping ratio in RuC [116]. By proving that RuC is a good sound absorber, rubber incorporation diminishes the velocity of the ultrasonic pulse and ultrasonic modulus [97] (refer to Figure 2.14).

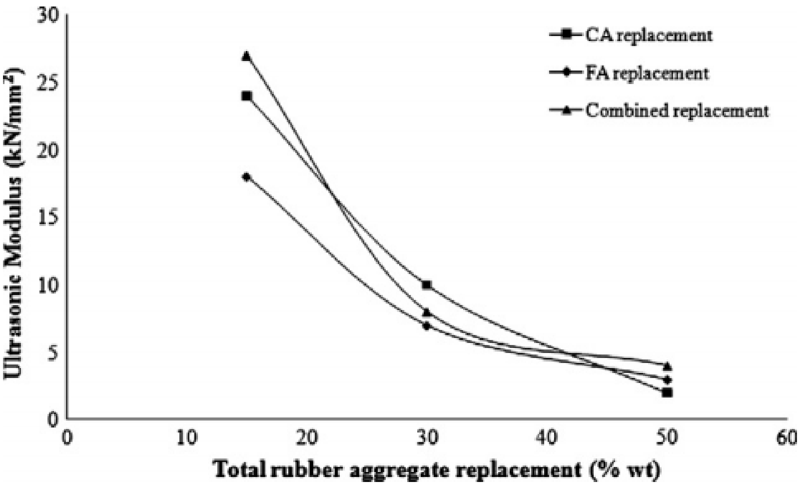


Figure 2.14. The effect of rubber content on the velocity of ultrasonic pulse [97]

2.11 Functional properties

2.11.1 Fire resistance and thermal conductivity

Under fire, rubber is a combustible material, therefore RuC is not safe as plain concrete in direct contact with fire [195][196]. The evaporation causes a loss in mass which lead to a decrease in compressive strength. Further, the study explained that at 300 ºC, concrete starts to shrinkage, above 400C C-S-H gels start to decay, and above 530 ºC cracking starts, and a decrease in compressive can experience [197][198]. In [195] concluded that by replacing 5%,10%, and 15% with crumb rubber at 800C for 1 hour, the compressive strength was reduced by 37.3%, 55.4%, and 69.5% of the control specimens. Therefore, increasing rubber content can cause a higher reduction in the fire resistance of concrete. Moreover, coarser rubber shows a higher reduction in fire resistance. The study [199] shows an average mass loss in concrete with 30% and 40% crumb rubber size between 5–10 mm is almost double the mass loss in concrete with 2–5 mm sized crumb rubber.

Due to the low thermal conductivity of rubber, RuC possesses better thermal insulation properties than plain concrete. Studies [49][200] revealed that rubber aggregate contains thermal conductivity in a range of 0.1 and 0.25 W/mK while mineral aggerate contains thermal conductivity of approximately 1.5 W/mK. Therefore, the thermal conductivity of RuC can be reduced by 50% of rubber aggregate [116], and this reduction continues with the finer size of rubber aggregate [52]. The study [116] on precast RuC concluded that low heat transfer ability whilst also being lighter in weight.

This was confirmed by the study with reference to the variation of [201] numerous rubber particle sizes, hence, the rubber content and size proportion to the variation in thermal conductivity. The same observation was also found by the study [94], which recorded decreased thermal conductivity for increasing rubber aggregate percentages. However, the studies highlighted the loss of mass, compressive strength, static modulus, and flexural strength of RuC at elevated temperatures. The study [202] highlighted the reduction of compressive strength variation with the exposure duration to heat, 30 min and 120 min, that the variation is increases but not significant in RuC. Further, reduction in static modulus was recorded as 46.1%, 49.2%, and 45.7% for 5% replacement, 25% replacement, and normal concrete, respectively at 300C for 120 minutes. However, with enhanced thermal insulated properties, RuC can be utilized in construction, where thermal resistance is critical in the building structure.

2.11.2 Electrical resistivity

Electric resistivity is defined as the ability to resist the flow of an electrical current. As rubber is a dielectric material and is used as an insulator for different purposes. Thereby RuC had shown better performance in electric resistivity compared to plain concrete. The study [166] concluded a 47% increment in electric resistance compared to the control specimen. Similarly, the study [81] confirmed that RuC generates improved positive potential than normal concrete, highlighting that high electrical resistivity caused less corrosion damage. As rubber is a nonconductive material for electricity, increasing the rubber percentage, enhances the electric resistivity and becomes more corrosion resistive [81]. As represented in [203], non-conducting electricity reduces the chances of steel getting corroding. The effect of rubber percentage on standard potential and the probability of corrosion is represented in Figure 2.15.

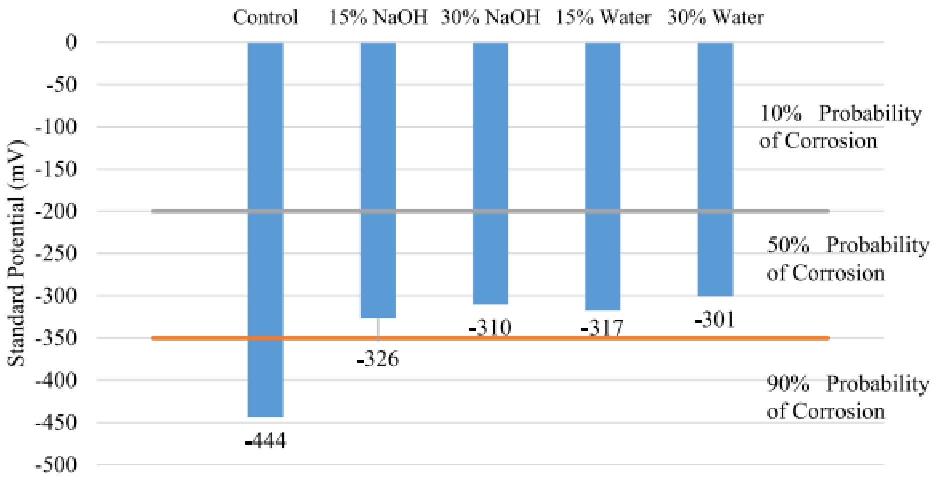


Figure 2.15. Standard potential for all mixes [81]

It is highlighted that the pretreatment of rubber aggregate can enhance the electric resistance [20]. Further, with increasing period RuC shows high resistance to electricity, however, the creation of calcium silicate hydrate produced in the hydration process act as a barrier to the electrical charges [113]. Moreover, the study recommends adding silica fume to enhance the electrical resistance.

2.11.3 Freeze-thaw resistance

RuC enhances the freeze-thaw resistance compared to plain concrete [6][204][162]. A comparison study [205] concluded the high freeze-thaw resistance possessed by RuC, as the results show that the dynamic modulus of elasticity achieved from RuC is after 150 cycles, while the conventional concrete achieves the same dynamic modulus after 50 cycles. Similar results were shown by the study [162] and further explained that the size of the rubber aggregate adversely affects freeze-thaw resistance. The study on finding the effect of optimum rubber size on freeze-thaw resistance [39], concluded that particle size less than 5 mm performance is the most effective. Moreover, it is identified that the finer particle creates high air entrain to the concrete matrix which generates higher freeze-thaw resistance over conventional concrete. Hence the use of finer rubber is more acceptable, with respect to freeze-thaw protection. The study [19] explained that micro cracks are generated as a result of water entering the porous concrete matrix and becoming ice at freezing temperatures. Thereby, the increment of volume and the pressure created in voids generate micro-cracks. However, to avoid micro-cracks, modification to the concrete mix is essential, and the use of fillers, admixture, and air void-reducing additives is possible [19].

2.12 Utilization of RuC

2.12.1 Non-structural application

Implementation of RuC in the construction field is the ultimate target of these research findings. One such area to utilize RuC is lightweight concrete. As rubber aggregates are less in weight than mineral aggregates, the density of the RuC possesses a lower value. According to EN standard [206], concrete was classified under the base of density class. Namely, lightweight concrete (LWC), normal concrete (NC), and heavyweight concrete (HWC) as shown in Table 2.12. Also, the code further sub-divide LWC into six classes namely: D1.0, D1.2, D1.4, D1.8, and D2.0 as shown in Table 2.13. Further, [207] classified lightweight under density and the respective compressive strength as shown in Table 2.14.

Table 2.12. Classification of concrete by density [210]

Types of concrete	Density (Kg/m3)
Lightweight concrete	800-2000
Normal weight concrete	2001-2600
Heavyweight concrete	>2600

Table 2.13. Classification of lightweight concrete by density [210]

Density class	Density range
D1.0	800–1000
D1.2	1001–1200
D1.4	1201–1400
D1.6	1401–1600
D1.8	1601–1800
D2.0	1801–2000

Table 2.14. Classification of lightweight concrete [211]

Properties	Low-density	Moderate strength	Structural concrete

Bulk density (Kg/m3)	320–800	801–1349	1350–1920
Compressive strength (N/mm2)	0.69–6.89	6.90–17.23	17.24–41.36

To identify the significant properties of RuC as lightweight concrete, it is essential to focus on concrete properties such as fresh properties, hard properties, and physical and chemical properties. The study [208] introduce block types, specified as a lightweight blocks, but the poor mechanical properties were highlighted as the product is not suitable for structural application. Further study concluded, even though mechanical strengths are poor, the energy absorption and flexible nature of blocks create possible applications[106]. Moreover, the study concluded that the aim of using waste crumb rubber in lightweight concrete was satisfied. Similarly, to use RuC in concrete pavements, proceed with good performance, with the replacement of 20% and 25% rubber in concrete at a water-cement ratio of 0.45 and 0.40 respectively.

The results of the study [209] on compressive strength gave a positive side of the research work because the 28 days compressive strength for the M16 concrete mix is higher than the recommended compressive strength of 15 N/mm2 for reinforced lightweight concrete as represented in Figure 2.16. Hence concluded that rubber crumbs can conveniently replace fine aggregate in concrete by up to 16% for lightweight concrete.

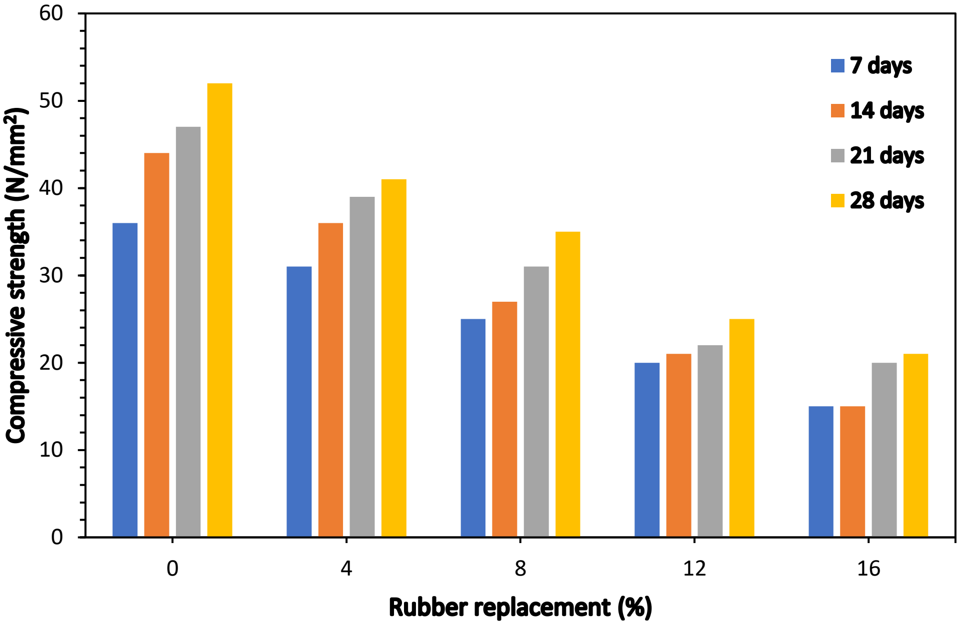


Figure 2.16. Compressive strength test [213]

The uncompacted density of lightweight RuC can be improved by replacing more crumb rubber with fine aggregate in concrete. As crumb rubber possesses low density, it is important to have significant compacted density. To achieve a well-compacted mix, the study [204] revealed an ideal packing of concrete by introducing an optimized mix portion. Further, the mix proportion enhances not only the density but the hardened concrete properties of RuC.

As a result of enhanced properties of absorption of impact energy and dynamic properties, RuC was identified to use in the protective structure against blast and impact loading [210]. Further, RuC can be helpful to reduce the impact force by up to 50% with an extended impact duration. Another application of RuC is to implement roadside barriers. The practical application of this can be seen in Thailand [211]. The study concludes that the accident costs can be reduced significantly, as the damage can mitigate from the server to a substantial stage. Similarly, the study [37] concludes the same results as it mitigates the accident cost. However, the modeling and feasibility should be further investigated.

2.12.2 Structural application

Reduction in compressive strength of rubberized concrete is a major concern when RuC to utilize in structural elements. However, investigations are carried out on overcoming the challenge of using rubberized concrete in structural elements. Studies

[212][164][213][25][37] discuss the enhancement of the strength of RuC under modifications. In less than 18% of rubber replacement, the behavior of stress-strain is similar to that of conventional concrete, but the reduction in axial strain is observed in RuC, which can be severe with high rubber percentages [25]. The increment in rubber content will lead to a reduction in axial strains was accompanied by a premature onset of localized micro-cracking. The dynamic responses of structural RuC are also a concern for researchers. Improving the mechanical strength by incorporating fiber reinforcement polymers is under investigation, hence the variation under impact loading is identified to increase significantly [37]. Enhancing the importance of CFST and CFDST methods, the study [213] mentioned the use of single and double steel tube use with the RuC improves the possible chances of use in structural elements.

The homogenous rubber replacement was found to be investigated, hence the variation in mechanical strength is significant with respect to coarse and fine rubber replacements[25]. Hence, investigating the non-homogenous rubber replacement is crucial, where the studies highlighted the enhanced concrete properties of high deformability and required workability. The following significant improvements were highlighted with confined FRP-confined rubberized concrete [25].

- Replacing aggregates with rubber increases the lateral deformation capacity of RuC by up to 300% over the plain mix.
- Confining such RuC with two and three layers of aramid FRP increased the compressive strength by up to 10.1 times over the control mix.
- Achieving significant enhancement, more than 14 times normal concrete.

With respect to the enhanced properties, Confined RuC is recommended for structural applications where high deformability is required. Moreover, RuC columns can be able to undergo more than two times lateral deformation without buckling failure compared to the control sample [214]. Prestressed member elements are also under concern, as it was proven that the negative effect of RA addition in concrete at the structural level is not as much as the material level [215]. The literature confirms the application of RuC is feasible with the proper modification to the concrete matrix and investigation of the critical parameters, where the RuC can be implemented not only in static loading but by extreme dynamic loading conditions[65], [176], [214], [216]–[219].

2.13 Future research needs

RuC is a prominent research material in the past decade. Researchers have investigated numerous concrete properties of RuC. However, most of the studies utilized CR as the replacement rubber type in the concrete matrix. Only a few studies have investigated the concrete properties with respect to other rubber types, hence it is important to compare the variation with respect to different material types. In addition, the physical properties and chemical properties of different rubber types are required to be investigated. Specifically, the element composition affects the concrete properties differently.

Even though literature discusses macro-level concrete properties, micro-level investigations are very less. To identify and overcome the actual burden of rubberized concrete, micro-level investigations are essential. It will help to modify the concrete matrix comprehensively. The dynamic properties of RuC were identified to be significant (refer to section 2.9), but no such application was implemented. Hence a comprehensive investigation of dynamic behavior with respect to different loading is crucial. Similarly, the durability properties of RuC are also not well discussed. Before the implementation of a structural element, the water penetration, chloride penetration, and impact under different weather conditions are essential to investigate. These areas need to be investigated comprehensively to explore the new dimensions of RuC.

CHAPTER 3 INVESTIGATION OF REPLACING AGGREGATE WITH NON-HOMOGENOUS WASTE TIRE RUBBER AGGREGATE IN CONCRETE - PHASE 1

3.1 Introduction

In the approach toward the sustainable construction industry, the use of waste material in concrete was identified as a turning point in innovative construction design. The use of waste tire rubber as a replacement for mineral aggregate is a viable way to reuse waste tire rubber through extensive-scale recycling projects [209]. Further, it is an approach of environmentally friendly and cost-effective. With respect to the variation of concrete properties, the range of applications of RuC has been identified. Table 3.1 represents the application of RuC with respect to the variation of concrete properties.

Table 3.1. Application of RuC

Properties	Application
Lower density [209]	As lightweight concrete
High damping ratio [20]	Industrial floor
	Pavement concrete
	Bridge sidewalks and decks
	Retaining structures
High energy absorption [220]	Roadside barriers
	Running tracks
	Bunkers and shooting ranges
High abrasion resistance [16]	Concrete highways and pavements
	Hydraulic structures
High freeze-thaw protection [19]	Construction in a cold climate

3.1.1 Dominant Reasons and Modifications on RuC

It is understood that the diminished mechanical properties of RuC restrict the implementation of structural elements. Therefore, identifying the dominant reasons for diminished mechanical properties and systematically addressing them is a crucial part of enhancing RuC properties.

- The high roughness of rubber particles creates higher friction between particles in the concrete mix and requires larger energy to flow, diminishing the workability and flowability of the concrete mix [22], [130].
- A high percentage of voids in the matrix creates a loss in strength and increases the porosity of the matrix [14], [15], [155].
- Poor adhesion between rubber aggregate and cement paste and lower density in the matrix as rubber acts as voids result in lower strength [14], [15], [116].
- A weak interfacial transition zone (ITZ) due to the hydrophobic nature of rubber creates poor bonding between rubber aggregate and cement resulting in a high rate of initial cracking [23], [66], [89].
- Non-uniform dispersion of rubber due to the same particle size and shape creates poor packing density creating a high percentage of voids that results in weak concrete strength [129].
- The rising of rubber aggregate to the top of the concrete mix due to the low specific gravity of rubber results weak interface layer of the matrix that affects the strength negatively [66], [89].

The aforementioned concerns are addressed through modifications to the design matrix and aggregate to enhance the RuC. Pretreatment of rubber particles was identified as one way of improving the rubber aggregate properties. To remove dust contains on the rubber surface and to reduce the roughness, divergent methods of pretreatment are used. Pretreating with Sodium Hydroxide (NaOH), Sulphuric Acid (H_2SO_4), Hydrogen Peroxide (H_2O_2), Calcium Chloride ($CaCl_2$), thermal treatment to rubber particles, and ultraviolet (UV) treatment are considered methods which thermal treatment idealized from the lot [221]. To achieve a well-packed matrix with enhanced density, the use of fillers, binders, and non -homogeneous rubber particle sizes and shapes in the concrete mix has been identified [6], [152]. Introducing solvents, resins, or medication to the matrix enhances the bonding behavior of rubber particles with cement paste [7], [52], [65], [68]. Further, to improve the workability, and limit segregation and bleeding, superplasticizers are introduced [54], [56], [129]. It is identified by the literature, RuC properties can be enhanced by modifying the concrete mix.

3.2 Experimental program

The rubber aggregate properties and mechanical properties of RuC were investigated experimentally using three series of concrete batches. A total of 12 cubes (150 x 150 x 150 mm) were cast from three different mixes.

3.2.1 Materials

All the mixtures were cast using Portland cement CEM 1 42.5 N. Commercial high range water reducing admixture was used. The fine aggregates were river-washed sand with a fineness modulus of 2.75 and a size range of 0-5 mm. The coarse aggregate was washed gravel with a size range of 5-10 mm and 10-20 mm.

The rubber particles were obtained from outdated vehicular tires through the process of mechanical shredding. The obtained rubber particles were separated into three ranges of sizes; a) fine rubber particles (0-5 mm) to replace fine aggregate, b) coarse rubber particles (5-10 mm), and c) (10-20 mm) to replace gravel. The particle size distribution of rubber aggregate was conducted according to ASTM C136 [222] and Figure 3.1 represents the particle distribution curve. To identify the rubber aggregate properties, density, water absorption, and bulk density experiments were carried out following Annex C of BS EN 1097-6 [223] (lightweight aggregate) and BS EN 1097-3 respectively [224]. However, these tests were not conducted for rubber aggregate size (0-5 mm) as it floats in water and agglomerate due to surface tension and inter-particular forces [25]. The test setup for investigating lightweight aggregate properties is represented in Figure 3.2.

3.2.2 Mix Design

The literature stated that to achieve enhanced fresh and hardened properties with a high rubber percentage in concrete the RuC mix should be modified. Further, to diminish the large-scale segregation and lack of cohesion, an optimized RuC mix with modification should be introduced. Raffoul et al. [54] introduced an optimized concrete mix with improved fresh and hardened concrete properties. It is mentioned that when compared to 100% fine aggregate replacement, the optimized mix was 2.6 times stronger. However, changes were made to the introduced optimized mix, as the used cement was 52.5 N and both plasticizers and superplasticizers were used. This was changed to a convenient Sri Lankan context by using Portland cement 42.5 N and only a superplasticizer as shown in Table 3.2.

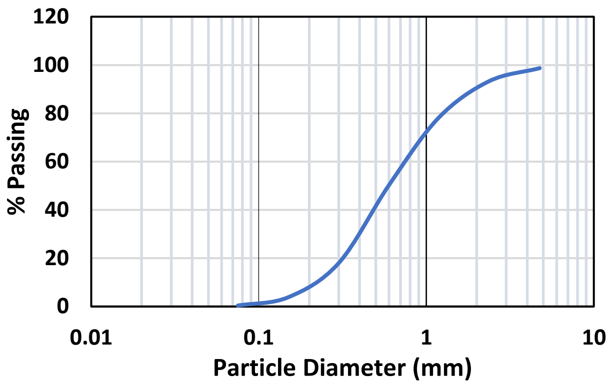


Figure 3.1. The particle distribution curve of rubber particles

The materials used in the concrete were mixed in the following procedure.

1. All minerals and rubber aggregates were saturated surface dried, and both were drily mixed for 30 seconds,
2. Half of the amount of mixing water was added and mixed for one minute, 3) The mixed was rested for three minutes,
3. The remaining half of the water and the binder materials were added with the gradual addition of admixtures, and
4. The concrete mix was mixed for another three minutes. The cubes were cast in three layers and vibrated for 15-20 seconds per layer on a vibrating table.
5. When the cast is finished, specimens were kept under laboratory conditions for 24 hours. After the cubes were de-molded and kept in a water curing tank for 27 days.

Figure 3.2. Instrumental setup for investigating rubber particle properties of size 5-10 mm and 10-20 mm

Table 3.2. Mix design [68][65]

Material	Quantity (kg/m^3)
Cement	340
Silica Fume	42.5
Fly ash	42.5
Fine aggregate (0-5 mm)	820
Coarse aggregate (5-10 mm)	364
Coarse aggregate (10-20 mm)	637
Water	150
Superplasticizer	7.66

3.2.3 Test Series and Instrumentation

The design mix was prepared to achieve high flowability as the water binder ratio is 0.35 with relatively high cement content. Three series were considered with respect to literature to obtain the highest mechanical properties with suitable rubber aggregate proportions. Rubber content varied from 0 to 20% of total aggregate by volume as 0% rubber replacement (Series 1), 20% fine aggregate replacement only (Series 2), and 20% total aggregate replacement by both 10% fine and 10% coarse aggregates (Series 3).

All cubes were subjected to compressive load using a 2000 KN capacity compressive machine. The cubes were tested at a rate of 0.6 MPa/s up to failure. Before the compressive strength testing, the specimens’ dimensions and density were measured to identify the rubber distribution and the density variation with respect to rubber content. Figure 3.3 shows the final setup during the test.

Figure 3.3. General view of the instrumental setup of compressive strength testing of sample specimen

3.3 Results and Discussion

Identification of the physical properties of rubber aggregate was a critical part of the experimental procedure, hence, to compare the variation with the literature an optimized design mix was used. Table 3.3 represents the physical properties of rubber aggregates. It was identified that the physical properties of rubber aggregate are almost the same or in the provided range by the literature.

Table 3.3. Physical properties of rubber aggregate

Property	Rubber (0-5 mm)	Rubber (5-10 mm)	Rubber (10-20 mm)
Apparent density (g/m^3)	-	933.1	1105.8
Oven dry density (g/m^3)	-	935.07	1120.65
SSD density (g/m^3)	-	967.1	1050.4
Water absorption (%)	-	6.18	1.25
Specific gravity	-	1.10	1.10
Loose bulk density (g/ml)	0.38	0.42	0.47
Rodded bulk density (g/ml)	0.47	0.54	0.62

3.3.1 Rubber Aggregate Properties

It is identified that the overall density of rubber particles has increased with rubber size. The comparison between loose bulk density and rodded bulk density shows a 19.14%, 22.2%, and 24% increment in each rubber size of 0-5 mm, 5-10 mm, and 10-20 mm, respectively. This can be explained by the higher percentage of voids created with larger sizes of rubber particles. Water absorption of coarser rubber particles showed a drastic reduction relative to fine rubber particles. It is understood that the surface area of the fine rubber aggregate is large respective to coarse rubber aggregate for a given mass. Therefore, the water absorption percentage is high in fine rubber aggregates.

3.3.2 Density and Compressive Strength

The mass of the rubber aggregate is very low compared to mineral aggregate. As low density was experienced in RuC samples over control samples. As the study focused on using various rubber sizes rather than using one particle size, approaching a well-packed matrix was studied.

It is observed that the dry density was reduced in RuC relative to the control sample as illustrated in Figure 3.4. But importantly using diverge rubber aggregate sizes enhanced the density over using only fine rubber aggregate. With respect to the control sample, a 9.07% and 3.39% reduction in density was experienced in Series 2 and Series 3 respectively. But an enhanced density of 5.88% was observed in Series 3 over Series 2.

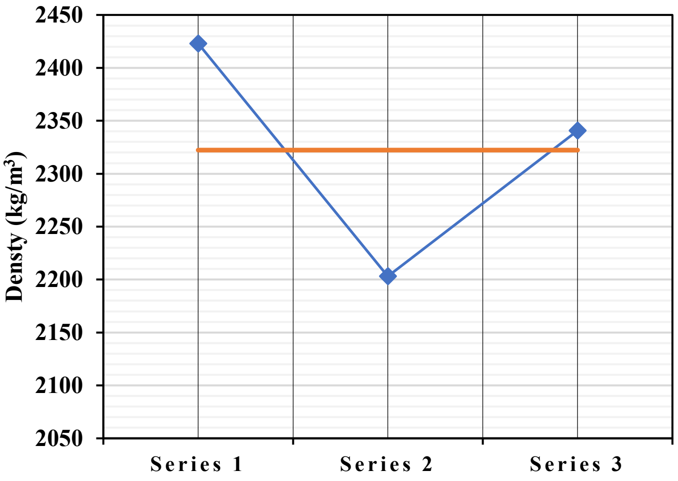


Figure 3.4. The average dry density variation of three Series of RuC specimens

It is identified that inadequate mix proportioning of aggregate causes a high impact on density reduction. The generation of a high percentage of voids in the matrix can be diminished by introducing non-homogeneous rubber aggregates. It is obvious from the results that the implementation of different rubber sizes reduced voids percentage and approached the ideal packing. Further, achieving a well-packed matrix improves the RuC properties such as compressive strengths, and tensile strength, and diminishes chloride ion penetration, porosity, and shrinkage.

The poor mechanical behavior of RuC is identified as the main drawback of RuC in using structural behavior. As represented in Table 3.4 compressive strengths were reduced with the inclusion of rubber aggregate. With respect to the control sample, 33.33% and 22.83% reductions were experienced in Series 2 and Series 3 respectively. However, compressive strength was enhanced by 10.5% in Series 3 over Series 2. The effect of non-homogenous rubber aggregate with adequate mix proportioning enhances the compressive strength of RuC. Further, it can be identified that compressive strength depends on the rubber content, size, shape, well-mixed parameters, and proportions of the concrete mix. The average 28-day compressive strength variation is shown in Figure 3.5.

The failure mode of RuC specimens showed a similarity, as specimen failure experienced from the top layer or bottom layer. This can be a result of the rising of rubber aggregate to the top layer of the concrete matrix due to the low specific gravity of rubber. Further, non-dispersion of rubber aggregate in the mix cause layer failures in the specimen. Figure 3.6 represents the failure patterns observed in Series 2 and Series 3.

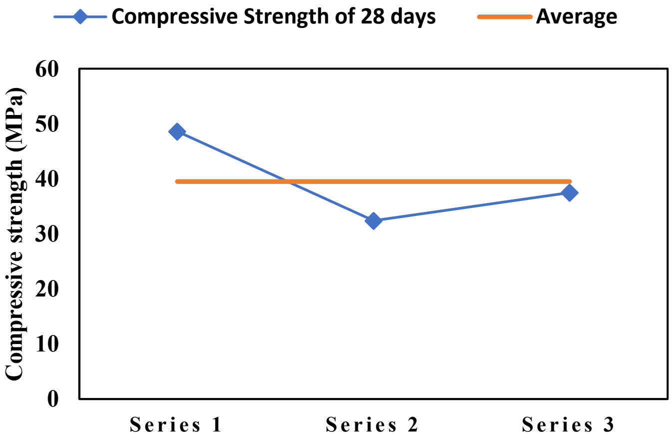


Figure 3.5. The average 28-day compressive strength of three series of RuC specimens

Based on the above discussion, it is evident that the divergent rubber aggregates enhance the density and compressive strength. However, premature failure can be experienced with non-disperse of rubber aggregate and more work is required to avoid the rising of rubber aggregate to the top layer.

Figure 3.6. Observed failure pattern of Series 2 and Series 3 specimens

3.4 Summary

This chapter investigates the properties of rubber aggregate and the advantage of implementing different rubber particle sizes with a modified design mix to enhance the properties of RuC. Based on the results of this study, the following conclusions have been made:

- The density of rubber aggregate increases with the particle size, while coarse aggregate shows enhanced rodded bulk density than fine aggregate.
- Water absorption of rubber aggregates results in high-value respect to mineral aggregate and smaller size of the rubber particles, showing higher water absorption. This enhancement is accompanied by the higher surface area created by a high amount of rubber particles.
- The density of RuC specimens with respect to the control sample is considerably less, but the combined replacement of fine and coarse rubber aggregate was concluded as the best option to improve density.
- The design mix achieved the targeted strength and enhanced the compressive strength of RuC. A modified optimum concrete mix design is the best solution to improve the properties of RuC.
- The compressive strength was reduced with the rubber inclusion, but the combined replacement of fine and coarse rubber aggregate is the best option to maximize compressive strength over a homogenous replacement of rubber.

CHAPTER 4 INVESTIGATION OF PRETREATMENT METHODS TO ENHANCE THE RUBBER MIXED CONCRETE PROPERTIES – A REVIEW

4.1 Introduction

The literature revealed that compressive strength results in lower values with the addition of rubber particles into the concrete [152], and it is mentioned that compressive strength further decrease with the increase of rubber content [4], [33], [170]. Similarly, tensile strength also shows poor behavior with rubber implements [154],[155] and poor tensile behavior increased with further replacement of rubber [22]. It was identified by the literature that there are several reasons such as; the high roughness of rubber particles [131], a high percentage of voids in the matrix [15], poor adhesion between rubber aggregate and cement, poor bonding behavior of rubber particles [116], weak ITZ due to hydrophobic nature of rubber [31] and non-uniform dispersion of rubber due to same particles size [24] effect on the poor mechanical behavior of rubber mixed concrete. The researchers have investigated to minimize and overcome the aforementioned reasons by considering an optimized concrete mix design [54], introducing plasticizers and superplasticizers [225], introducing fillers, and binders to reduce the voids [125], applying solvents, emulsion, resin or medication to improve the bonding of rubber particles [154], using non-homogenous rubber aggregates to diminish the void percentage and to improve the density [106]. However, mentioned improvising technics do not show a constant variation in improving the mechanical properties of rubber-mixed concrete.

It is revealed by the literature, the lack of proper bonding between the rubber particles and the hardened cement matrix is the major reason for diminished mechanical properties. Further, as a result of the hydrophobic nature of rubber, which repels water and entraps air on its surface leading to the ITZ between rubber particles and the hardened cement matrix and increasing the porosity of the composite [226][227]. Moreover, rubber particles entrain air bubbles during the mixing, increasing the number of minor flaws within the matrix [228][229]. Therefore, research investigations are taking place to modify rubber surfaces by introducing different pretreatment methods and techniques, to improve the bonding between rubber and harden the cement.

Researchers have investigated using of chemical treatments, thermal treatments, and physical treatments on the rubber surface. The chemical treatments such as Sodium Hydroxide (NaOH), Sulphuric Acid (H_2SO_4), Hydrogen Peroxide (H_2O_2), Sodium bisulfite ($NaHSO_3$), Calcium Chloride ($CaCl_2$), Silane coupling agent (SCA), and Potassium permanganate ($KMnO_4$) were investigated [230]. However, the study revealed that the results were inconsequential in improving the mechanical properties of the concrete matrix. Further, the variation resulted in a strength range of -2% to 10% for all the treatment types [230]. Washing the rubber particles with water is also considered a type of pretreatment. Pham et al. [75] chose washing with water over NaOH pretreatment as no conclusive variation resulted from NaOH pretreatment.

It is observed that the crumb rubber (CR) was treated thermally, as to enhance the capacity of bonding with hardening the cement. Abd-Elaal et al. [231] heated the rubber constituents to 200 °C before using rubber in the concrete mix. The study revealed a massive improvement of 60.3% in compressive strength over non -treated concrete matrix with rubber. Similarly, several physical treatments like UV treatment [87], pre-coating with cement paste and mortar [89], pre-coating with limestone powder [91], oxidizing the CR using $KMnO_4$ followed by sulfonating using $NaHSO_3$ [232] are investigated on rubber surface to improve bonding with the cement matrix.

Even though numerous methods were investigated, the bonding issue between rubber particles and the cement paste is not still addressed. As a result that the rubber mixed concrete still does not show enhanced mechanical properties. It is understood by the literature, a diverse number of research studies were focused on finding a feasible pretreatment method on rubber to improve the bonding. Hence, it is important to identify the effect of crucial parameters like, the time, size of rubber particles, amount of rubber particles, cost, accessibility, practicality, and safety of the pretreatment but not only the type of pretreatment method and its outcome.

4.2 Reasons for poor mechanical properties

The major drawback of rubber-mixed concrete is its poor mechanical properties. Hence, rubber-mixed concrete is still not applied in structural elements, but it is revealed and implemented on non-structural elements like, road pavements [16], road barriers [211], shooting ranges, sidewalks, bridge decks, and retaining structures [18]–[20], [36]. It is obvious and critical to improve the mechanical properties of rubber mixed concrete, hence it will break the limitation of the construction industry and success towards sustainability.

In generally, the compressive strength results poor behavior with the rubber inclusion. Further, the increment of rubber percentage diminishes the compressive strength [17], [99], [152]. By replacing only fine aggregate only respect to three proportions (2.5%, 5%, 7.5%) by CR, a gradual reduction is observed in compressive strength [233]. Replacing only coarse aggregate, a study [234] revealed a massive decrease of 83% in compressive strength with 30% rubber replacement. However, it can be observed that coarse rubber replacement shows a larger decrease in compressive strength rather than fine rubber replacement [29][235]. The decrease in the compressive strength was attributed to the poor bonding behavior of rubber aggregates and harden the cement, the lower density posed by the concrete matrix, the weaker ITZ created between rubber aggregate and cement, impurities, dust, and the smooth texture of rubber particles lean to poor boding between constituents. Further, it is observed that the high variation in rigidity between rubber aggregate and cement made high-stress concentration at the ITZ. Hence this leads to generating paths of least resistance to the load defamation to form cracks in the concrete matrix [233]. However, the literature concludes that the poor bonding between the rubber particles and cement matrix thereby creates weak ITZ attributes on poor compressive strength. Further, it is understood that the treatment of rubber particles is essential to improve the interfacial bond in rubber mixed concrete.

The resulting tensile strength of rubber-mixed concrete is comparatively low respect to conventional concrete. It is observed that 41% and 58% reduction in tensile strength with 4% and 16% of rubber replacements [154]. Further, the study revealed that a high percentage of micro-crack and premature failure occurs due the weak ITZ. The literature explains variations that occur with different rubber types, as powdered rubber shows improved tensile strength over chipped rubber replacement [155], and using non-homogenous rubber particle sizes improves the tensile strength [22]. Hence, the literature recommended to have modification to the concrete matrix, but importantly, treatment of rubber is critical to enhancing the bonding between rubber and cement. Similarly, flexural strength is also affected by the introduction of rubber aggregate. Unlikely both compressive strength and tensile strength, variation in flexural strength does not imply constant variation in rubber mixed concrete. The replacement of 10%

fine rubber aggregate enhanced flexural strength by 17.6% [38]. Conversely, the study [9] concluded that 25-27% reduction in flexural strength with 20% coarse aggregate replacement. The literature revealed that, with the rubber inclusion brittle nature of the concrete matrix diminished. Hence, the ductility index of rubber-mixed concrete was reported to be improved with a high percentage of rubber [236]. Further, to improve the flexural strength, studies were investigated on using steel fibers [237] and cement replace with zeolite [238] are considered a success with the enhance flexural strength of rubber mixed concrete. However, the initial origination of micro-cracks develops at the macro level as the quantity of rubber increased [239]. Moreover, the originated cracks transmitted from tensile to shear cracks as the load was applied, which implies the poor bonding behavior of concrete constituents in rubber-mixed concrete.

In general, the abrasion resistance of rubber-mixed concrete is higher than that of conventional concrete. Further, using non-homogenous rubber particles improves the abrasion resistance by the improving of well-packed concrete matrix resulting in high density [40]. Using three different sample sizes of rubber with varying percentages (25% of 2-4 mm, 35% of 0.8-2 mm, and 40% of powdered rubber) resulted in better abrasion resistance [16]. It was concluded that CR particles acted as confinement as a result of the high flexibility possessed by rubber [16]. However, controversial results are also reported, as rubber aggregate containing 4, 4.5, 5, and 5.5% of 0.6 mm CR shows the wear of depth increased with the rubber percentage [240]. Similarly, Ridgley et al [241] observed increasing in wear depth with an increment of rubber percentage in the mixture. It is revealed that the reduction in the abrasion resistance resulted by the means of poor bonding in the ITZ between rubber and cement paste [190], [240].

It is well defined by the literature, the mechanical properties of rubber-mixed concrete are poor with respect to conventional concrete. As the main reason, the studies highlighted the poor bonding possess by the rubber particles and the cement. Further, rubber repels water and entraps air on the surface as a result of the hydrophobic nature of rubber, leading the concrete matrix to the weak ITZ [26][242]. Therefore, it is essential to investigate innovative approaches to enhance the bonding mechanism of rubber particles with concrete constituents.

4.3 Types of Treatment on Rubber Particles

4.3.1 Washing with water or Water soaking

It is identified that one of the main reasons to have poor mechanical properties is the entrapped air in rubber particles as a result of the hydrophobic characteristics of rubber. Further, the impurities and dust include in the rubber surface weaken the bonding of concrete constituents and rubber particles [233]. The researchers have investigated several procedures of water soaking, out of which Mohamadi et al. [12] investigated introducing rubber particles in a wet process. (i) 20% of the required water for the concrete mix was measured and mixed with CR. (ii) in certain intervals of 5 min, 12 hours, and 24 hours, stirring the mixture was taken place. (iii) any water loss occurs was measured at a 24-hour stirring interval and the water amount was adjusted (iv) The water-treated rubber solution was added to the concrete mixture with the remaining amount of 80% of water. The study concluded that increments of 22% and 8% in compressive strength and flexural strength respectively than the untreated samples.

However, the failure pattern was described as, no major cracks responsible for the failure, but the number of cracks together on the surface failed [12]. Hence, this can be identified as the poor ITZ of rubber mix specimens resulting number of surface cracks. Therefore, the major reason for weak bonding between rubber particles and hardened cement was not overcome by water soaking, hence improving the bonding should be further investigated.

4.3.2 Sodium hydroxide treatment

Introducing sodium hydroxide (NaOH) solution to the rubber aggregate to remove oil and other chemical adhering to the CR and to remove dust [243]. The hydrophilicity and roughness of the rubber surface can be increased by introducing NaOH solution by changing the polarity of the rubber surface [80], [244], [245]. Moreover, NaOH reacts with the zinc stearate of the rubber layer to enhance the bonding of concrete constituents [33], [79], [244]. As a result of the aforementioned behavior of NaOH solution with rubber, most of the investigations were adhere to NaOH pretreatment. The literature has explained that increasing hydrophilicity and roughness increase the adhesion between the CR and the cement paste, but roughness should be maintained to a certain level as increasing roughness could increase the entrapped air in concrete [246]

Natural rubber is mainly composed of carbon is-polyisoprene, hence carboxylic acid group could form on the surface of rubber particles with the effect of aging. The effect of the chemical reaction between the acid carboxylic group with alkaline components of cement cause damage to concrete structure (i.e. $Ca(OH)^2$ and C-S-H) [244]. But, with the treatment of NaOH, the hydrogen ion on the acid functional group can be replaced by Na^+. Hence, the structure creates can provide a weak interfacial zone near the rubber aggregate and cement [94]. The method of introducing NaOH solution to the concrete matrix is by washing rubber aggregate with NaOH solution. To remove the impurities and dust, rubber particles are washed with NaOH solution. Hence, to maintain the pH 7, the treated rubber particles are washed with water to maintain adherence pH value. As well as, the final washing of rubber particles prevents the negative effect on concrete strength [78], [247]–[249].

The improvement of flexural strength, fracture energy, and good wear resistance was observed with NaOH-treated samples [250]. Similarly, the durability was improved by using 15% and 25% NaOH-treated CR. The drying shrinkage was reduced as a result of improved adhesion between rubber particles and cement. Further, NaOH-treated particles reduce the loss of moisture due to the low transport connection of rubber aggregate [187]. However, from the literature, it is observed that several factors such, as rubber type, rubber size, the rate of pretreatment, and solution concentration makes the difference in mechanical properties. Further, it is explained that particle size and surface dirt of the rubber is varying with the source, which results inconstant in mechanical properties [243]. Similarly, the effect of different NaOH solution concentrations affects the roughness of the rubber surface [251] which can cause an adverse effect on the performance of rubber-mixed concrete.

The studies revealed that the surface modification of rubber by NaOH enhances the bond between rubber and cement and overall mechanical and durability properties improved [238]. However, controversy results are observed, as treatment with NaOH solution decreases the strength of rubber-mixed concrete [245], [252]. Moreover, some studies do not show significant variation in mechanical properties with the treatment of NaOH solution [89], [187], [252]. Therefore, it is obvious that no constant variation occurs with the treatment of NaOH solution and hence no conclusion can be made regarding the effectiveness of NaOH treatment. Further, rather than removing impurities, and dust and making the rubber surface rough, the crucial issue of poor bonding between rubber and cement is not addressed by NaOH treatment. Hence, further investigations are in need of improving the machinal properties of NaOH treatment on rubber.

4.3.3 Treating with an acid solution

The idea of treating with an acid solution is to remove the impurities and to convert the hydrophobic to the hydrophilicity of rubber [253], [254]. Further, the roughness and shape of the rubber particle surface can be modified using acid treatment [68], [244], [255]. Importantly, enhancing the roughness of rubber surface to improve the adhesion between rubber and cement matrix was identified to be a positive affect [244], [256]. Literature shows that there have been investigated several acidic solutions like H_2OS_4, CH_3COOH, HNO_3, HCl, and $HClO_4$. It is found that the H_2SO_4 treatment decreases the WCA of rubber and enhances the compressive strength of the foam concrete with acid-treated rubber by 56% [244]. Likewise, most of the literature revealed that treating with H_2SO_4 shows a significant strengthening of the mechanical properties of rubber-mixed concrete [249], [256], [257]. It is shown by optical micrographs, introducing H_2SO_4 indicating corrosion in the rubber surface and the presence of zinc sulfate Sulphur crystals. Hence, zinc stearate and H_2SO_4 react with each other to improve the adhesion between the rubber and cement matrix [253].

The study which compared the effects of H_2SO_4 and HNO_3 revealed that H_2SO_4 improves the damping and strength of cementitious-rubber composites. The 1 mol/L H_2SO_4 was used to treat the rubber and the surface energy, and the damping was increased by 16% and 250% respectively over non-treated rubber samples [257]. Further, rubber treated with H_2SO_4 and CH_3COOH solutions improves water absorption, compressive strength, and flexural strength [68]. Similarly, Colom et al. [254] revealed that 5% of H_2SO_4 and 5% CH_3COOH show significant improvement in compressive strength, elastic modulus, and fracture modulus of rubber mixed molar. But HCl (5%) treatment did not strengthen the rubber-mixed concrete and 35% of HCl reduced the strength by 75%. This can be explained by the deformation of the rubber particles. The HCl solution deforms the corners of the rubber particles and reduces their irregularities which is essential to the anchoring bond between rubber and cement matrix [258]. Therefore, it is identified that H_2SO_4 and CH_3COOH enhance the mechanical strength of rubber-mixed concrete while HNO_3, HCl, and $HClO_4$ reduce the strength of mechanical strength of concrete, hence not suitable for surface treatment. In generally, acid solution treatment can dissolve esters, lactones, or carboxylic compounds on the rubber surfaces, which increases the hydrophilicity of rubber [253][254].

After the treatment of rubber with acid, the acidity of the concentration should be investigated. Further, studies revealed that after the acid treatment, the rubber should be washed with water to neutralize the rubber components [244][257]. Moreover, explained that to react with water in a concrete matrix, the pH value should be neutralized. However, the effect of acidity on the concrete matrix should investigate further, cause the effect on concrete durability is not discussed in the depth.

4.3.4 Oxidation and Sulphation

The oxidation of rubber is to diminish its hydrophobic nature while inducing the formation of hydrophilic functional groups on the surface of the rubber. Thereby enhancing the hydration of cement and improving the mechanical strength of rubber-mixed concrete [86][259]. The same concept was explained by He et al. [232] as to create a chemical bond between rubber and cement by providing strong polar groups to the CR surface. Further, this is to reduce the contact angle between the rubber and water.

The CR was treated by oxygen to nitrogen ratio conditions at different temperature levels [86]. The optimum temperature level was identified as 250 ^{0}C with an oxygen-to-nitrogen ratio of 0.04 or lower which results increased compressive strength, flexural strength, and splitting tensile strength of 153%, 63%, and 83% respectively. Similarly, the rubber is partially oxidized with an oxygen-nitrogen ratio of 0.25 [259], forming a hydrophilic S-O functional group on the rubber surface. The study revealed that the treatment changes the rubber-water contact angle, and enhances the bond between rubber and C-S-H.

The surface modification procedure followed by He el at. [232] is mentioned below.

1. CR was immersed in a 5% NaOH solution for 24 hours
2. CR was washed with tap water to neutralize the CR

3. CR was cured by 5% $KMnO_4$ and sulfuric acid was used to maintain the pH between 2 and 3
4. 60 ^{0}C thermal curing was used for 2 hours, and stirring was used to achieve better oxidation
5. To maintain the 60 ^{0}C temperature, CR was immersed in a saturated sodium bisulfite for 0.5-1 hour

The study revealed that the contact angle was reduced from 95^0 to 90.5^0 by oxidation. But 1-hour Sulphonation resulted reduced contact angle of up to 71^0 [232]. Thereby, the maximum adhesion between rubber and cement resulted in 41.1%. Similar results were obtained by the increment of compressive strength by 50% than the untreated rubber mixed concrete, which concluded that the bond between CR and C-S-H was enhanced by 23% [260].

Even though there were comparisons between treated and untreated rubber-mixed concrete there is no such positive increment in mechanical properties with respect to conventional concrete. Therefore, it is obvious to achieve the required mechanical strength by investigating pretreatment methods further. As well as one of the draw backs of oxidation and sulphation is the release of harmful gases, such as Sulphur dioxide and Nitrogen dioxide.

4.3.5 Silane coupling agent (SCA) treatment

The idea of introducing SCA treatment to rubber is to enhance the adhesion between rubber and cement since SCA acts as an adhesion promoter at the interface between materials [83][94]. The studies revealed that SCA shows significant reactions on the organic or inorganic interface to combine the rubber and cement matrix strongly [92][261].

Different methods of SCA treatment have been used by the researchers, while Dong et al. [92] introduced a method of treatment to coat SCA on rubber with a combination of thermal treatment. The procedure is as follows:

1. Aqueous ethyl alcohol solution was prepared in selected concentrations.
2. Silane added solution was stirred for 10 min using a magnetic stirrer.
3. The solution was followed up by adding rubber particles and the mixture was stirred for 20 min.

4. The mixture was heated up to 80 ^{0}C and cooled to room temperature while stirring the mixture.
5. The treated rubber was rinsed with alcohol by filtration and dried at 110 ^{0}C for 12 h.

It is reported that the stiffness and tensile strength improved [253][262] which was explained by the modified microstructure of the ITZ and enhanced bonding between rubber and cement [263][264]. The compressive strength and flexural strength were found to be increased by 43% and 20% [265]. Moreover, SCA agents of Z-6020 and Z-6040 were used to pretreat the rubber and revealed that bonding strength improved in rubber and cement aggregate [94]. In the process of cement hydration, the methoxy groups of SCA convert to hydroxyl groups (OH), which will bond chemically to inorganic cement paste in dehydration condensation [94].

The sol-gel technique was used in studies as a new technique to modify rubber with reactive precursors TEOS and SCA [266]. The idea is to promote the swelling of the precursor TEOS to the surface of rubber by implementing SCA. Thereby, introducing OH groups to react with cement paste. Further, it is revealed that the sol-gel technique is more efficient than using conventional SCA treatment. Another technique used was treating rubber in two stages process, in which rubber particles were washed with saturated NaOH solution and then treated with SCA on the rubber surface [267]. Results show a 73% increment in treated specimens over the non-treated specimen in compressive strength and significant enhancement in thermal conductivity.

4.3.6 Plasma polymerization

The target of achieving an improved reaction between rubber and water has been investigated by using the method of plasma polymerization. The idea of the investigation is to decrease the contact angle between rubber and water, thereby achieving enhanced bonding [268]. The study was conducted to identify the properties of rubber mixed concrete with the effect of plasma polymerization of ethanol on the surface of CR particles. The low-temperature plasma (LTP) treatment and polymerization of rubber method were used and listed below.

1. CR was kept inside the chamber by maintaining the pressure at 10 Pa.
2. At a flow rate of 60 standard cubic centimeters per minute, the chamber was injected with oxygen.

3. The variety of power levels (60,80, 100, 120 W) and durations (60, 150, and 300 s) were considered.
4. Before CR was introduced to the plasma reactor, the Ethanol was injected into the vaporizer chamber.

The literature claims that the plasma surface treatment improves the surface free energy, surface toughness, and surface wettability of polymers [269]. Further, as a physical treatment on rubber, plasma polymerization is widely used in modification polymers. The consists of charge particles (i.e., electrons and ions), neutral particles, photons, free radicals, and other activated molecules to improve the surface as required [270][271]. The study on the high-temperature performance of rubber mixed concrete conveyed significant enhancement in high-temperature performances, regardless of base asphalt source, CR size, and aging state [272]. Further, improved thermal storage stability, phase angle, and rotational viscosity were exhibited by the statical analysis.

The hydrophilicity was investigated by the sedimentation and contact angle tests. It appeared to be 95% of the LTP-treated rubber CR particles settled down [268]. A significant reduction in contact angle was reported for a power of 120 W for a duration of 300 s arrangement. The contact angle was reduced from 122° to 33° [268]. Even though literature discussed asphalt made up of rubber mixed, there are no specific conclusions on mechanical properties with respect to the plasma polymerization treatment of rubber.

4.3.7 UV treatment

The modification of polymers is a range of mechanisms, such as photo-oxidation, generating free radicals, cross-link polymers, and reducing toughness [87]. The literature explained that the presence of radical species, polar groups, and dangling bonds created surface-free energy which was identified to be the reason for enhanced adhesive bonding of the surfaces [273][274].

The treatment method was conducted using a simple light box apparatus. Treatment was taken place spreading the CR on a reflective metal substrate to an even thickness. 10-minute intervals of UV treatments were undertaken to cast the rubber-mixed concrete specimen. Further, a minimum time elapsed is attributed for the rubber particles to return to ambient temperature after the UV treatment [87].

The UV radiation in the presence of allamine results in reducing the WCA of rubber and enhancing the roughness of the rubber particles. Moreover, concluded that the wavelength and processing time influenced the rubber mixed concrete [275]. The study by Ossola et al. [87] revealed an optimum of 253.7 mm wavelength and an exposure time of 40 hours which was classified to obtain the best results. The improvement in bonding between rubber and cement matrix has resulted which leads to enhanced compressive and flexural strengths.

The study by [87] concluded that the enhancement in ITZ between rubber and cement matrix reduced the loss of mechanical properties. The pretreatment process can be optimized by arranging the wavelength and by modifying the apparatus to obtain uniform treatment, which leads to enhanced mechanical properties. However, it is concluded that alternative methods should be investigated on pretreatment to improve the obtained results which influence the rubber cement interface [87].

4.3.8 Thermal Treatment

The idea of thermal treatment on CR is to treat the surface by removing the dust and the impurities of the rubber particles. Hence to improve the bonding between rubber and cement matrix [276]. The literature revealed significant enhancement in mechanical properties resulting in almost 100% strength recoveries. However, the critical part is that the comparisons were made with respect to conventional concrete strengths. The strength recoveries were listed in Table 4.1

Table 4.1. Summary of strength recovery by thermal treatment

Rubber percentage	Compressive strength	Tensile strength
10%	93%	106%
20%	60%	82%
0%	47%	57%

The researcher pointed out two resulting factors claiming the significant increment of thermal pretreatment. (i) impurities were removed from the rubber surface, and this was justified by the SEM images, (ii) A hard shell was developed by the rubber particle surface. These effects enhance the ITZ and stress transfer between rubber and cement matrix [277]. However, it is important to identify the optimum time of thermal treatment, hence, several reactions could harm the rubber texture and concrete afterward. The study by [276] concluded an optimum heating duration of 1.5 hours.

The rubber size and duration of treatment are crucial in achieving the optimum result of pretreatment. It is identified that, the smaller size of the CR, the higher enhancement of mechanical strength [231]. The samples made with 0.425, 0.6, 1-3, and 2-5-mm rubber particles resulted 40%, 28%, 18%, and 3% strength increments with respect to untreated samples. Hence, this shows that a smaller size is more beneficial in rubber-mixed concrete [231].

Even though thermal treatment can be identified as superior in recovering mechanical strengths, a full-strength recovery was not achieved by the investigated pretreatment methods. However, researchers claim that numerous construction applications which do not require high-strength concrete can implement rubber-mixed concrete with resulting strengths. But it is be found that rubber-mixed concrete should be further investigated in implementing structural elements.

4.4 Shortcomings of Pretreated Rubber

It is observed that several treatment methods were utilized to improve the mechanical properties of rubber-mixed concrete. The treatment methods have been divided into chemical and physical treatments. The idea of removing the impurities, dust, and lubricants precipitated on the rubber surface is the expected outcome of the pretreatment. Idolizing the rough texture of rubber particles is a purpose of pretreatment to enhance the bonding between rubber and cement matrix. However, the spotlight on enhancing the bonding of rubber mix concrete with respect to conventional concrete should be further investigated regarding pretreatment methods.

The study by Raffoul et al. [33] concluded that the variation in performance of concrete properties of the samples with pretreated particles compared to samples with as-received rubber particles shows a similar standard variation with respect to conventional concrete. Further, the study claims that using pretreatment methods is costly, time-consuming, and aggressive to the concrete and rubber particles, and pretreatment does not exhibit significant gain to the concrete samples. However, this contradicts the results shown by the studies, as pretreatment enhances the concrete properties over as-received samples. The disquiet is not achieving the target strength to comply with the conventional concrete. Table 4.2 represent the variation of pretreated rubber mixed concrete with respect to its control sample.

Table 4.2. Percentage of strength reduction with respect to the control sample

Reference	Pretreatment type	Percentage of reduction with respect to control mix		
		Compressive strength	Tensile strength	Flexural strength
[230]	Water wash	23.45	6.92	
	NaOH	45.21		
	H_2O_2	48.4		
	$CaCl_2$	45.4		
	H_2SO_4	48.59		
	SCA	45.77		
[87]	UV exposure for 40 min	0.23		5.84
[278]	Cement pre-coating	60.82		59.41
	Water Soaking for 24 hrs	18.75		20
[279]	NaOH (5% rubber replacement)	59.79	27.10	
[12]	SCA (5% rubber replacement)	61.87	30.84	
[68]	NaOH treated mortar	12.82		1.5 (increased)
	H_2SO_4 treated mortar	20.51		7.81 (increased)
	$Ca(OH)_2$ treated mortar	15.38		4.68
	CH_3COOH treated mortar	33.33		7.81

It is recognized by the studies that the pretreatment methods have not achieved the strength reciprocated by conventional concrete. But it is examined that the treatments to rubber particles have enhanced the mechanical properties of as-received rubber specimens. Hence, the general detriments of pretreatment methods are identified in Table 4.3

Table 4.3. general detriments of pretreatment methods

Pretreatment method	Detriment	Reference
Washing with water	Poor significant enhancement	[12], [72], [74], [89], [280]
Pre-coating with cement	Complex in use	[33], [80], [281], [282]
	Time-consuming	
	Poor significant enhancement	
NaOH	Complex in use	[80], [94], [146], [243], [283]
	Time-consuming	
	Negative effect on the environment	
SCA	Negative effect on the environment	[83], [92], [279], [283], [284]
	Complex in use	
Acid treatment	Complex in use	[256]–[258]
	Time-consuming	
	May be harmful to the concrete	
Partial oxidation	May damage to rubber surface with high temperature	[86][259]
	May harm the environment	
	Time-consuming	
UV	Poor significant enhancement	[87]
Soaking with acetone	Toxic to the direct use	[153]
	May harm the environment	
	Complex in use	

4.5 Significance of Graphene Oxide

In recent, the investigation of using nanomaterials in the construction industry is identified to be increased. Out of several candidates, GO is promising with exquisite mechanical properties and enhanced dispersibility [285]. GO is a carbon-based nanomaterial, which was introduced by Benjamin C Brodie in 1859 [286]. GO possesses a similar aromatic structure to any other derivation of graphene, but the covalent bonds were broken as a result of chemical reactions occurred. Hence, the functional groups of epoxy, carbonyl, hydroxyl, phenol, etc. are attached to the sp^2 aromatic monolayer structure of GO [287][288].

In correspondence to functional groups, GO resultant to be highly reactive which produces outstanding mechanical, electrical, thermal, and chemical properties [289][290]. Further, the affluence of functional groups causes GO compatible with the rubber matrix [291]. The enhanced properties of GO can be identified with the interaction of cement. The easy absorption of free radicals of water from cement resulted due to the hydrophilic nature of functionalities and the peculiar layer structure also effect immensely in modifying cement-based material [292]. GO shows preeminence over other nanomaterials because of the easy dispersion in water. This behavior also resulted in the oxidative functionalities possessed in GO [292]. The literature concluded that the concrete properties are enhanced with the use of GO. Table 4.4 represents a summary of the mechanical properties of concrete incorporated with GO.

Table 4.4. Summary of mechanical properties of concrete incorporated with GO

Reference	Property	Description
[293]	Compressive strength	For 0.03% of GO replacement by cement weight, a 40% increment resulted.
	Tensile strength	
[294]	Compressive strength	For 0.05% of GO replacement by cement weight, a 15-33% strength increment resulted.
	Flexural strength	For 0.05% of GO replacement by cement weight, a 41-59% strength increment resulted.

[295]	Compressive strength	For 1% of GO replacement by cement weight, a 63% strength increment resulted
[296]	Compressive strength	For 0.04% of GO replacement by cement weight, a 15.1% strength increment resulted
[297]	Compressive strength	For 0.06% of GO replacement by cement weight, a 21-55% strength increment resulted
	Tensile strength	For 0.06% of GO replacement by cement weight, a 16-38% strength increment resulted
[221]	Compressive strength	For 1 mg/ml of GO pretreatment on rubber, a 50.3% strength increment resulted
	Flexural strength	For 1 mg/ml of GO pretreatment on rubber, a 70.4% strength increment resulted
	Tensile strength	For 1 mg/ml of GO pretreatment on rubber, a 68.3% strength increment resulted

A comprehensive behavior was identified in the enhanced mechanical properties, hence GO acts as a filler [298], added nucleation sites were provided in the cement hydration process [299] and adjusting the production of the cement hydration process [300]. However, an optimum amount of GO should be identified, because the mechanical properties of cementitious material may diminish by exceeding the optimum amount, by forming clusters [301]. This can be explained by the larger surface area of GO which tends to absorb higher water amounts. Hence, create clusters of GO in the concrete matrix [302][303]. Further, this results in poor workability.

With respect to the significant properties owned by GO, it is a prominent candidate to enhance the mechanical properties of rubber mixed concrete, as to enhance the bonding between rubber particles and cement.

4.6 Summary

The poor mechanical properties of RuC were identified as the main drawback of the novel material. Rubber aggregate does not possess equal bonding properties as mineral aggregates, hence poor bonding between rubber and cement is highlighted by the literature. In addition, the weak ITZ creates due to poor bonding leads to diminished mechanical strength. Therefore, improvement in bonding between concrete constituents is essential.

- The bonding between rubber and cement should be enhanced to improve the mechanical strength of RuC.
- Surface change to waste rubber is crucial to enhance the bonding behavior with concrete constituents. Hence, pretreatment methods are introduced to improve the bonding between rubber and cement.
- NaOH pretreatment was identified as the most utilized pretreatment method in studies. The pretreatment method improves the mechanical properties, but the variations are inconsistent.
- Thermal treatment identified as the most effective method used thus far, improving the compressive strength by 98% with respect to untreated RuC.
- Several shortcomings such as pretreatment methods are costly, time-consuming, and aggressive to the concrete and rubber particles. Further, pretreatment does not exhibit significant gain to the concrete samples.
- The significant characteristics of high reactivity, outstanding mechanical, electrical, thermal, and chemical properties, and affluence of high functional groups identified GO as a prominent candidate for the pretreatment of CR.

CHAPTER 5 GRAPHENE OXIDE PRETREATMENT ON WASTE RUBBER AGGREGATE – PHASE 2

5.1 Introduction

For the past couple of decades, researchers investigated using waste tire rubber as a replacement for coarse aggregate in the concrete matrix. As the massive population of waste tire rubber keeps increasing annually, it is critical to identify sustainable approaches to using waste tire rubber to safeguard the environment. A 2.8% compounded growth was forecasted annually to the year 2025 [25] and an estimated value of 5 billion annual waste tire rubber by 2030 [226], [304]. If the issue is abandoned, this will lead to hazardous environmental conditions [46], [226], [304]. The inclusion of CR in concrete matrix enhances the properties of concrete; energy absorption, damping ratio, impact resistance, thermal resistivity, sound resistivity, freeze-thaw resistance, decrease in acid penetrations and chloride penetrations [226], [246], [305].

Even though numerous advantages were reported, poor mechanical properties hinder the expansion of rubber mix concrete. Compressive strength was identified to be decreased with the percentage of rubber in the concrete matrix. A 71%, 13%, and 60% reduction in compressive strength were reported with 30%, 10%, and 100% rubber replacement, respectively [306] [25][99]. However, the compressive strength of concrete mix varies with rubber percentage, type, particle sizes, and shape [62]. Akinyele et al. [4] reported that 41% and 58% reduction in tensile strength with 4% and 16% rubber replacement, respectively. However, the non-homogenous rubber particles show enhanced tensile properties[131]. Flexural strength does not show a certain pattern in strength variation. Hence 17.6% flexural strength was increased by 10% fine rubber particles, whilst 30% rubber inclusion indicated a 21.4% decrease in flexural strength [38]. Further, sudden failure has been avoided with the diminishing brittle nature of concrete matrix [16][100]. Several reasoning has been concluded by researchers for the poor mechanical behavior such as the high roughness of rubber particles, a high percentage of voids, and non-uniform dispersion of rubber [131][24][16]. However, the loss of mechanical strength is directly associated with poor adhesion between CR and cement matrix. This is due to the hydrophobic nature of rubber which entrapped a high percentage of air on its surface, drives off the water, and leads to a weak interfacial transition zone between CR and the hardened cement matrix [226][246][17].

It is understood that the surface of rubber has to be incorporated with a surface treatment to enhance the bonding between CR and cement matrix, hence researchers investigated distinctive pretreatment methods. Mohamadi et al. [15] investigated introducing rubber particles in a wet process. The water-treated rubber particles responded with 22% and 8% increments in compressive strength and flexural strength with respect to untreated concrete samples. However, the study indicated the appearance of cracks as a result of ITZ. An enhancement in durability, flexural strength, fracture energy, and wear resistance were observed with 15% and 25% NaOH-treated samples [250][187]. However, Chou et al. [93] concluded diminished mechanical properties with NaOH-treated samples, hence, no certain variation was identified. Sulphuric acid (H_2SO_4) treatment overruled the other types of acid treatments. The study by Kashani et al. [244] shows a 56% enhancement in compressive strength relative to untreated concrete samples. Similarly, Leung et al. [257] concluded that 16% and 250% increment in surface energy and damping with 1 mol/L of H_2SO_4 treatment. However, the chemical treatments were identified to be varying the mechanical strength by -2% to 10% [221]. These techniques are not successful, may result in durability problems in service, and are also practically not feasible. The physical treatments of rubber aggregates such as thermal treatment, ultraviolet (UV) treatment, pre-coating with cement paste, oxidization, sulphation, silane coupling agent treatment (SCA), and plasma polymerization were investigated by the researchers. Abd-Elaal et al. [277] claimed that thermal heating recovers the strength over other treatment methods saying that 60.3% recovery strength at 20% rubber replacement. Further, the study revealed that other physical treatment recovers the strength in the range of 4% to 48.79% [277].

It is obvious from the literature; a 100% strength recovery was not achieved thus far. The use of chemicals and washing and drying process of these treatments are time-consuming and practically not feasible. In addition, added alkalinity or acidity may cause long-term problems in concrete. Further, the poor bonding between CR and cement matrix has not been expounded and the pretreatment investigations are continuing. This study focuses on implementing GO as a pretreatment method to enhance the mechanical properties of rubber-mixed concrete. The GO draws the attention of researchers because of its significant mechanical, electrical, thermal, and chemical bonding [289][290]. The sp^2 (the mix of one s and two p atomic orbitals) aromatic monolayer structure composed of functional groups of epoxy, carbonyl, hydroxyl, phenol, etc. results the high reactivity of GO [287][288]. The functional groups produce modified cement-based materials, by easily absorbing free radicals of water from cement by concluding high interaction with the cement matrix [292]. Hence, the research paper aims to investigate the effectiveness of using the GO pretreatment method on CR in enhancing the mechanical properties of rubber-mixed concrete, especially for high-strength structural applications.

5.2 Materials and experimental program

5.2.1 GO treatment on CR

A range of GO concentrations relative to the literature, 0.5, 1.0, 2.0, and 3.0 mg/ml concentrations were used for the pretreatment of crumb rubber. Diverse GO concentration solutions were prepared using highly dispersed graphene oxide. In preparation for GO samples, X-ray powder diffraction (XRD), RAMAN analysis, and Fourier Transformed Infrared Spectroscopy (FIIR) were tested by the producer (refer to Annex 3). The element composition obtained from X-ray photoelectron spectroscopic (XPS) analysis of GO is elaborated in Table 5.1.

Table 5.1. The element composition obtained from XPS analysis of GO

Element	Oxygen	Carbon	Sulphur	Chloride
Concentration (%)	30.62	67.30	1.63	0.44

To identify the effective time of pretreatment, pH testing was conducted for all GO concentrations separately. Then the CR was immersed in GO solutions for two hours of time which was the effective time of pretreatment obtained from pH testing (see 5.3.1). The treated CR samples were poured into a tray by spreading out as represented in Figure 5.1(a) and left for water to be evaporated by means of air dry (see Figure 5.1(b)). The GO-deposited CR particles were examined in Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM) tests as shown in Figure 5.7. Since the study is focused on obtaining the maximum effective results by using GO, three series of treated crumb rubber with graphene oxide (CR-GO) were introduced. Implementation of CR-GO after the solvent is fully airdried (S3), implementation of CR-GO only after 2 hours without the remaining GO solution (S4), implementation of CR-GO only after 2 hours with the remaining GO solution by replacing water amount (S5).

(a) (b)

Figure 5.1. (a) Air drying the treated CR, (b) air-dried CR

5.2.2 Analytical tests

The reaction of GO and CR needs to be analyzed to identify the behavior of the two constituents comprehensively. Hence, pH testing was carried out to identify the optimum settlement time where the pH value become constant. Measurements were taken of five samples, four CR-GO mixed concentrations, and distilled water mixed CR for a better comparison, as represented in Figure 5.2. Ion chemometric (IC) test was performed to identify the ion concentration of the treated CR-GO mixture with respect to as received GO samples and distilled water mixed CR. Further, the test was crucial to separate the reaction between solvent and CR material. Figure 5.3(a) represents the filtering of the solutions, (b)filtered samples and (c)conducting the IC test.

(a) (b)

Figure 5.2. pH testing on GO concentrations

The variation in the functional group of GO-treated CR was checked by the FTIR. As received CR, four concentrations of GO-CR, and acetic acid-treated CR were tested to compare the functional behavior and variation in structure due to acidic treatment by using the Perkin Elmer FTIR spectrometer set up at 400 cm^{-1} to 4000 cm^{-1} infrared spectrum range. To identify the physical evidence of changes that appeared on the CR surface with GO treatment, SEM has been performed on selected samples. The samples were coated with a gold sputter for better imaging.

(a) (b) (c)

Figure 5.3. (a) Filtration of the solutions, (b) filtered samples and (c) conducting the IC test

5.3 Results and discussion

5.3.1 pH test

The prepared GO solutions were identified to be acidic, hence the reaction with CR is crucial to analyze the material and functional behaviors. Further. pH test is essential to identify the minimum time required for the settling of the pH value of CR-GO solution. Figure 5.4 depicts the variation of pH value with 15 min time intervals and optimum pH values obtained.

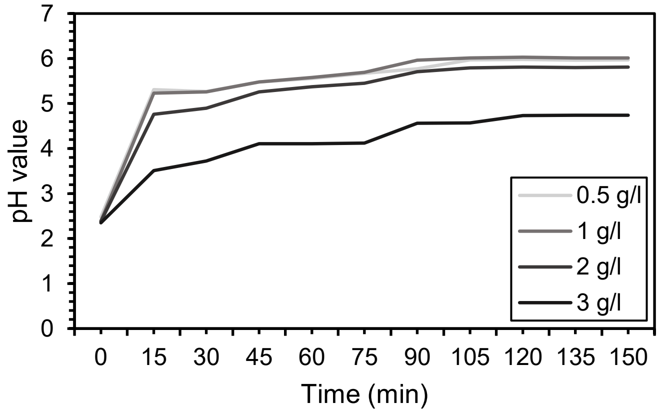


Figure 5.4. pH value variation of different GO concertation with time

The pH test results revealed that the addition of CR to GO solution increases the pH value from the high acidic to neutral range. All four GO concentrations of 0.5, 1, 2, and 3 g/l settle their pH value at 5.96, 6.01, 5.8, and 4.74 respectively. Importantly, it is identified that a minimum of 2 hours is necessary to complete the reaction between GO and CR, hence it is decided that the minimum pretreatment time is 2 hours. With the significant variation in pH, it is identified to have an obvious reaction between CR and GO. Therefore, identifying the chemical behavior is crucial.

5.3.2 Ion chemometric (IC) test

As a result of a definite chemical reaction, GO solution, GO treated CR solution, and distilled water mixed CR solution were compared to identify ion concentrations of solvents. Moreover, the IC test is crucial to identify and separate the chemical reactions among materials and solvents. Table 5.2 represents the ion concentrations of three solutions.

Table 5.2. Ion concentration of as received CR, CR-GO solution, and DI mixed CR

Component name	Concentration (ppm)		
	As received GO solution	CR-GO solution	DI-CR solution
Floride	0.023	-	0.026
Chloride	0.823	0.014	0.133
Bromide	0.047	-	0.041
Sulfate	1.129	0.191	0.212

The three solutions result in the same ion components of fluoride, chloride, bromide, and sulfate in different concentration levels. Comparing the distilled water mixed with CR solution with GO solution does not represent a significant variation in concentration levels. Hence, it is understood that there is no possible reaction between the solvent used to dilute GO and CR.

However, the CR-GO solution compared to the GO solution showed a significant reduction in every ion component. Since there is no reaction between the solvents, the momentous reduction in ion concentration is due to the GO reaction with CR material. This is due to the GO being acidic and activating the rubber polymer, hence the reduction in ion concentrations is due to the reaction of CR material with the GO.

5.3.3 FTIR of GO-CR

The variation of functional groups of CR due to the GO treatment is crucial to understand the structural changes as well as the effect of different concentrations. Figure 5.5 reveals the interaction of GO with CR. Further, to compare the effect of acid with CR, acetic acid treatment was analyzed.

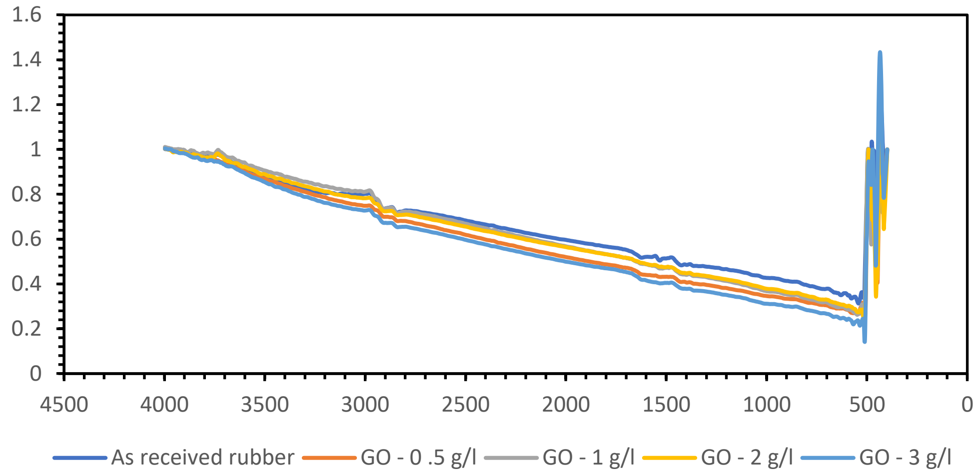


Figure 5.5. FTIR test results of GO and CR-GO samples at different GO concentrations

The peaks of spectra revealed that there is a minimum change in the structure of as received CR and CR-GO since the peaks resulting from FTIR of the two components are almost similar. However, a variation in transmittance percentage was identified as the GO concentration level increased. It can be identified that the higher transmittance level is due to the higher GO deposition on the CR surface. It is understood that GO concentration levels have a direct effect on enhancing the CR-cement bonding, as higher GO deposition reveals higher reactivity and hydrophilic nature of the CR-cement matrix.

The specific variation observed from GO treatment and acetic acid treatment is the structural changes that occurred in CR due to acetic acid treatment as shown in Figure 5.6. As most of the studies follow acid as a pretreatment, the variation in CR structure was not discussed. Moreover, GO being an exception of acid treatment, neither the structural changes nor acidic nature is possessed in CR-GO.

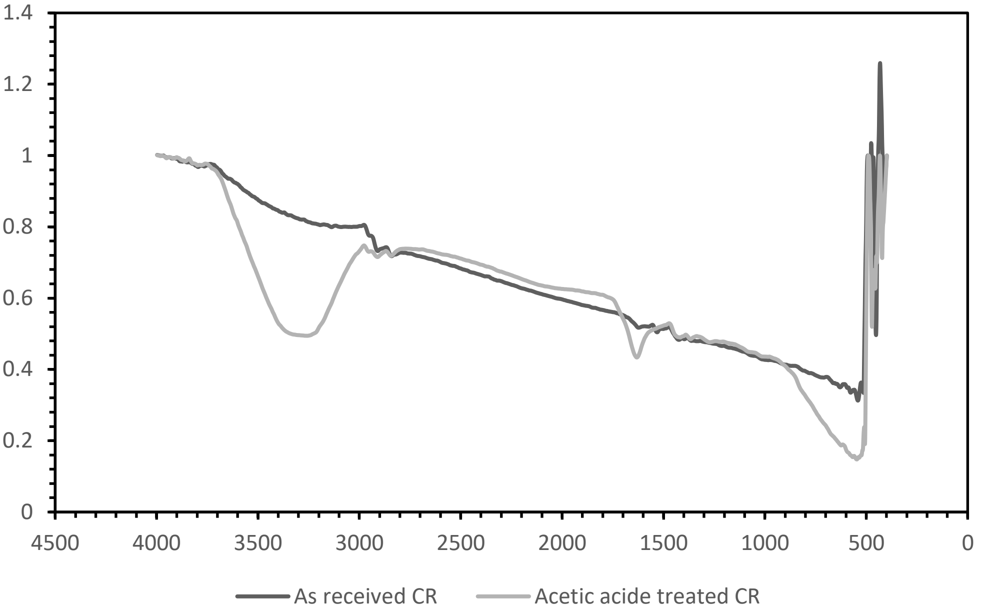


Figure 5.6. FTIR test results of as-received CR and acetic acid-treated CR

5.3.4 SEM imaging

Figure 5.7 (a), (b), (c), and (d) represent the received CR and samples treated with different GO concentrations respectively. By revealing physical evidence of GO deposition on CR surfaces as depicted in (b), (c), and (d), wrinkle-bright areas and flaky areas are known to be GO particles implanted on CR surfaces [221], [307], [308]. Further, the sharp contrast and flaky areas to the non-reflective surface reveled the deposition of GO nanoparticles around the CR surface.

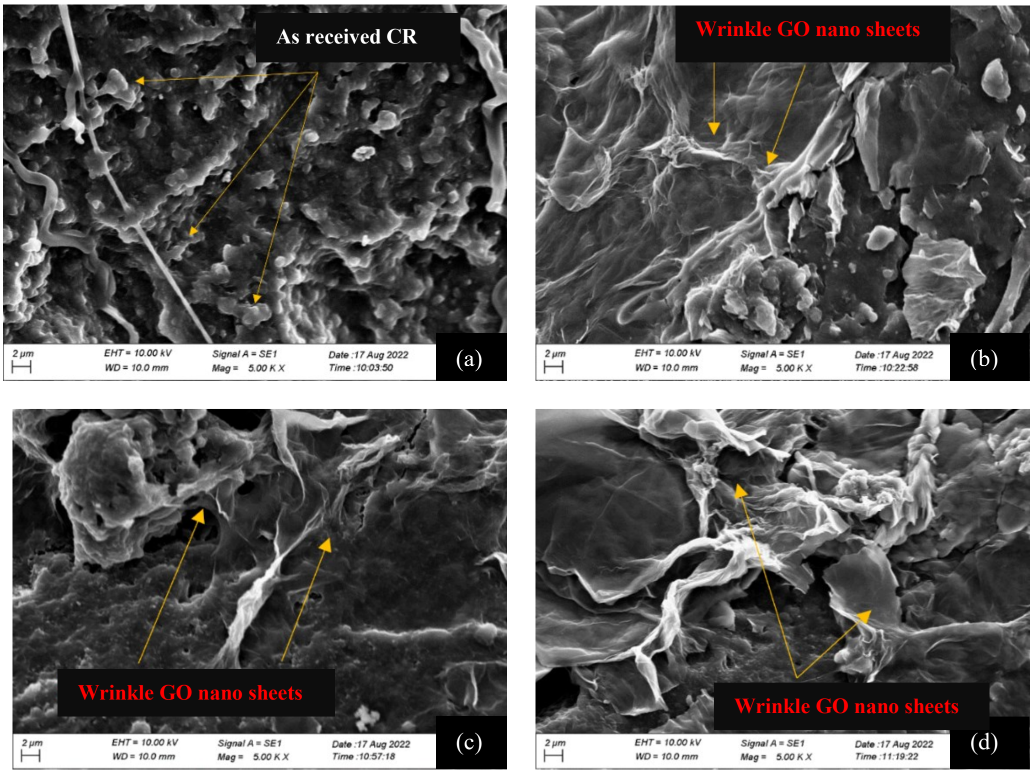


Figure 5.7. SEM images of (a) as received CR (b) CR-GO treated by 0.5 g/l GO (d) CR-GO treated by 2 g/l GO and (c) CR-GO treated by 3 g/l GO

By comparing (b), (c), and (d), the density of wrinkle and flaky areas has been enhanced with the increment of GO concentrations. This implies that the pretreating with higher GO concentrations results higher GO deposition on the CR surface, hence enhancing the bonding between the CR and cement to increase the performance of the concrete composite.

5.4 Summary

The effect of CR-GO with different concentrations with respect to different pretreatment methods on the compressive strength was experimentally investigated. In addition, the material behavior of CR with GO pretreatment was comprehensively investigated in this chapter. The findings of this research grant are summarized below.

- Graphene oxide produces strong adhesion between polymer membranes due to the high Van der Waals forces, hence can be used as a pretreatment method for crumb rubber to improve the mechanical strength of rubberized concrete.
- The acidic nature of graphene oxide is diminished by reacting with crumb rubber. It takes a minimum of two hours to settle the pH value, whereas the minimum time of pretreatment is two hours.

- There is no reaction between the solvents, whereas the graphene oxide directly reacts with crumb rubber and neutralizes the solution. Graphene oxide creates no structural changes to crumb rubber and creates no changes to the structure, unlike other acid treatments.
- Pretreatment cause the deposition of GO around CR and enrich the density of deposition of GO with the increment of using higher GO concentrations.

CHAPTER 6 MECHANICAL PROPERTIES OF GRAPHENE OXIDE TREATED RUBBERIZED CONCRETE – PHASE 3

6.1 Introduction

The partial replacement of rubber in concrete creates poor mechanical behavior. Instead of several improvements in concrete properties, such as abrasion resistance, impact resistance, energy dissipation, chloride ion penetration, resistance to fatigue, damping ratio, elasticity, etc., the mechanical properties as compressive strength, tensile strength, and flexural strength shows a declined trend in RuC.

Research studies have identified several reasons that accompany the poor mechanical behavior of RuC. High roughness of rubber particles, poor adhesion between rubber aggregates, poor bonding behavior of rubber and cement, weak interfacial transition zone due to the hydrophobic nature of rubber, and non-uniform dispersion of rubber die to same particle size and shape have been identified as the prominent reasons. Hence, there is a requirement to address the identified issue. This study has introduced a high reactive nano material as a pretreatment method to strengthen the bonding properties of rubber aggregates and concrete constituents. As the study requires physical verifications to identify the effect of GO pretreatment, the compressive strength test, tensile strength test, and flexural strength test have been conducted on 9 different series.

6.2 Experimental program

6.2.1 Materials

The ordinary Portland cement (OPC) of strength class 42.5 N/mm^2 in accordance with BS EN 197 incorporated with chemical properties of Sulphur trioxide (SO_3) < 2.8%, Chloride <0.05% and LOI ($C_{15}H_{16}N_6OS_2$) < 3.0% was utilized. As a fine aggregate, river sand, and CR were used in respective proportions. Two different CR sizes were incorporated in the range of 0-5 mm (fine aggregate) and 5-10 mm (coarse aggregate) as shown in Figure 6.1 and the physical properties of CR are represented in Table 6.1. The CR density, water absorption, flakiness index, and bulk density were gained with respect to Annex C of BS EN 1097-6 (lightweight aggregate) [222], BS EN 933-3 [223], and BS EN 1097-3 [224]. Commercial high-range water-reducing admixture and silica fumes were added as an additive to the rubber-added concrete matrix.

Figure 6.1. As received CR (a) 0-5 mm (b) 5-10 mm

Table 6.1. Physical properties of CR

Property	Rubber (0-5 mm)	Rubber (5-10 mm)	Rubber (10-20 mm)
Apparent density (g/m^3)	-	933.1	1105.8
Oven dry density (g/m^3)	-	935.07	1120.65
SSD density (g/m^3)	-	967.1	1050.4
Water absorption (%)	-	6.18	1.25
Specific gravity	-	1.10	1.10
Loose bulk density (g/ml)	0.38	0.42	0.47
Rodded bulk density (g/ml)	0.47	0.54	0.62

6.2.2 Mixing and testing procedure

As the previous literature [309] indicated that the replacement of rubber could lead to poor concrete behaviors including mechanical properties, a high percentage of segregation, and poor cohesion, hence, the study focused on an optimized concrete mix outlined by Raffoul et al. [33]. The mix was designed to be high-strength concrete with high cement content to water ratio (w/b = 0.31). The rubber was replaced by 20% of aggregate by weight considering two categories, 0-5 mm and 5-10 mm since non-homogenous rubber replacement show enhanced RuC properties over homogenous rubber replacement [310]. Table 6.2 summarizes the rubber and mineral aggregate proportions, and mix design with the pretreatment method used in this study.

The concrete constituents were mixed in the following procedure.

1. The aggregates (both mineral and CR) were dry-mixed for 30 s, with respect to the pretreatment series.
2. Half of the mixing water was added and mixed for another minute (the remaining GO solvent was mixed with water and added).
3. The mix was kept still for three minutes.
4. The binder materials and the remaining water was added followed by the gradual addition of the admixtures.
5. The mixture was mixed for another three minutes. The 100 mm cubes were cast in two layers and vibrated using a vibration table. After 24 hours in laboratory condition, the specimens were cured in a curing tank for 28 days.

Figure 6.2. Preparation of sample cubes

With reference to the specifications provided by BS EN 12390-3, the compressive strength was determined by using three samples of 100 mm cubes in each mix at 7, 14, and 28 days. The tensile strength was conducted under specification to the code ASTM C496/C496 -04. 18 cylinders of length 300 mm and diameter 150 mm were cast with reference to 03 series and tested at 7 and 28 days.

Figure 6.3. Preparation and testing of splitting tensile cylinders

Table 6.2. GO-treated RuC mix proportioning and quantities of materials used

Type	GOC (mg/ml)	CR-GO (%)	Materials (kg/m^3)									
			CR (0-5 mm)	CR (5-10 mm)	Mass of CA (5-10 mm)	Mass of CA (10-20 mm)	Mass of FA	Water	Remaining GO solution	Cement	SF	SP
Control (S1)	-	-	-	-	355	848	666	140	-	450	27	9
Non-treated rubber only (S2)	-	Plain CR	66.6	35.5	319.5	848	599.4	140	-	450	27	9
S3-1	0.5	10F&10C	66.6	35.5	319.5	848	599.4	140	-	450	27	9
S3-2	1	10F&10C	66.6	35.5	319.5	848	599.4	140	-	450	27	9

S3-3	2	10F&10C	66.6	35.5	319.5	848	599.4	140	-	450	27	9
S3-4	3	10F&10C	66.6	35.5	319.5	848	599.4	140	-	450	27	9
S4	2	10F&10C	66.6	35.5	319.5	848	599.4	140	-	450	27	9
S5	2	10F&10C	66.6	35.5	319.5	848	599.4	63	77	450	27	9

Since the study is focusing on identifying the most effective way of GO pretreatment (refer to 2.2), the efficiency of GO reacted with CR should be measured. As a result of that, a mass analysis was conducted for the 2 hours treated GO-CR and for the remaining solution of it. In the process, the following sequence was followed and shown in Figure 6.2.

1. A rubber was selected and sieved through the 0.075 mm sieve size by repeatedly washing with distilled water until no rubber fine particles pass through the sieve.
2. The remained rubber particles were oven dried to evaporate the water and measured the weight of the dried sample was.
3. The dried rubber sample was treated with respective GO amount by the means of a shaker for 2 hours.
4. The treated CR with GO was sieved through the 0.075 mm sieve and washed with distilled water to make sure only treated CR particles remains in the sieve.
5. Before the passed GO solution was filtered, the filter paper was oven-dried and taken measurements of it.
6. The passed GO solution was filtered using oven-dried filter paper to extract the GO particles from the solution.
7. The filter paper with GO particles was oven dried to measure the amount of GO extracted from the remaining solution.

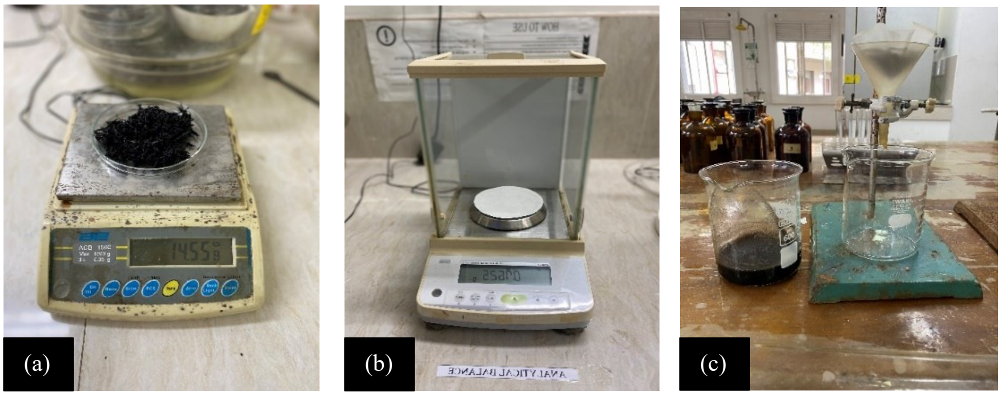


Figure 6.4. Process of GO mass analysis after pretreatment (a) measuring the oven-dried rubber sample, (b) measuring the oven-dried filter paper, and (c) extraction of GO by filtering

6.3 Results and discussion

6.3.1 Workability and dry density

Good working performance is proportional to the workability of the concrete mix. Since the developed mix design incorporated rubber and GO, the workability of the matrix is a concern. The water absorption of rubber was found to be higher (refer to Table 6.1) in finer rubber particles due to the higher surface area. Further, the implementation of GO reduces the free water content of cement slurry [311].GO is identified to absorb a high percentage of water, because of its hydrophilic nature and higher surface area [294]. Hence, GO incorporated RuC should result poor workability measurements.

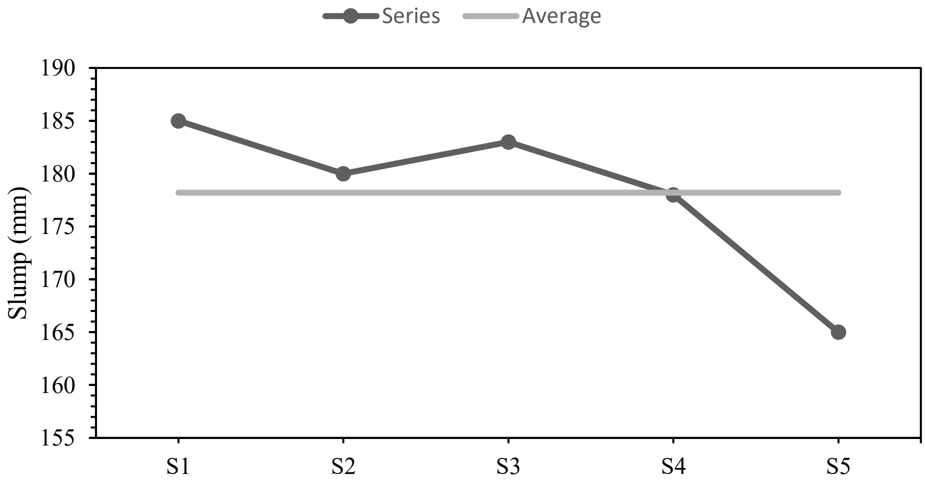


Figure 6.5. The slump of concrete series

Figure 6.3 depicts that the workability of every series has been diminished compared to the control sample, hence the results are in line with the aforementioned behavior of CR and GO implementation to the concrete. However, the workability results are in the range of normal workable concrete [312], with only 1.08%, 3.78%, and 10.81% reduced in respective S3, S4, and S5 series compared to S1. The enhancement of workability results due to the implementation of a naphthalene-based superplasticizer and incorporation of silica fume [313], which emphasize that the incorporation of superplasticizer and silica fume is crucial to avoid the loss of workability in the GO-incorporated RuC concrete matrix.

Figure 6.4 represents the dry density variation of the series, and the use of CR to concrete reduces the density due to the low mass of rubber with respect to the mineral aggregate. A significant reduction of 5.40%, 5.28%, and 5.07% in density was observed in every series compared to control samples. However, the densities are around the value quoted by AS 3600:2018 [314]. A marginal increment resulted from the inclusion of GO, as it is in line with previous literature [315][316].

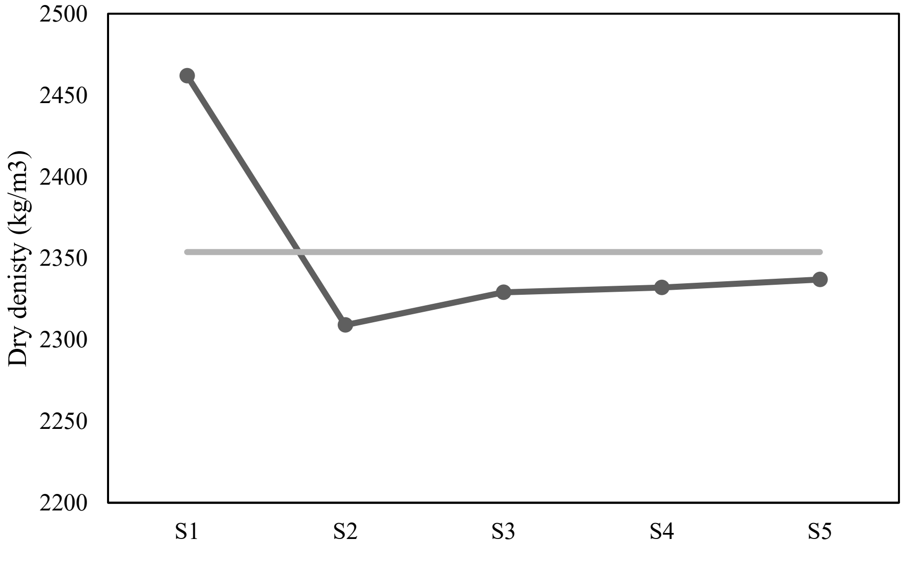


Figure 6.6. The dry density of concrete series

6.3.2 Compressive strength

The compressive strength laid as the base for investigating the effect of GO treatment on the mechanical properties of RuC. In this regard, 9 series were investigated to identify the most suitable pretreatment method and the most efficient concentration of

GO. It is critical to identify an efficient way of implementing GO to CR since the cost of GO is a concern in the long run. Figure 6.5 depicts the compressive strength results of three series. As desired, the compressive strength has been increased in all three methods with the implementation of GO. With respect to the non-treated rubber-only mixture (S2), 21.17%, 43.47%, and 88.18% enhancements were observed in S1, S2, and S3 series respectively. The highest compressive strength resulted in the full evaporation of the GO solution, with the deposition of GO nanosheet around the CR surface. It is understood that the implementation of 2 hours of treated CR-GO is not efficient as the maximum deposition of GO is not experienced by having the remaining solvent in the mixture. It is found that 31.10% reduction in compressive strength with respect to the S3 series. Hence, the study was focused on using the remaining solvent as a replacement for water, as to optimize the use of GO solution in the concrete mix. However, the compressive strength of the S5 series is reduced by 55.23% compared to S3. Therefore, the maximum deposition of GO nanosheet resulted in the fully air-dried condition, hence identified to be the most efficient method.

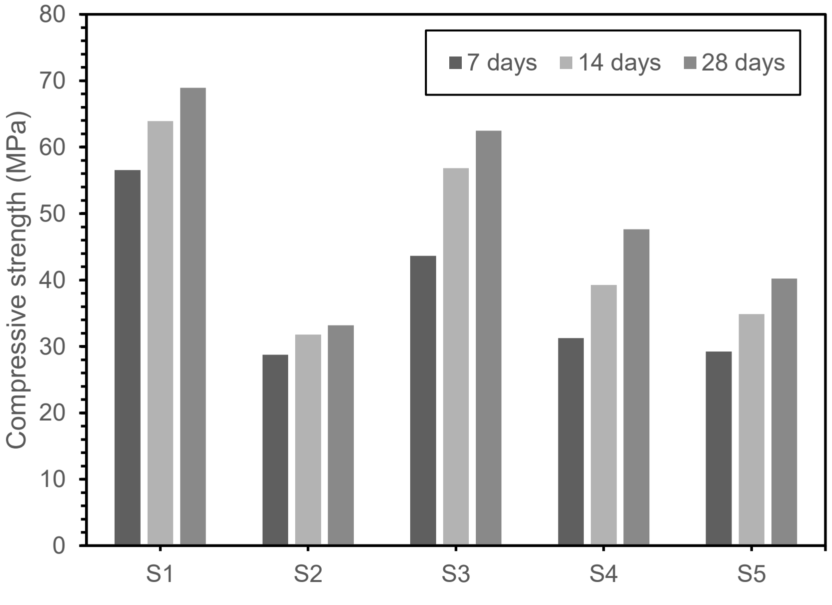


Figure 6.7. Compressive strength of control sample, non-treated and treated GO-RuC

With respective the most efficient pretreatment method (S3), the compressive strength varying with respective GO concentrations is represented in Figure 6.6. Although there was a notable increase in compressive strength as the GO concentration increases, the S3-3 mixture with 2 g/l GO concentration shows the highest compressive strength over other GO concentrations. With respect to the S2 series, 37.38%, 61.02%, 94.47%, and

58.34% were observed in S3-1 to S3-4 respectively. However, a marginal reduction of 6.35% was observed in the S3-3 series compared to the control sample.

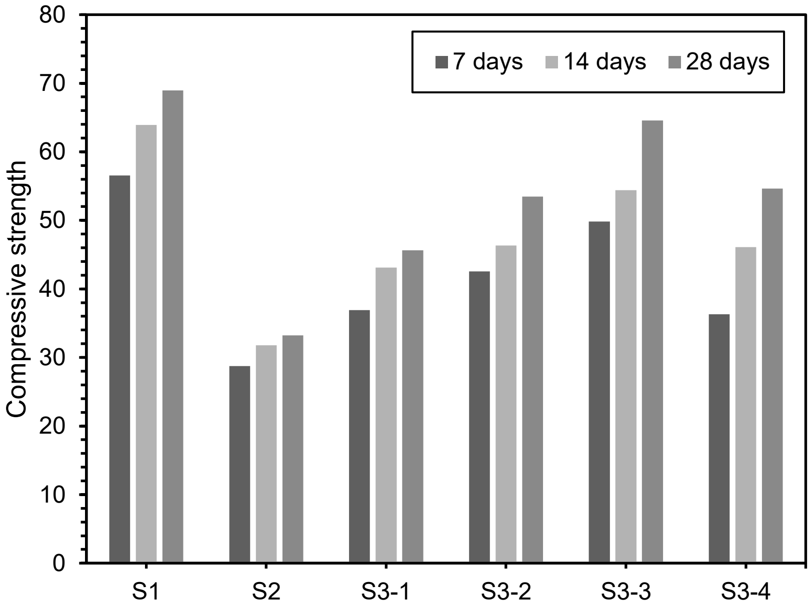


Figure 6.8. Compressive strength of control sample, non-treated and treated with various GO concentrations RuC

It can be seen that the GO concentration is not directly proportional to the compressive strength of RuC. Hence, it reduces the compressive strength by increasing GO concentration from 2 g/l to 3 g/l. Further, exceeding the GO concentration will not only reduce the concrete performance but effect the concrete in the opposite way [317]. It is not the amount of GO that enhances the performance, but the excellent dispersion is crucial for better performance of concrete. Moreover, higher GO will agglomerate in cement paste, due to the higher reactivity with Ca^{+2} [318]. This will create a cluster in the concrete matrix which results worst boding behavior with cement paste. Ultimately poor concrete properties will experience due to high GO concentration.

6.3.3 Tensile strength

The tensile strength was identified as a prominent parameter in waste rubber-incorporated concrete. Hence, the sudden failure of conventional concrete has been diminished with the incorporation of rubber. Further, the stress-strain behavior of RuC is crucial to measure. The study assembled to 03 series with reference to the results obtained from compressive strength. Since, the splitting tensile strength was measured for S1, S2, and S3-3. Testing was carried out for 7 days and 28 days.

As expected, the rubber incorporation has reduced the tensile strength as well. However, the GO pretreatment has enhanced the tensile strength with respect to non-treated RuC specimens. Figure 6.7 represents the variation of splitting tensile strength into 7 days and 28 days. With respect to the control sample, GO-treated and non-treated RuC samples show 43% and 22% strength reduction respectively. Hence, the reduction is obvious and in line with the pattern of compressive strength variation. But a significant improvement was shown by the GO-treated RuC samples as it enhances the splitting tensile strength by 36% with respect to non-treated RuC.

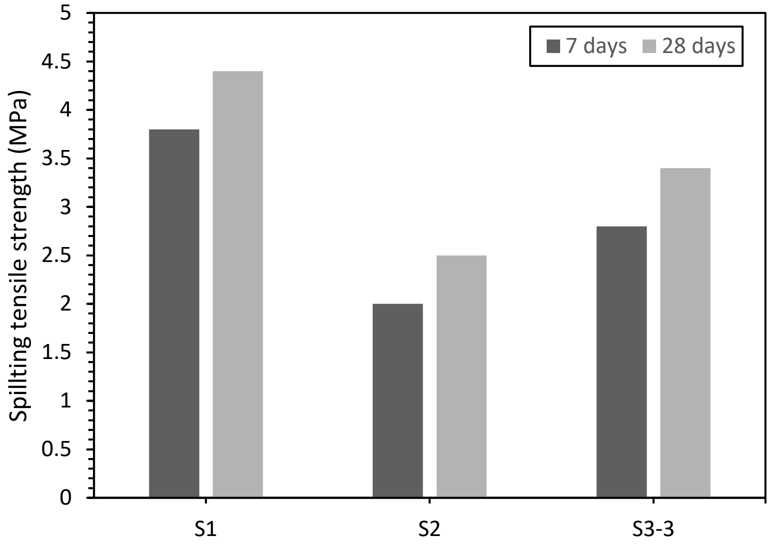


Figure 6.9. Splitting tensile strength

The enhancement of tensile capacity is due to the possible improvement in bonding between CR and cement paste, as a result of enhanced reactivity in the CR surface. Hence, the improved bonding behavior results in high-stress transfer at the interfaces which raised the tensile capacity of GO-treated RuC. In addition to surface treatment, GO particles can act as fillers and densify the concrete matrix. However, the effect is unreliable.

6.4 Summary

- Water absorption is increased with the graphene oxide treatment; however good working performance resulted only in a 10% deviation in slump value with respect to the control mixture.

- The dry density reduces significantly with the inclusion of crumb rubber aggregates. But the dry density increases with the inclusion of graphene oxide and enhances with higher concentration.
- The air-dried pretreated crumb rubber was identified to be the most efficient and effective method of pretreatment, because of the highest graphene precipitation on crumb rubber. Two hours of pretreatment shows less than 50% precipitation of graphene oxide, while more than 50% of graphene oxide remains in the solution.
- Concrete with air-dried graphene oxide treated crumb rubber capable of achieving high strength, resulting in only a 9.42% reduction with respect to the control mixture. In addition, a significant enhancement of 88.18% was obtained over non-treated crumb rubber concrete.
- The compressive strength increases with a high concentration of graphene oxide, but a graphene oxide concentration of 2 g/l is identified to be optimum as exceeding the optimum concentration reduces the compressive strength. The poor behavior of compressive strength is due to the agglomeration of graphene oxide. Hence, excellent dispersion and the appropriate amount of graphene oxide are crucial for the enhancement of concrete strength.
- Rubber incorporation reduced the split tensile strength of concrete. However, the GO treatment significantly reduced the strength loss. A significant enhancement of 36% resulted from GO treatment compared to non-treated RuC samples.

CHAPTER 7 BONDING STRUCTURE AND MORPHOLOGY – PHASE 3

7.1 Introduction

The major concern for the reduction of strength in RuC is the poor bonding behavior between CR and the cement matrix. The incorporation of GO as a pretreatment method to CR has improved the strength parameters (refer to 6.3.2 and 6.3.3) with respect to non-treated RuC. Hence, it is crucial to identify the changes that occurred to the bonding of CR and cement interfaces through physical evidence.

The study by Noor Azlin et al. [319] concluded that less ITZ has resulted from NaoH and metakaolin treatment to CR. Further, low pressure of capillary suction in the concrete was ensured with better binder agglomeration. In addition, a poorly packed concrete matrix results poor strengthening. Especially, incorporating a lightweight aggregate, CR, into the concrete matrix requires special attention. Hence, the bonding between CR and the cement matrix and the density of concrete constituents should be evaluated by physical imaging.

The literature highlighted the enhanced nature of ductility with the incorporation of rubber [170][4]. In addition, the nature of the failure is distinct with respect to the failure of conventional concrete. RuC avoids sudden failures and creates a high percentage of micro cracks [320]. With respect to the specific characteristic, unit mass analysis GO pretreatment, SEM imaging, and crack patterns were observed and investigated.

7.2 Experimental program

The variation of strength occurred with respect to different pretreatment types. Hence, it is required to identify the effect of GO with respect to pretreatment methods. As is highlighted, the concentration of GO precipitated on the CR surface is the major reason for improving the strength of concrete. Therefore, the amount of GO needs to be analyzed to identify the effect of GO comprehensively and the percentage precipitated on the GO surface. Hence, the following sequence was followed to conduct the unit mass analysis.

1. A rubber was selected and sieved through the 0.075 mm sieve size by repeatedly washing with distilled water until no rubber fine particles pass through the sieve.
2. The remained rubber particles were oven dried to evaporate the water and measured the weight of the dried sample was.
3. The dried rubber sample was treated with respective GO amount by the means of a shaker for 2 hours.
4. The treated CR with GO was sieved through the 0.075 mm sieve and washed with distilled water to make sure only treated CR particles remains in the sieve.
5. Before the passed GO solution was filtered, the filter paper was oven-dried and taken measurements of it.
6. The passed GO solution was filtered using oven-dried filter paper to extract the GO particles from the solution.
7. The filter paper with GO particles was oven dried to measure to identify the amount of GO extracted from the remaining solution.

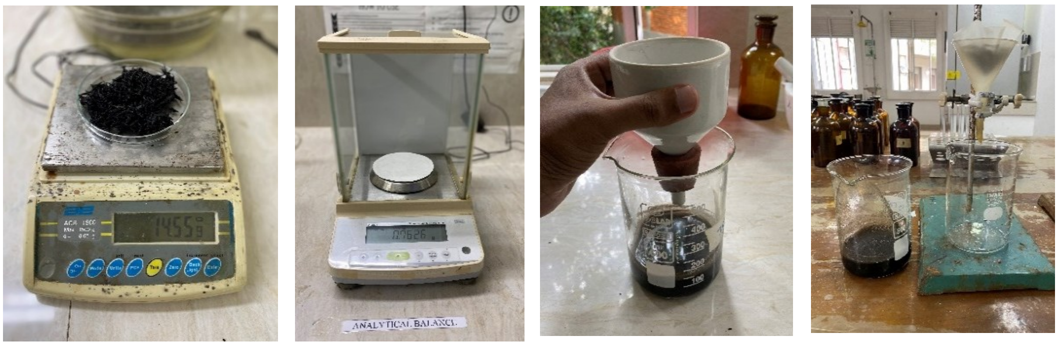


Figure 7.1. Conducting unit mass analysis for pretreated CR

The structural variation of occurred in rubber and cement matrix has to be investigated to identify the bonding behavior in the concrete matrix. Hence, the SEM imaging was performed on selected control samples, treated and non-treated RuC samples.

7.3 Results and discussion

7.3.1 Mass analysis of GO pretreatment

The significant variation between the air-dried pretreatment method and the other two pretreatment methods needs to be analyzed. The remaining solution after 2 hours of pretreatment, an emphasis that the full amount GO has not precipitated or reacted with CR with respect to the compressive results. Therefore, it is crucial to identify the GO amount that reacted with CR and precipitated on the CR surface. Table 6.3 represents the weights at each condition of the test.

Table 7.1. Weight at each condition of mass analysis

	Weight (grams)
The weight of the oven-dried CR sample	15
The weight of GO used to pretreat the CR sample	0.06
The weight of oven-dried filter paper	0.7625
The weight of oven-dried filter paper with extracted GO	0.7986
The weight of GO extracted on filter paper	0.0361
The weight of GO reacted with CR after 2 hours of treatment	0.0239

The concentration of the GO solution used was 2 g/l, hence identifying the mass of GO dissolved in the respective amount used for the analysis. It is understood by the results that the remaining solution of GO after pretreatment time contains GO with it, further washing with distilled water removes the GO which is not reacted with CR and precipitated well. It resulted that only 39.83% precipitated on CR while 60.16% remains in the solution. By this analysis, the reduction of compressive strength in the S4 and S5 series can be concluded. Hence, the significant enhancement in the S3 series is due to the higher percentage of GO precipitation with the air-dried method used and identified to be the most efficient method.

7.3.2 SEM imaging

It is identified that the strength parameters, compressive strength, and tensile strength have been enhanced by the GO pretreatment with respect to non-treated RuC. Hence, it is crucial to observe the changes that occurred in the bonding of CR and cement interface.

Figure 7.1 (a) represents the interface bonding of the non-treated CR surface and cement surface, which implies a clear separation between the two surfaces, hence the poor adhesion. In addition, Figure 7.1 (b) provides the improved adhesion between the rubber surface and the cement interface. As the study anticipated, GO pretreatment on rubber has improved the interfacial boding between CR and cement matrix, which is carried out for better permeance in strength.

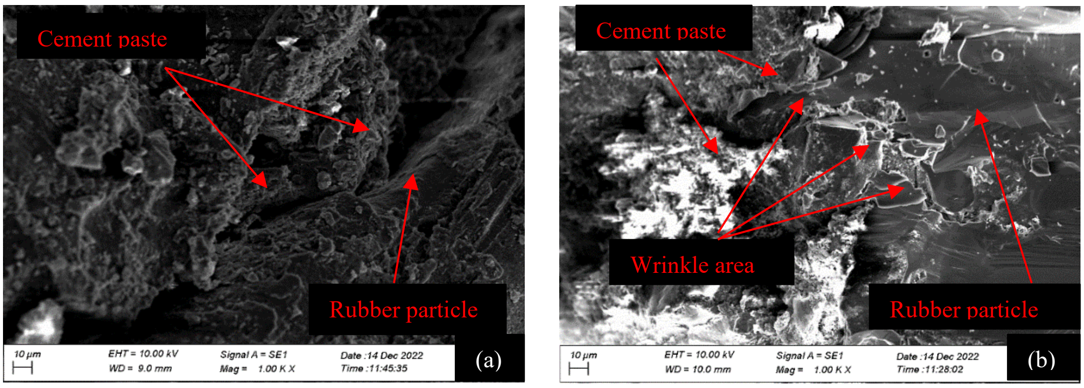


Figure 7.2. SEM images of interfacial surfaces of (a) non-treated CR and cement matrix (b) treated CR and cement matrix

7.3.3 The crack pattern of concrete specimens

The behavior of crack widths and propagation is significant in RuC compared to conventional concrete. As most studies incorporated rubber, observed and concluded the propagation of micro-cracks which is contrasting to conventional concrete cracks [319][320].

Figures 7.2, 7.3, and 7.4 represents the crack propagations of control samples, and non-treated and treated RuC samples respectively. Hence, it is observed that the samples with rubber incorporation propagate micro-cracks while the control sample with a major crack. Further, the high-strength concrete fails as a blast and it is conventional in high-strength concrete. However, the rubber mixed samples have not shown a such type of blast failure, but with a high percentage of micro cracks.

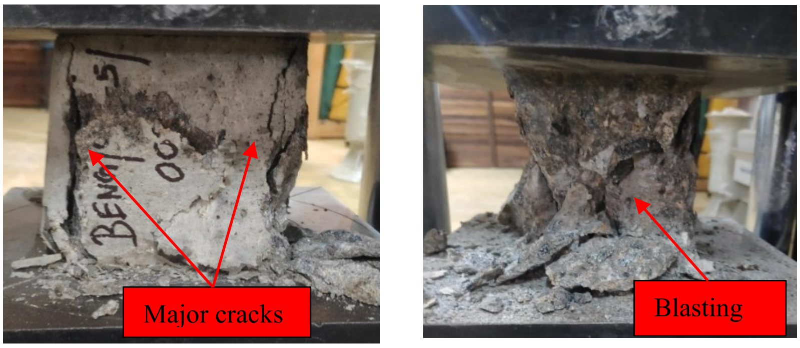


Figure 7.3. Major cracks and blasting in control sample

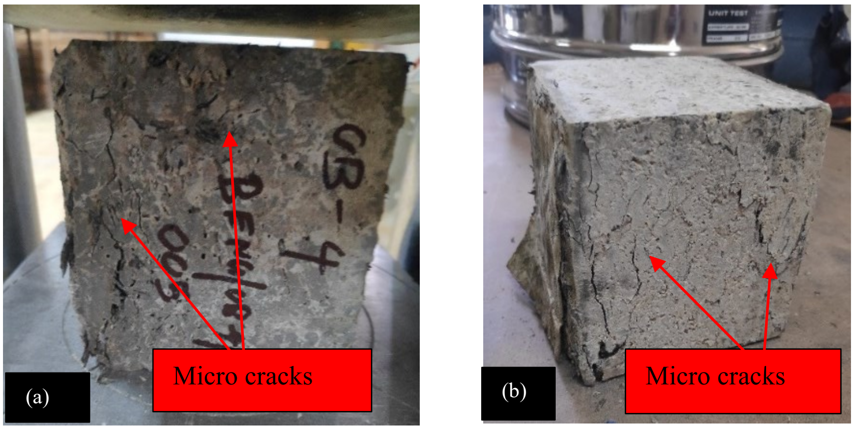


Figure 7.4. Micro crack propagation of non-treated RuC

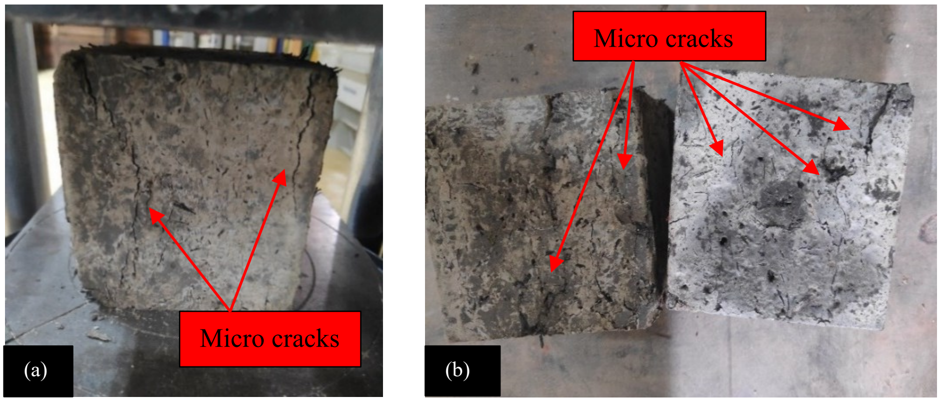


Figure 7.5. Micro crack propagation of treated RuC

With respect to the crack patterns, the ductility of RuC has improved compared to conventional concrete. Hence, the RuC possesses a larger deformation ability under equal loading conditions. In addition, RuC enhances energy dissipation. With reference to the crack width sizes, the micro-cracks help to understand the structural disorder with specific and uniform cracks. Hence, the crack behavior of RuC is beneficial in forecasting element failures.

7.4 Summary

The effect of pretreatment methods was investigated by identifying the percentage of GO precipitation. With respect to the strength variation that occurred, the investigation of CR and cement matrix bonding was carried out through SEM imaging. In addition, the propagation of cracks on the concrete surface was observed. The findings of those parameters are concluded as follows.

- Only 40% of GO will precipitate on the CR surface after 2 hours of GO treatment. Whilst the reduction of strength occurs due to the less percentage of GO precipitation.
- A clear separation between CR and cement matrix was observed in non-treated RuC samples as it shows poor strength respective to treated RuC.
- An improved bonding behavior has resulted between CR and cement matrix, hence the reduction of ITZ resulted in GO-treated RuC specimens.
- The blasting and major crack propagation of high-strength concrete have reduced with the CR incorporation; hence CR enhances the toughness and ductility of concrete.
- Propagation of definitive crack patterns on concrete surface states RuC as a forecasting mechanism of structural failure

CHAPTER 8 DURABILITY PROPERTIES OF GRAPHENE OXIDE TREATED RUBBERIZED CONCRETE – PHASE 3

8.1 Introduction

It is critical to investigate the durability properties of RuC, hence application of concrete is concentrated on the effect on properties in the longer run. Further, modification to concrete constituents created an impact on concrete durability, and the incorporation of waste substituent, waste tire rubber, enhances the necessity of investigating the durability properties of RuC. However, the durability properties of RuC have not been investigated thoroughly as much as mechanical properties.

The literature has highlighted several reasons for the variation that occurred in the durability properties of RuC. The high percentage of voids created in RuC due to the size and shape of rubber aggregate, the poor development of ITZ, the porous nature of RuC, the high-water absorption percentage of rubber aggregate, the hydrophobic nature of rubber, and the lower density of rubber are significant criteria [14][22][129]. However, the variation in RuC does not provide a pattern with respect to previous studies. In addition, the study has developed a modified concrete mix incorporating a nano material pretreatment, hence the study requires investigating the durability properties. As a result of that, the study has focused to conduct the water penetration and rapid chloride penetration test on RuC.

8.2 Experimental program

8.2.1 Materials

The exact same materials were used to cast the concrete samples to test the durability properties. OPC cement of strength class 42.5 N/mm^2, river sand as fine aggregate (0-5 mm), mineral rocks as coarse aggregate (5-20 mm), and waste tire rubber aggregate was used in proportion in two different sizes (0-5 mm and 5-10 mm) by replacing aggregates. Silica fume and water-reducing admixture were included in the modified mixture. Refer to Table 6.2 for the mix design used and for further details, refer to Section 6.2.1.

8.2.2 Mixing and testing procedure

The rapid chloride ion penetration test was conducted by casting cylinders of 50 mm thickness and 100 mm diameter. A total of 6 cylinders were cast under the specification provided by ASTM C 1202-12 [321]. The test was analyzed by monitoring the amount of electrical current passed through cylindrical samples for 6 hours period. The following steps were followed in conducting the experiment.

1. A total of 6 cylinders were cast by following the exact procedure referred to in section 6.2.1.
2. Specimens were removed after 56 days of curing, blotted off excess water, and transferred to a sealed container. The relative humidity was maintained at greater than 95%.
3. A high-viscosity specimen-cell sealant was mounted hence, the specimen was set onto the screen and applied sealant around the specimen-cell boundary.
4. The exposed surface was covered by plastic sheeting to restrict moisture movement.
5. Specimen mounting was taken place by keeping 100 mm outside diameter by 75 mm inside diameter by 6 mm thick circular vulcanized rubber gasket in each half of the test cell.
6. The top surface containing the negative terminal was filled with 3 % NaCl solution while the positive terminal was filled with 0.3 N NaOH solution.
7. The posts of the apparatus were attached by lead wires and electrical connections were given to the voltage application and data readout apparatus. The power supply was set at 60.0 ± 0.1 V and the initial current reading was recorded.
8. A reading was recorded every 30 minutes for 6 hours. Each half of the test cell was maintained at fill with the appropriate solution for the entire period of the test.

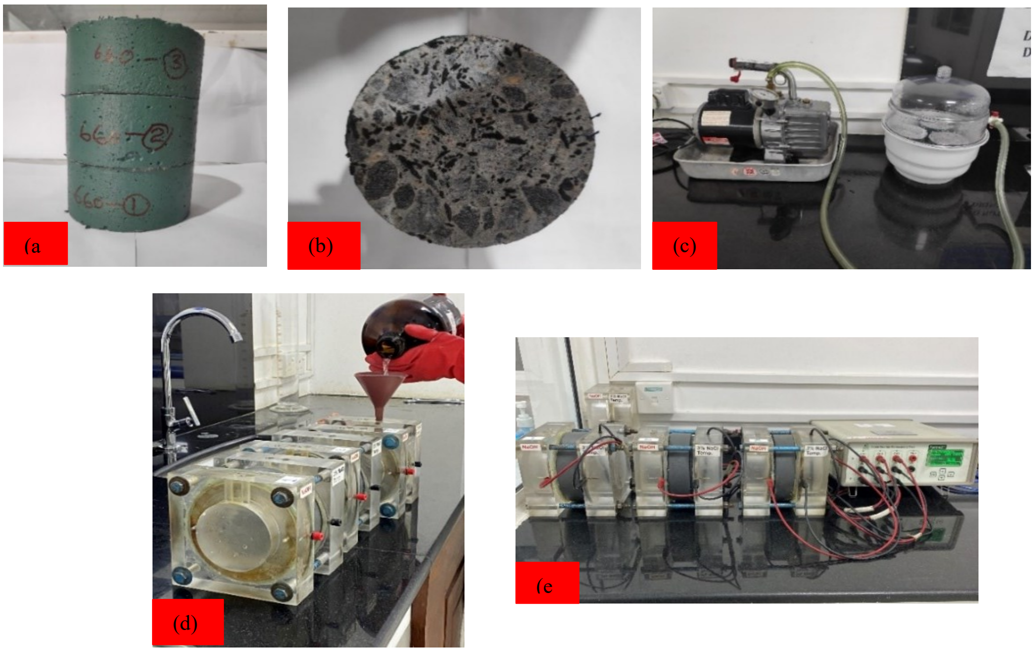


Figure 8.1. Specimen preparation and setting the specimen in the apparatus for rapid chloride penetration test

The depth of water penetration was measured under pressure in hardened concrete which has been water cured by the water penetration test. The test has been conducted with the code BS EN 12390-8 [322]. 6 cubes of 150 mm dimensions were cast with reference to the 03 series. The following steps were followed in the experiment.

1. Immediately after the specimen was de-molded, the surface exposed to water pressure was roughened with the use of a wire brush.
2. The specimen was placed in the apparatus and applied water pressure of (500 ± 50) kPa for 72 hours.
3. The specimen was removed from the apparatus after 28 days old. The excess water was wiped off and a specimen was spilled to half, perpendicularly to the face on which the water pressure was applied.
4. After the splitting surface was dried where the water penetration front can be seen, the depth of water penetration was marked. Hence, the maximum depth of penetration under the test area was recorded to the nearest millimeter.

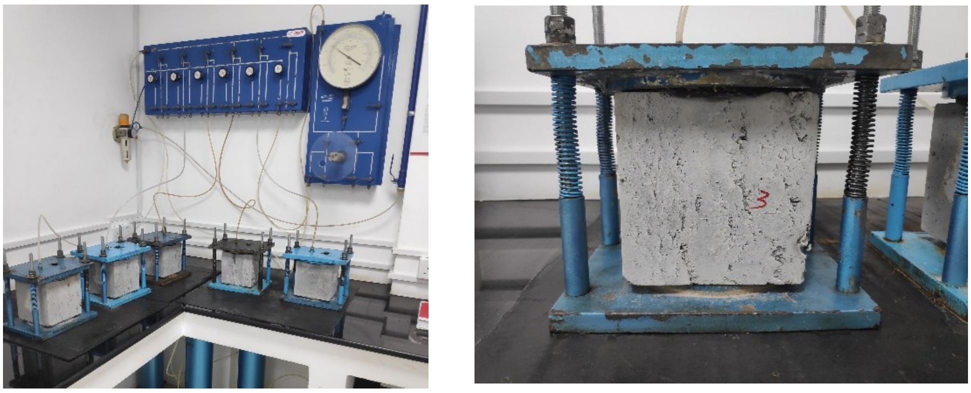


Figure 8.2. Conducting the water penetration test

8.3 Results and discussion

8.3.1 Water penetration test

The water penetration measurements are crucial in the research project, of the incorporation of waste tire rubber aggregates in the concrete mix design. Further, it is essential to investigate, as the water absorption of rubber aggregate is significantly high. Figure 8.3 represents the marked area of water penetration of three samples.

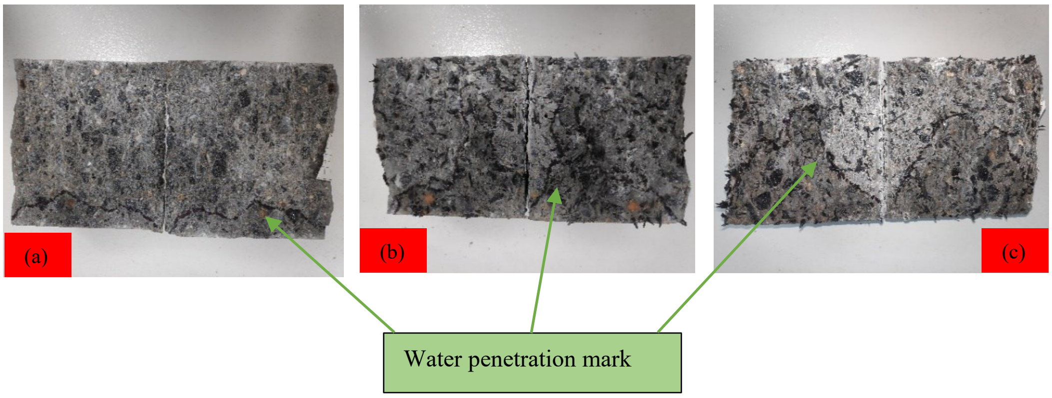


Figure 8.3. Water penetration test results (a) S1 (b) S2 (c) S3-3

As expected, the water penetration of RuC is high compared to the control sample. The peak penetrated depth, represented in Figure 8.3, resulted in 78 mm (average) in the S2 series, whereas the control sample (s1) resulted in a penetration depth of 31 mm. In addition, the series S3-3 observed the highest penetration depth of 106 mm. Comparing the non-treated and treated samples, the incorporation of GO results in more penetration, a 35% increment. This can be due to the result of more water absorption created with GO precipitation, a high percentage of pores, poor compaction of concrete matrix, and higher water absorption of waste rubber and not been investigated thus far.

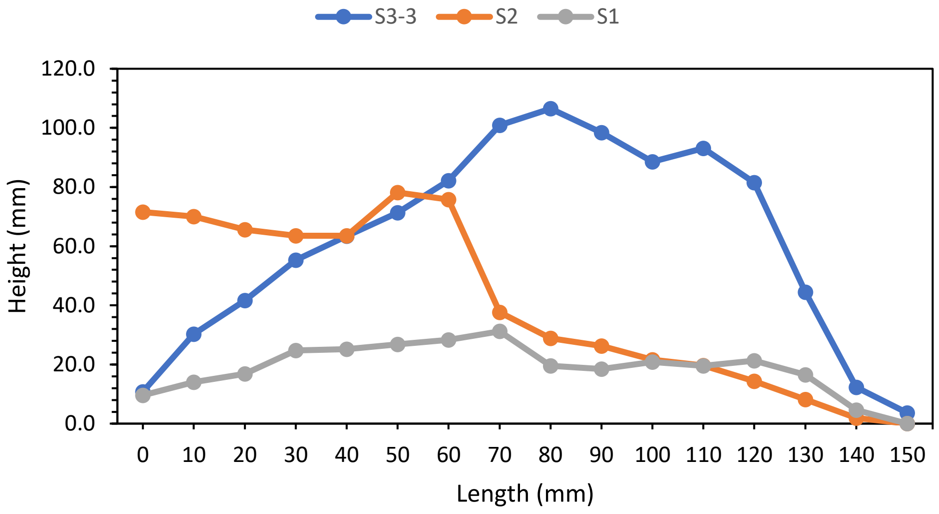


Figure 8.4. Water penetration results of samples

As a result of higher penetration, the application of RuC in structural elements must be investigated. Further, micro-level concrete matrix investigation is required for better understanding.

8.3.2 The rapid chloride penetration test

It is essential to identify the ability to resist chloride penetration in concrete, specifically in reinforced concrete. In addition, the requirement of assessing the chloride ingress is critical since the replacement was done to the concrete matrix. Table 8.1 represents the electric charge passed through each sample in 6 hours.

Table 8.1. The electric charge passed through sample specimens

	S1		S2		S3-3	
Channel	Charge (C)	Average	Charge (C)	Average	Charge (C)	Average
CH1	615	560	838	629	604	559
CH2	517		532		609	
CH3	550		518		464	

Figure 8.5 represents the variation of electric current (mA) with time in the control sample. The average of three channels results in only 560 coulombs, whereas chloride penetration results in very low classification (100 – 1000 coulombs) according to ASTM C1202. Hence, the control sample exhibits the anticipated result.

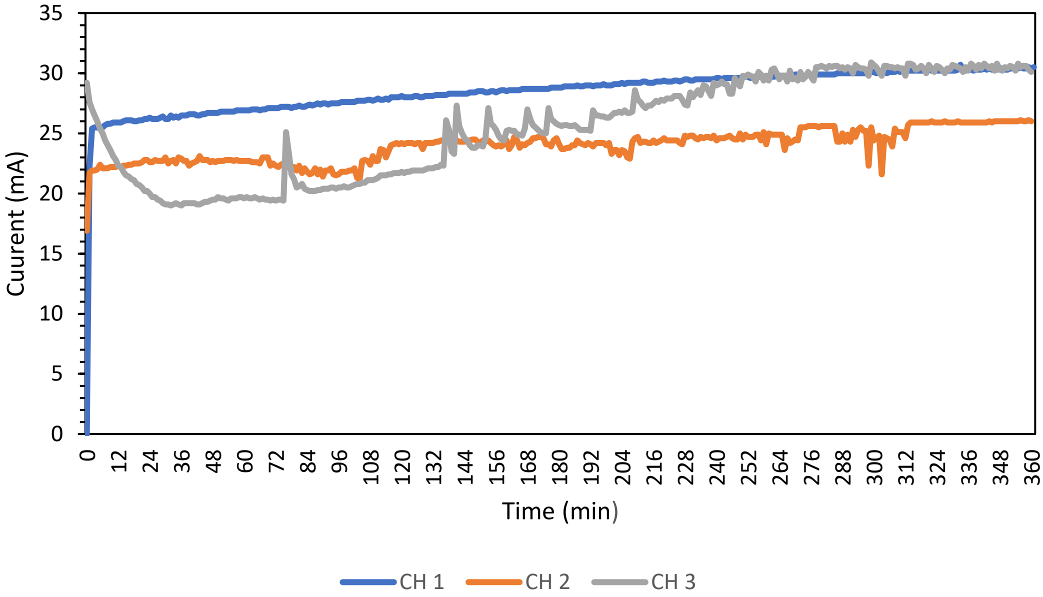


Figure 8.5. RCPT results of the S1 sample

RCPT results of the S2 series are represented in Figure 8.6 and show the highest of 629 coulombs out of the three samples. Hence, incorporating rubber has increased chloride penetration. However, according to ASTM C1202, sample classification is in the region of very low and acceptable for a concrete application. Significantly high chloride penetration resulted in channel 1 compared to the other two channels. This crucial variation can be a reason for CR floating on the concrete matrix, since channel 1 refers to the top of the specimen, the lower specific gravity of CR causes higher chloride penetration compared to mineral aggregates.

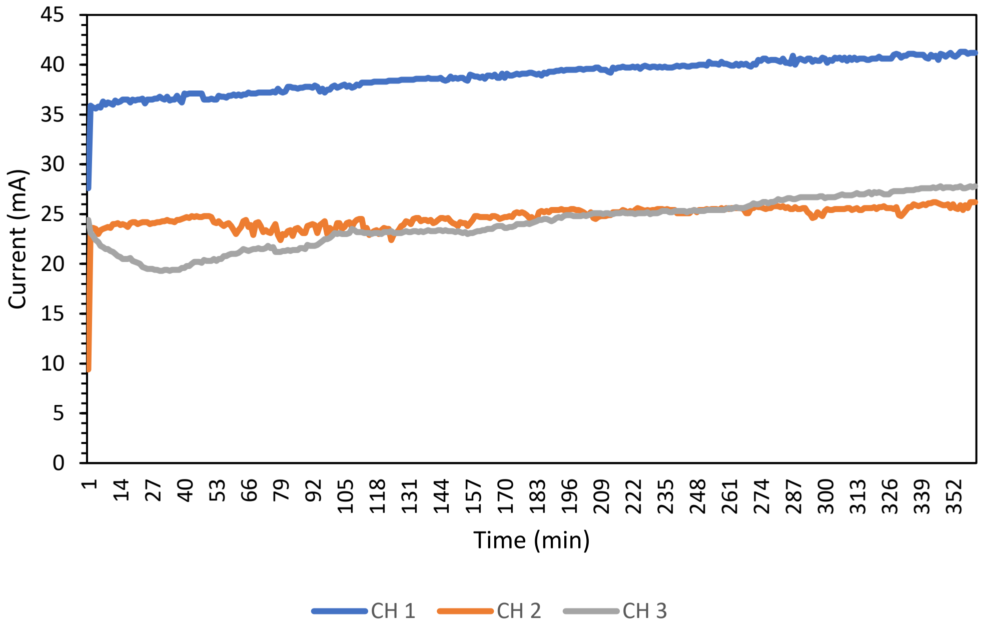


Figure 8.6. RCPT results of the S2 sample

The treated GO rubber incorporated sample shows the lowest out of the three series, an average of 559 coulombs. The treatment of GO has diminished the chloride ion penetration, but further investigations are required. Mostly, the improvement in packing density with GO diminishes the chloride penetration. In addition, an 11.12 % reduction occurred in S3-3 compared to the S2 series. The contrast shown in the top channel of the S2 sample has vanished in the S3-3 sample, even though S3-3 has incorporated rubber. The difference can be explained by the GO treatment, hence the improvement in rubber-cement bonding diminishes the floatation of rubber in preparation for GO-treated RuC.

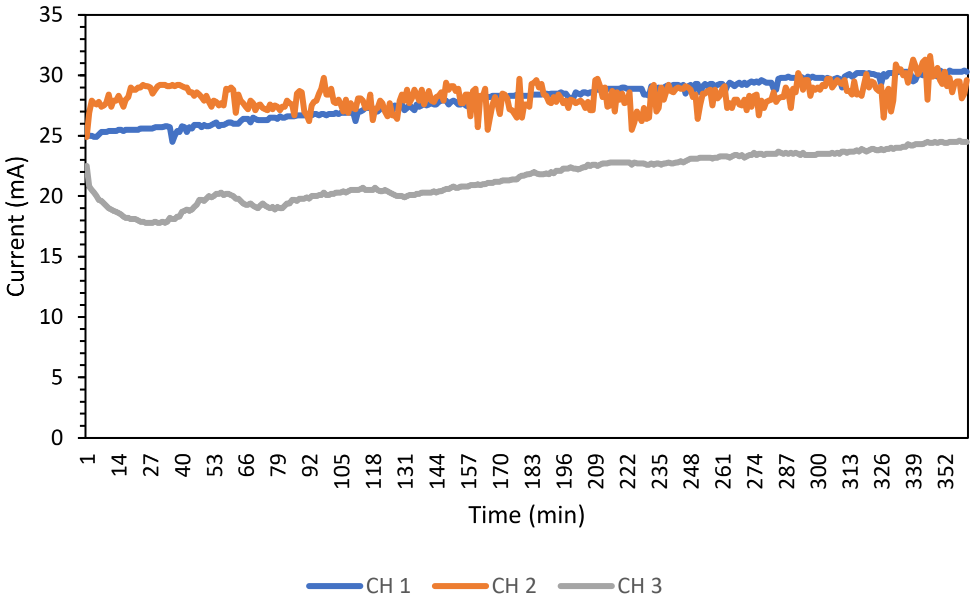


Figure 8.7. RCPT results of the S3-3 sample

8.4 Summary

Investigation of the durability properties of waste tire rubber-incorporated concrete is crucial. Importantly, the replacement of the waste product or a novel constituent to concrete matrix could create sever durability issues. Hence, this chapter investigates the durability properties of developed concrete matrices in selected experiments. With reference to the investigations, the following conclusions were made.

- A significant increase in water penetration resulted from rubber incorporation. The high percentage of water absorption of CR, and the poorly packed nature of RuC are the major reasoning for high water penetration.
- GO treatment to rubber increases water absorption compared to non-treated rubber-only concrete. Hence, 35 % of water penetration has resulted in the S3-3 sample over S2. However, the micro-level material investigation is critical and essential.
- All three samples show chloride ion penetration in a very low classification range under the ASTM C1202. Hence, the RuC application is feasible.
- Pretreatment of CR with GO reduces the chloride ion penetration and is identified to be the lowest of the three series. This can be a result of improved packing density with GO treatment.
- Non-treated rubber floats on the top surface due to low specific gravity, hence, creating higher chloride penetration with respect to bottom channels. However,

further investigations are required to clarify the specific material changing and its distribution.

CHAPTER 9 CONCLUSION AND RECOMMENDATION

The objective of this research project is to develop an optimized waste tire rubber incorporated concrete mix design with enhanced mechanical properties. The bonding behavior of rubber and cement matrix was investigated, and GO was used as a pretreatment method to enhance the material bonding. In addition, the short- and long-term mechanical and durability properties were examined.

9.1 Conclusions

The following conclusions were made from the detailed research program in developing an optimized rubber-incorporated concrete mix design.

- The water absorption of CR is significantly high compared to natural minerals aggregates and the finer particles enhance the water absorption further, as the surface area increases.
- The density of RuC is comparatively less as a result of the low specific gravity possessed by CR, but the non-homogenous rubber replacement improves the specimen density compared to homogenous specimens.
- The inclusion of rubber decreases the compressive strength of concrete. But the non-homogenous rubber replacement was identified to be the prominent replacement with a 10.5 % strength increment over homogenous rubber. This could be a result of improved packing density in the concrete matrix.
- GO possesses exquisite mechanical properties and enhanced dispersibility. The oxidative functionalities found in GO create easy absorption of free radicals of water and the peculiar layer structure also effect immensely in modifying cement-based material.
- Graphene oxide produces strong adhesion between polymer membranes due to the high Van der Waals forces, hence can be used as a pretreatment method for crumb rubber to improve the mechanical strength of RuC.
- The alkaline nature of rubber diminishes the acidic nature of GO and neutralizes the rubber mix GO solution. A minimum of two-hour pretreatment is required as the pH value of the mixture settles after 120 minutes.

- Graphene oxide creates no structural changes to crumb rubber, unlike other acidic treatments. Further, GO pretreatment deposits GO around CR and enriches the density of deposition of GO results with the increment of using higher GO concentrations.
- The loss of dry density in RuC can be enhanced with GO pretreatment. Hence, precipitated GO results as fillers in the concrete matrix to improve the dry density.
- The air-dried pretreated crumb rubber is the most efficient and effective method of pretreatment, as a concrete matrix with air-dried graphene oxide-treated crumb rubber resulting only a 9.42% strength reduction with respect to the control mixture. In addition, a significant strength enhancement of 88.18% was obtained over non-treated crumb rubber concrete.
- The compressive strength increases with a high concentration of graphene oxide, but a graphene oxide concentration of 2 g/l was identified to be optimum as exceeding the optimum concentration reduces the compressive strength as a result of the agglomeration of GO. Hence, excellent dispersion and the appropriate amount of graphene oxide are crucial for the enhancement of concrete strength.
- A clear separation between CR and cement matrix was observed in non-treated RuC samples as it shows poor strength compared to GO-treated RuC. GO precipitation around CR enhances the bonding of CR and the cement matrix and reduces the ITZ.
- The inclusion of rubber aggregate increases the water penetration significantly, Hence the durability of RuC in the structural application is a real concern. The GO results in higher absorption and thereby enhances the water penetration by 35% compared to non-treated RuC.
- The water absorption of CR, as well as the GO with respect to a mineral aggregate increase the water penetration significantly. Hence, further research studies require to identify the mitigation of water absorption.
- All three samples show chloride ion penetration in a very low classification range under the ASTM C1202. However, pretreatment of CR with GO reduces the chloride ion penetration and is identified to be the lowest of the three series. This can be a result of improved packing density with GO treatment.

9.2 Recommendations

The scarcity of natural aggregates and the rapid expansion of construction has created the requirement of finding environmentally friendly aggregates. Waste tire rubber is one such prominent candidate to replace mineral aggregate with enhanced concrete properties. Hence, investigating rubber-incorporated concrete matrices is recommended. However, the poor mechanical properties hinder the application of RuC, where the novel concrete mix should be developed. In addition, the major reasoning for poor bonding behavior between rubber and cement paste should be addressed.

The incorporation of nanomaterial into the concrete matrix was identified to improve the mechanical properties of concrete. Hence the pretreatment with GO is recommended as a result of the enhanced mechanical strength. However, it is recommended to identify the optimum GO concentration, or else the concrete strength reduced drastically.

Even though GO pretreatment increases the mechanical strength, the application of RuC in reinforced structures should be investigated further. The diminished durability properties were identified to be the main reasoning, specifically the significant enhancement in water penetration. Hence, further research studies are essential on the durability properties of GO-treated RuC. In addition, micro-level material behavior investigations are recommended, which identify the chemical and physical changes of concrete constituents.

With new investigations and overcoming the limitations, rubber-incorporated modified concrete mix is an appropriate candidate for structural application with the necessary investigation and identifying the required limitations.

REFERENCES

[1] M. J. Forrest, *Recycling and Re-use of Waste Rubber*. Berlin, Boston: De Gruyter, 2019.

[2] A. Hameed, Afia S and Shashikala, “Suitability of rubber concrete for railway sleepers,” *Perspect. Sci.*, vol. 8, pp. 32–35, 2016.

[3] S. N. Xiao, Feipeng and Zhao, PE Wenbin and Amirkhanian, “Fatigue behavior of rubberized asphalt concrete mixtures containing warm asphalt additives,” *Constr. Build. Mater.*, vol. 23, no. 10, pp. 3144–3151, 2009.

[4] D. Li, Y. Zhuge, R. Gravina, and J. E. Mills, “Compressive stress strain behavior of crumb rubber concrete (CRC) and application in reinforced CRC slab,” *Constr. Build. Mater.*, vol. 166, pp. 745–759, 2018.

[5] E. Güneyisi, M. Gesoglu, N. Naji, and S. İpek, “Evaluation of the rheological behavior of fresh self-compacting rubberized concrete by using the Herschel–Bulkley and modified Bingham models,” *Arch. Civ. Mech. Eng.*, vol. 16, no. 1, pp. 9–19, 2016.

[6] J. Xie, C. Fang, Z. Lu, Z. Li, and L.-J. Li, “Effects of the addition of silica fume and rubber particles on the compressive behaviour of recycled aggregate concrete with steel fibres,” *J. Clean. Prod.*, vol. 197, pp. 656–667, 2018.

[7] H. Liu, X. Wang, Y. Jiao, and T. Sha, “Experimental Investigation of the Mechanical and Durability Properties of Crumb Rubber Concrete,” *Materials (Basel).*, vol. 9, p. 172, 2016.

[8] J. O. Akinyele, R. W. Salim, and W. K. Kupolati, “the Impact of Rubber Crumb on the Mechanical and Chemical Properties of Concrete,” *Eng. Struct. Technol.*, vol. 7, no. 4, pp. 197–204, 2016.

[9] B. S. Thomas and R. C. Gupta, “Long term behaviour of cement concrete containing discarded tire rubber,” *J. Clean. Prod.*, vol. 102, pp. 78–87, 2015.

[10] M. K. Batayneh, I. Marie, and I. Asi, “Promoting the use of crumb rubber concrete in developing countries,” *Waste Manag.*, vol. 28, no. 11, pp. 2171–2176, 2008.

[11] J. Kang and Y. Jiang, “Improvement of cracking-resistance and flexural behavior of cement-based materials by addition of rubber particles,” *J. Wuhan Univ. Technol. Sci. Ed.*, vol. 23, no. 4, pp. 579–583, 2008.

[12] I. Mohammadi, H. Khabbaz, and K. Vessalas, “In-depth assessment of Crumb Rubber Concrete (CRC) prepared by water-soaking treatment method for rigid pavements,” *Constr. Build. Mater.*, vol. 71, pp. 456–471, 2014.

[13] I. Mohammadi and H. Khabbaz, “Shrinkage performance of Crumb Rubber Concrete (CRC) prepared by water-soaking treatment method for rigid pavements,” *Cem. Concr. Compos.*, vol. 62, pp. 106–116, 2015.

[14] E. Ganjian, M. Khorami, and A. A. Maghsoudi, “Scrap-tyre-rubber replacement for aggregate and filler in concrete,” *Constr. Build. Mater.*, vol. 23, no. 5, pp. 1828–1836, 2009.

[15] F. Aslani, “Mechanical properties of waste tire rubber concrete,” *J. Mater. Civ. Eng.*, vol. 28, no. 3, p. 04015152, 2016.

[16] B. S. Thomas and R. Chandra Gupta, “Properties of high strength concrete containing scrap tire rubber,” *J. Clean. Prod.*, vol. 113, pp. 86–92, 2016.

[17] R. K. Gupta, Trilok and Chaudhary, Sandeep and Sharma, “Assessment of mechanical and durability properties of concrete containing waste rubber tire as fine aggregate,” *Constr. Build. Mater.*, vol. 73, pp. 562–574, 2014.

[18] A. Grinys, H. Sivilevičius, and M. Daukšys, “Tyre Rubber Additive Effect on Concrete Mixture Strength,” *J. Civ. Eng. Manag.*, vol. 18, no. 3, pp. 393–401, 2012.

[19] T. Gonen, “Freezing-thawing and impact resistance of concretes containing waste crumb rubbers,” *Constr. Build. Mater.*, vol. 177, pp. 436–442, 2018.

[20] R. Si, J. Wang, S. Guo, Q. Dai, and S. Han, “Evaluation of laboratory performance of self-consolidating concrete with recycled tire rubber,” *J. Clean. Prod.*, vol. 180, pp. 823–831, 2018.

[21] F. Liu, G. Chen, L. Li, and Y. Guo, “Study of impact performance of rubber reinforced concrete,” *Constr. Build. Mater.*, vol. 36, pp. 604–616, 2012.

[22] F. Aslani, G. Ma, D. L. Yim Wan, and V. X. Tran Le, “Experimental investigation into rubber granules and their effects on the fresh and hardened properties of self-compacting concrete,” *J. Clean. Prod.*, vol. 172, pp. 1835–1847, 2018.

[23] T. Gupta, S. Chaudhary, and R. Sharma, “Assessment of mechanical and durability properties of concrete containing waste rubber tire as fine aggregate,” *Constr. Build. Mater.*, vol. 73, pp. 562–574, 2014.

[24] Y. Zheng, L and Huo, X Sharon and Yuan, “Strength, modulus of elasticity, and brittleness index of rubberized concrete,” *J. Mater. Civ. Eng.*, vol. 20, no. 11, pp. 692--699, 2008.

[25] S. Raffoul, R. Garcia, D. Escolano-Margarit, M. Guadagnini, I. Hajirasouliha, and K. Pilakoutas, “Behaviour of unconfined and FRP-confined rubberised concrete in axial compression,” *Constr. Build. Mater.*, vol. 147, pp. 388–397, 2017.

[26] M. Forrest, “Recycling and re-use of waste rubber,” in *orrest2014recycling*, 2014.

[27] U.S. Tire Manufacturers Association, “The national trade association for tire manufacturers that make tires in the U.S.” [Online]. Available: https://www.ustires.org/.

[28] “ETRMA’s statistics on scrap tire collection and recycling in Europe.” [Online]. Available: //weibold.com/etrmas-statistics-on-scrap-tire-collection-and%02recycling-in-europe/.

[29] S. Zhang, Zhigang and Ma, Hui and Qian, “Investigation on properties of ECC incorporating crumb rubber of different sizes,” *J. Adv. Concr. Technol.*, vol. 13, no. 5, pp. 241--251, 2015.

[30] A. Azevedo, F and Pacheco-Torgal, F and Jesus, C and De Aguiar, JL Barroso and Camoes, “Properties and durability of HPC with tyre rubber wastes,” *Constr. Build. Mater.*, vol. 34, no. 186–191, 2012.

[31] R. Asutkar, Priyanka and Shinde, SB and Patel, “Study on the behaviour of rubber aggregates concrete beams using analytical approach,” *Eng. Sci.*

Technol. an Int. J., vol. 20, no. 1, pp. 151–159, 2017.

[32] J. E. Li, Danda and Zhuge, Yan and Gravina, Rebecca and Mills, "Compressive stress strain behavior of crumb rubber concrete (CRC) and application in reinforced CRC slab," *Constr. Build. Mater.*, vol. 166, pp. 745–759, 2018.

[33] S. Raffoul, R. Garcia, K. Pilakoutas, M. Guadagnini, and N. F. Medina, "Optimisation of rubberised concrete with high rubber content: An experimental investigation," *Constr. Build. Mater.*, vol. 124, no. October, pp. 391–404, 2016.

[34] V. J. Thomas, Blessen Skariah and Gupta, Ramesh Chandra and Panicker, "Recycling of waste tire rubber as aggregate in concrete: durability-related performance," *J. Clean. Prod.*, vol. 112, pp. 504--513, 2016.

[35] A. S. Senin, Mohamad Syamir and Shahidan, Shahiron and Abdullah, Siti Radziah and Guntor, Nickholas Anting and Leman, "A review on the suitability of rubberized concrete for concrete bridge decks," in *IOP Conference Series: Materials Science and Engineering*, 2017, p. 012074.

[36] S. I. Li, Guoqiang and Pang, Su-Seng and Ibekwe, "FRP tube encased rubberized concrete cylinders," *Mater. Struct.*, vol. 44, no. 1, pp. 233–243, 2011.

[37] T. M. Pham, X. Zhang, M. Elchalakani, A. Karrech, H. Hao, and A. Ryan, "Dynamic response of rubberized concrete columns with and without FRP confinement subjected to lateral impact," *Constr. Build. Mater.*, vol. 186, pp. 207–218, 2018.

[38] H. H. Ibrahim, A. Z. Saber, and H. A. Hodhod, "Abrasion Resistance, Flexural Toughness and Impact Resistance of Rubberized Concrete," *Int. J. Eng. Adv. Technol.*, vol. 9, no. 3, pp. 1032–1038, 2020.

[39] A. Richardson, K. Coventry, V. Edmondson, and E. Dias, "Crumb rubber used in concrete to provide freeze–thaw protection (optimal particle size)," *J. Clean. Prod.*, vol. 112, pp. 599–606, 2016.

[40] A. Topccu, Ilker Bekir and Demir, "Durability of rubberized mortar and concrete," *J. Mater. Civ. Eng.*, vol. 19, no. 2, pp. 173–178, 2007.

[41] A. Ocholi, S. P. Ejeh, and S. M. Yinka, “An Investigation into the Thermal Performance of Rubber-Concrete,” *Acad. J. Interdiscip. Stud.*, vol. 3, no. 5, p. 29, 2014.

[42] K. B. Najim and M. R. Hall, “A review of the fresh/hardened properties and applications for plain- (PRC) and self-compacting rubberised concrete (SCRC),” *Constr. Build. Mater.*, vol. 24, no. 11, pp. 2043–2051, 2010.

[43] F. O. S. Division, “Tracking the Fate of Scrap Tires in Texas: An Audit Report,” 2006.

[44] NEPIS, “U.S. Environmental Protection Agency : 1991 Management Control Plan,” 1991.

[45] R. Policy, Planning and Glass, “United States Environmental Protection Agency EPA/530-SW-90-071A,” 1992.

[46] “SCRAP TIRE MARKETS IN THE UNITED STATES 9th BIENNIAL REPORT,” 2009.

[47] N. N. Gerges, C. A. Issa, and S. A. Fawaz, “Rubber concrete: Mechanical and dynamical properties,” *Case Stud. Constr. Mater.*, vol. 9, p. e00184, 2018.

[48] A. Fazli and D. Rodrigue, “Waste Rubber Recycling: A Review on the Evolution and Properties of Thermoplastic Elastomers,” *Materials (Basel).*, vol. 13, no. 3, 2020.

[49] A. Siddika, M. A. Al Mamun, R. Alyousef, Y. H. M. Amran, F. Aslani, and H. Alabduljabbar, “Properties and utilizations of waste tire rubber in concrete: A review,” *Constr. Build. Mater.*, vol. 224, pp. 711–731, 2019.

[50] Instron - USA, “Testing Applications and Solutions,” 25AD. [Online]. Available: https://www.azom.com/article.aspx?ArticleID=14881. [Accessed: 02-Aug-2021].

[51] S. Ramarad, M. Khalid, C. T. Ratnam, A. L. Chuah, and W. Rashmi, “Waste tire rubber in polymer blends: A review on the evolution, properties and future,” *Prog. Mater. Sci.*, vol. 72, pp. 100–140, 2015.

[52] S. Herrero, P. Mayor, and F. Hernández-Olivares, “Influence of proportion and

particle size gradation of rubber from end-of-life tires on mechanical, thermal and acoustic properties of plaster–rubber mortars," *Mater. Des.*, vol. 47, pp. 633–642, 2013.

[53] L. Li, S. Ruan, and L. Zeng, "Mechanical properties and constitutive equations of concrete containing a low volume of tire rubber particles," *Constr. Build. Mater.*, vol. 70, pp. 291–308, 2014.

[54] S. Raffoul, R. Garcia, K. Pilakoutas, M. Guadagnini, and N. F. Medina, "Optimisation of rubberised concrete with high rubber content: An experimental investigation," *Constr. Build. Mater.*, vol. 124, no. April 2018, pp. 391–404, 2016.

[55] A. Toumi, T.-H. Nguyen, and A. Turatsinze, "Debonding of a thin rubberised and fibre-reinforced cement-based repairs: Analytical and experimental study," *Mater. Des.*, vol. 49, pp. 90–95, 2013.

[56] M. Elchalakani, *High strength rubberized concrete containing silica fume for the construction of sustainable road side barriers*. Elsevier, 2015.

[57] EPA, "Identification of Non-Hazardous Secondary Materials That Are Solid Waste," 2021. [Online]. Available: https://www.epa.gov/rcra/identification-non-hazardous-secondary-materials-are-solid-waste. [Accessed: 10-Aug-2021].

[58] M. C. Samolada and A. Zabaniotou, "Potential application of pyrolysis for the effective valorisation of the end of life tires in Greece," *Environ. Dev.*, vol. 4, pp. 73–87, 2012.

[59] ETRMA, "End-of-life Tyre REPORT 2015," 2015.

[60] Conserve Energy Future, "Tire Recycling." [Online]. Available: https://www.conserve-energy-future.com/tirerecycling.php. [Accessed: 10-Aug-2021].

[61] F. Elshazly, S. Mustafa, and H. Fawzy, "Rubberized concrete properties and its structural engineering applications – An overview," *Egypt. J. Eng. Sci. Technol.*, vol. 30, no. 1, pp. 1–11, 2020.

[62] N. N. Gerges, C. A. Issa, and S. A. Fawaz, "Rubber concrete: Mechanical and

dynamical properties," *Case Stud. Constr. Mater.*, vol. 9, 2018.

[63] Maryland Department of Enviroment, "Maryland Scrap tire Annual Report 2008," 2008.

[64] J. Prabakar, N. Dendorkar, and R. K. Morchhale, "Influence of fly ash on strength behavior of typical soils," *Constr. Build. Mater.*, vol. 18, no. 4, pp. 263–267, 2004.

[65] O. Youssf, R. Hassanli, and J. E. Mills, "Retrofitting square columns using FRP-confined crumb rubber concrete to improve confinement efficiency," *Constr. Build. Mater.*, vol. 153, pp. 146–156, 2017.

[66] K. B. Najim and M. R. Hall, "Mechanical and dynamic properties of self-compacting crumb rubber modified concrete," *Constr. Build. Mater.*, vol. 27, no. 1, pp. 521–530, 2012.

[67] T. Wang, F. Xiao, S. Amirkhanian, W. Huang, and M. Zheng, "A review on low temperature performances of rubberized asphalt materials," *Constr. Build. Mater.*, vol. 145, pp. 483–505, 2017.

[68] B. Muñoz-Sánchez, M. J. Arévalo-Caballero, and M. C. Pacheco-Menor, "Influence of acetic acid and calcium hydroxide treatments of rubber waste on the properties of rubberized mortars," *Mater. Struct.*, vol. 50, no. 1, p. 75, 2016.

[69] E. Fraile-Garcia, J. Ferreiro-Cabello, M. Mendivil-Giro, and A. S. Vicente-Navarro, "Thermal behaviour of hollow blocks and bricks made of concrete doped with waste tyre rubber," *Constr. Build. Mater.*, vol. 176, pp. 193–200, 2018.

[70] A. F. Angelin, F. M. Da Silva, L. A. G. Barbosa, R. C. C. Lintz, M. A. G. De Carvalho, and R. A. S. Franco, "Voids identification in rubberized mortar digital images using K-Means and Watershed algorithms," *J. Clean. Prod.*, vol. 164, pp. 455–464, 2017.

[71] J. H. Chen, K. S. Chen, and L. Y. Tong, "On the pyrolysis kinetics of scrap automotive tires," *J. Hazard. Mater.*, vol. 84, no. 1, pp. 43–55, 2001.

[72] A. E. Richardson, K. A. Coventry, and G. Ward, "Freeze/thaw protection of concrete with optimum rubber crumb content," *J. Clean. Prod.*, vol. 23, no. 1,

pp. 96–103, 2012.

[73] D. Flores-Medina, N. Flores Medina, and F. Hernández-Olivares, “Static mechanical properties of waste rests of recycled rubber and high quality recycled rubber from crumbed tyres used as aggregate in dry consistency concretes,” *Mater. Struct.*, vol. 47, no. 7, pp. 1185–1193, 2014.

[74] A. B. Eldin, Neil N and Senouci, “Rubber-tire particles as concrete aggregate,” *J. Mater. Civ. Eng.*, vol. 5, no. 4, pp. 478–496, 1993.

[75] T. M. Pham, W. Chen, A. M. Khan, H. Hao, M. Elchalakani, and T. M. Tran, “Dynamic compressive properties of lightweight rubberized concrete,” *Constr. Build. Mater.*, vol. 238, p. 117705, 2020.

[76] N. Ganesan, B. Raj, and A. P. Shashikala, “Behavior of self-consolidating rubberized concrete beam-column joints,” *ACI Mater. J.*, vol. 110, no. 6, pp. 697–704, 2013.

[77] G. Li, M. A. Stubblefield, G. Garrick, J. Eggers, C. Abadie, and B. Huang, “Development of waste tire modified concrete,” *Cem. Concr. Res.*, vol. 34, no. 12, pp. 2283–2289, 2004.

[78] O. Youssf, M. A. ElGawady, J. E. Mills, and X. Ma, “An experimental investigation of crumb rubber concrete confined by fibre reinforced polymer tubes,” *Constr. Build. Mater.*, vol. 53, pp. 522–532, 2014.

[79] N. Segre, P. J. M. Monteiro, and G. Sposito, “Surface Characterization of Recycled Tire Rubber to Be Used in Cement Paste Matrix,” *J. Colloid Interface Sci.*, vol. 248, no. 2, pp. 521–523, 2002.

[80] A. Meddah, M. Beddar, and A. Bali, “Use of shredded rubber tire aggregates for roller compacted concrete pavement,” *J. Clean. Prod.*, vol. 72, pp. 187–192, 2014.

[81] T. M. Pham, M. Elchalakani, H. Hao, J. Lai, S. Ameduri, and T. M. Tran, “Durability characteristics of lightweight rubberized concrete,” *Constr. Build. Mater.*, vol. 224, pp. 584–599, 2019.

[82] D. Raghavan, H. Huynh, and C. F. Ferraris, “Workability, mechanical properties, and chemical stability of a recycled tyre rubber-filled cementitious

composite," *J. Mater. Sci.*, vol. 33, no. 7, pp. 1745–1752, 1998.

[83] B. Huang, X. Shu, and J. Cao, "A two-staged surface treatment to improve properties of rubber modified cement composites," *Constr. Build. Mater.*, vol. 40, pp. 270–274, 2013.

[84] L.-H. Chou, C.-N. Lin, C.-K. Lu, C.-H. Lee, and M.-T. Lee, "Improving rubber concrete by waste organic sulfur compounds," *Waste Manag. \& Res.*, vol. 28, no. 1, pp. 29–35, 2010.

[85] A. Benazzouk, O. Douzane, T. Langlet, K. Mezreb, J. M. Roucoult, and M. Quéneudec, "Physico-mechanical properties and water absorption of cement composite containing shredded rubber wastes," *Cem. Concr. Compos.*, vol. 29, no. 10, pp. 732–740, 2007.

[86] L.-H. Chou, C.-K. Yang, M.-T. Lee, and C.-C. Shu, "Effects of partial oxidation of crumb rubber on properties of rubberized mortar," *Compos. Part B Eng.*, vol. 41, no. 8, pp. 613–616, 2010.

[87] G. Ossola and A. Wojcik, "UV modification of tire rubber for use in cementitious composites," *Cem. Concr. Compos.*, vol. 52, pp. 34–41, 2014.

[88] Z. Li, F. Li, and J. S. L. Li, "Properties of concrete incorporating rubber tyre particles," *Mag. Concr. Res.*, vol. 50, no. 4, pp. 297–304, 1998.

[89] K. B. Najim and M. R. Hall, "Crumb rubber aggregate coatings/pre-treatments and their effects on interfacial bonding, air entrapment and fracture toughness in self-compacting rubberised concrete (SCRC)," *Mater. Struct.*, vol. 46, no. 12, pp. 2029–2043, 2013.

[90] F. Pelisser, N. Zavarise, T. A. Longo, and A. M. Bernardin, "Concrete made with recycled tire rubber: Effect of alkaline activation and silica fume addition," *J. Clean. Prod.*, vol. 19, no. 6, pp. 757–763, 2011.

[91] O. Onuaguluchi and D. K. Panesar, "Hardened properties of concrete mixtures containing pre-coated crumb rubber and silica fume," *J. Clean. Prod.*, vol. 82, pp. 125–131, 2014.

[92] Q. Dong, B. Huang, and X. Shu, "Rubber modified concrete improved by chemically active coating and silane coupling agent," *Constr. Build. Mater.*,

vol. 48, pp. 116–123, 2013.

[93] L. H. Chou, C.-K. Lu, J.-R. Chang, and M. T. Lee, “Use of waste rubber as concrete additive,” *Waste Manag. Res.*, vol. 25, no. 1, pp. 68–76, Feb. 2007.

[94] S. Guo, Q. Dai, R. Si, X. Sun, and C. Lu, “Evaluation of properties and performance of rubber-modified concrete for recycling of waste scrap tire,” *J. Clean. Prod.*, vol. 148, pp. 681–689, 2017.

[95] H. A. Toutanji, “The use of rubber tire particles in concrete to replace mineral aggregates,” *Cem. Concr. Compos.*, vol. 18, no. 2, pp. 135–139, 1996.

[96] F. M. Khatib, Zaher K and Bayomy, “Rubberized Portland cement concrete,” *J. Mater. Civ. Eng.*, vol. 11, no. 3, 1999.

[97] A. R. Khaloo, M. Dehestani, and P. Rahmatabadi, “Mechanical properties of concrete containing a high volume of tire–rubber particles,” *Waste Manag.*, vol. 28, no. 12, pp. 2472–2482, 2008.

[98] N. Holmes, A. Browne, and C. Montague, “Acoustic properties of concrete panels with crumb rubber as a fine aggregate replacement,” *Constr. Build. Mater.*, vol. 73, pp. 195–204, 2014.

[99] R. T. M. M., E.-D. A. S., A. E.-W. M. A., and A.-H. M. E., “Mechanical, Fracture, and Microstructural Investigations of Rubber Concrete,” *J. Mater. Civ. Eng.*, vol. 20, no. 10, pp. 640–649, Oct. 2008.

[100] F. Liu, W. Zheng, L. Li, W. Feng, and G. Ning, “Mechanical and fatigue performance of rubber concrete,” *Constr. Build. Mater.*, vol. 47, pp. 711–719, 2013.

[101] B. J. Neville A, *Concrete Technology. Revised Edition-2001 Standards Update*. Harlow: Pearson Education Limited, 1990.

[102] N. N. Eldin and A. B. Senouci, “Measurement and prediction of the strength of rubberized concrete,” *Cem. Concr. Compos.*, vol. 16, no. 4, pp. 287–298, 1994.

[103] A. Turatsinze and M. Garros, “On the modulus of elasticity and strain capacity of Self-Compacting Concrete incorporating rubber aggregates,” *Resour. Conserv. Recycl.*, vol. 52, no. 10, pp. 1209–1215, 2008.

[104] D. G. Daniel, “Chapter 8: Factors Influencing Concrete Workability,” in *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, J. F. Lamond and J. H. Pielert, Eds. West Conshohocken, PA: ASTM International, 2006, pp. 59–72.

[105] M. A. Aiello and F. Leuzzi, “Waste tyre rubberized concrete: Properties at fresh and hardened state,” *Waste Manag.*, vol. 30, no. 8, pp. 1696–1704, 2010.

[106] P. Sukontasukkul and C. Chaikaew, “Properties of concrete pedestrian block mixed with crumb rubber,” *Constr. Build. Mater.*, vol. 20, no. 7, pp. 450–457, 2006.

[107] P. N. Fowler, David W and Quiroga, “The effects of aggregate characteristics on the performance of Portland cement concrete,” 2003.

[108] A. El-Gammal, A. K. Abdel-Gawad, Y. El-Sherbini, and A. Shalaby, “Compressive strength of concrete utilizing waste tire rubber,” *J. Emerg. Trends Eng. Appl. Sci.*, vol. 1, no. 1, pp. 96–99, 2010.

[109] A. O. Atahan and A. Ö. Yücel, “Crumb rubber in concrete: Static and dynamic evaluation,” *Constr. Build. Mater.*, vol. 36, pp. 617–622, 2012.

[110] F. M. da Silva, L. A. Gachet Barbosa, R. C. C. Lintz, and A. E. P. G. A. Jacintho, “Investigation on the properties of concrete tactile paving blocks made with recycled tire rubber,” *Constr. Build. Mater.*, vol. 91, pp. 71–79, 2015.

[111] Z. Cheng and Z. Shi, “Vibration attenuation properties of periodic rubber concrete panels,” *Constr. Build. Mater.*, vol. 50, pp. 257–265, 2014.

[112] G. Montella, A. Calabrese, and G. Serino, “Mechanical characterization of a Tire Derived Material: Experiments, hyperelastic modeling and numerical validation,” *Constr. Build. Mater.*, vol. 66, pp. 336–347, 2014.

[113] B. S. Mohammed, K. M. Anwar Hossain, J. T. Eng Swee, G. Wong, and M. Abdullahi, “Properties of crumb rubber hollow concrete block,” *J. Clean. Prod.*, vol. 23, no. 1, pp. 57–67, 2012.

[114] M. M. Al-Tayeb, B. H. Abu Bakar, H. Ismail, and H. M. Akil, “Effect of partial replacement of sand by recycled fine crumb rubber on the performance of hybrid rubberized-normal concrete under impact load: experiment and

simulation," *J. Clean. Prod.*, vol. 59, pp. 284–289, 2013.

[115] B. S. Mohammed, "Structural behavior and m–k value of composite slab utilizing concrete containing crumb rubber," *Constr. Build. Mater.*, vol. 24, no. 7, pp. 1214–1221, 2010.

[116] P. Sukontasukkul, "Use of crumb rubber to improve thermal and sound properties of pre-cast concrete panel," *Constr. Build. Mater.*, vol. 23, no. 2, pp. 1084–1092, 2009.

[117] A. T. Noaman, B. H. Abu Bakar, and H. M. Akil, "Experimental investigation on compression toughness of rubberized steel fibre concrete," *Constr. Build. Mater.*, vol. 115, no. C, pp. 163–170, 2016.

[118] O. Youssf, R. Hassanli, and J. E. Mills, "Mechanical performance of FRP-confined and unconfined crumb rubber concrete containing high rubber content," *J. Build. Eng.*, vol. 11, pp. 115–126, 2017.

[119] V. Corinaldesi and J. Donnini, "4 - Waste rubber aggregates," in *New Trends in Eco-efficient and Recycled Concrete*, J. de Brito and F. Agrela, Eds. Woodhead Publishing, 2019, pp. 87–119.

[120] N. I. Fattuhi and L. A. Clark, "Cement-based materials containing shredded scrap truck tyre rubber," *Constr. Build. Mater.*, vol. 10, no. 4, pp. 229–236, 1996.

[121] C. E. Pierce and M. C. Blackwell, "Potential of scrap tire rubber as lightweight aggregate in flowable fill," *Waste Manag.*, vol. 23, no. 3, pp. 197–208, 2003.

[122] L. Zheng, X. Sharon Huo, and Y. Yuan, "Experimental investigation on dynamic properties of rubberized concrete," *Constr. Build. Mater.*, vol. 22, no. 5, pp. 939–947, 2008.

[123] E. Güneyisi, M. Gesoğlu, and T. Özturan, "Properties of rubberized concretes containing silica fume," *Cem. Concr. Res.*, vol. 34, no. 12, pp. 2309–2317, 2004.

[124] M. del Río Merino, J. Santa Cruz Astorqui, and M. G. Cortina, "Viability analysis and constructive applications of lightened mortar (rubber cement mortar)," *Constr. Build. Mater.*, vol. 21, no. 8, pp. 1785–1791, 2007.

[125] F. Pacheco-Torgal, Y. Ding, and S. Jalali, “Properties and durability of concrete containing polymeric wastes (tyre rubber and polyethylene terephthalate bottles): An overview,” *Constr. Build. Mater.*, vol. 30, pp. 714–724, 2012.

[126] J. J. Gheni, Ahmed A and ElGawady, Mohamed A and Myers, “Mechanical Characterization of Concrete Masonry Units Manufactured with Crumb Rubber Aggregate,” *ACI Mater. J.*, vol. 114, no. 1, 2017.

[127] H. Cheng, C. Chen, S. Wu, Z. A. Mirza, and Z. Liu, “Emergy evaluation of cropping, poultry rearing, and fish raising systems in the drawdown zone of Three Gorges Reservoir of China,” *J. Clean. Prod.*, vol. 144, pp. 559–571, 2017.

[128] F. M. Khatib, Zaher K and Bayomy, “Rubberized Portland cement concrete,” *J. Mater. Civ. Eng.*, vol. 11, pp. 206--213, 1999.

[129] H. Su, J. Yang, T.-C. Ling, G. S. Ghataora, and S. Dirar, “Properties of concrete prepared with waste tyre rubber particles of uniform and varying sizes,” *J. Clean. Prod.*, vol. 91, pp. 288–296, 2015.

[130] M. Bravo and J. de Brito, “Concrete made with used tyre aggregate: durability-related performance,” *J. Clean. Prod.*, vol. 25, pp. 42–50, 2012.

[131] {Vinh Xuan} Farhad Aslani and Guowei Ma and {Yim Wan}, {Dominic Law} and {Tran Le}, “Experimental investigation into rubber granules and their effects on the fresh and hardened properties of self-compacting concrete,” *J. Clean. Prod.*, vol. 172, pp. 1835--1847, 2018.

[132] A. Anil Thakare *et al.*, “A study on rheological properties of rubber fiber dosed self-compacting mortar,” *Constr. Build. Mater.*, vol. 262, p. 120745, 2020.

[133] E. Medina, J. M. Medina, A. Cobo, and D. M. Bastidas, “Evaluation of mechanical and structural behavior of austenitic and duplex stainless steel reinforcements,” *Constr. Build. Mater.*, vol. 78, pp. 1–7, 2015.

[134] A. Kaci, R. Bouras, V. T. Phan, P. A. Andréani, M. Chaouche, and H. Brossas, “Adhesive and rheological properties of fresh fibre-reinforced mortars,” *Cem. Concr. Compos.*, vol. 33, no. 2, pp. 218–224, 2011.

[135] S. As’ad, P. Gunawan, and M. Syarif Alaydrus, “Fresh State Behavior of Self

Compacting Concrete Containing Waste Material Fibres," *Procedia Eng.*, vol. 14, pp. 797–804, 2011.

[136] V. P. Villar, N. F. Medina, M. M. Alonso, S. G. Diez, and F. Puertas, "Assessment of parameters governing the steel fiber alignment in fresh cement-based composites," *Constr. Build. Mater.*, vol. 207, pp. 548–562, 2019.

[137] S. Grünewald, "9 - Fibre reinforcement and the rheology of concrete," in *Understanding the Rheology of Concrete*, N. Roussel, Ed. Woodhead Publishing, 2012, pp. 229–256.

[138] E. Güneyisi, "Fresh properties of self-compacting rubberized concrete incorporated with fly ash," *Mater. Struct.*, vol. 43, no. 8, pp. 1037–1048, 2010.

[139] M. Sienkiewicz, H. Janik, K. Borzędowska-Labuda, and J. Kucińska-Lipka, "Environmentally friendly polymer-rubber composites obtained from waste tyres: A review," *J. Clean. Prod.*, vol. 147, 2017.

[140] R. Wang, X. Gao, H. Huang, and G. Han, "Influence of rheological properties of cement mortar on steel fiber distribution in UHPC," *Constr. Build. Mater.*, vol. 144, pp. 65–73, 2017.

[141] M. Djalili-Moghaddam and S. Toll, "Fibre suspension rheology: effect of concentration, aspect ratio and fibre size.," *Rheol. Acta*, vol. 45, no. 3, pp. 315–320, 2006.

[142] S. P. Weiss, W Jason and Yang, Wei and Shah, "Influence of specimen size/geometry on shrinkage cracking of rings," *J. Eng. Mech.*, vol. 126, no. 1, pp. 93–101, 2000.

[143] I. B. Topcu, "The properties of rubberized concretes," *Cem. Concr. Res.*, vol. 25, no. 2, pp. 304–310, 1995.

[144] S. Rajaei *et al.*, "RETRACTED: Rubberized alkali-activated slag mortar reinforced with polypropylene fibres for application in lightweight thermal insulating materials," *Constr. Build. Mater.*, vol. 270, p. 121430, 2021.

[145] W. H. Yung, L. C. Yung, and L. H. Hua, "A study of the durability properties of waste tire rubber applied to self-compacting concrete," *Constr. Build. Mater.*, vol. 41, pp. 665–672, 2013.

[146] M. Khorrami, Morteza and Vafai, Abolhassan and Khalilitabas, Ahmad A and Desai, Chandrakant S and Ardakani, “Experimental lnvestigation on mechanical characteristics and environmental effects on rubber concrete,” *Int. J. Concr. Struct. Mater.*, vol. 4, no. 1, pp. 17–23, 2010.

[147] K. Jingfu, H. Chuncui, and Z. Zhenli, “Strength and shrinkage behaviors of roller-compacted concrete with rubber additives,” *Mater. Struct.*, vol. 42, no. 8, pp. 1117–1124, 2009.

[148] P. Sukontasukkul and K. Tiamlom, “Expansion under water and drying shrinkage of rubberized concrete mixed with crumb rubber with different size,” *Constr. Build. Mater.*, vol. 29, pp. 520–526, 2012.

[149] A. P. C. Duarte, B. A. Silva, N. Silvestre, J. de Brito, E. Júlio, and J. M. Castro, “Tests and design of short steel tubes filled with rubberised concrete,” *Eng. Struct.*, vol. 112, pp. 274–286, 2016.

[150] E. Ozbay, M. Lachemi, and U. K. Sevim, “Compressive strength, abrasion resistance and energy absorption capacity of rubberized concretes with and without slag,” *Mater. Struct.*, vol. 44, no. 7, pp. 1297–1307, 2011.

[151] R. Hassanli, J. Mills, D. Li, and T. Benn, “Experimental and Numerical Study on the Behavior of Rubberized Concrete,” *Adv. Civ. Eng. Mater.*, vol. 6, no. 1, pp. 134–156, Apr. 2017.

[152] M. Turki, Mohamed and Zarrad, Ines and Bretagne, Estelle and Queneudec, “Influence of filler addition on mechanical behavior of cementitious mortar-rubber aggregates: experimental study and modeling,” *J. Mater. Civ. Eng.*, vol. 24, no. 11, pp. 1350–1358, 2012.

[153] L. P. Rivas-Vázquez, R. Suárez-Orduña, J. Hernández-Torres, and E. Aquino-Bolaños, “Effect of the surface treatment of recycled rubber on the mechanical strength of composite concrete/rubber,” *Mater. Struct.*, vol. 48, no. 9, pp. 2809–2814, 2015.

[154] W. K. Akinyele, Joseph Olawale and Salim, Ramadhan Wanjala and Kupolati, “The impact of rubber crumb on the mechanical and chemical properties of concrete,” *Eng. Struct. Technol.*, vol. 7, no. 4, pp. 197–204, 2015.

[155] A. Sofi, “Effect of waste tyre rubber on mechanical and durability properties of concrete – A review,” *Ain Shams Eng. J.*, vol. 9, no. 4, pp. 2691–2700, 2018.

[156] M. Gesoğlu, E. Güneyisi, G. Khoshnaw, and S. İpek, “Investigating properties of pervious concretes containing waste tire rubbers,” *Constr. Build. Mater.*, vol. 63, pp. 206–213, 2014.

[157] M. M. Al-Tayeb, B. H. Abu Bakar, H. M. Akil, and H. Ismail, “Performance of Rubberized and Hybrid Rubberized Concrete Structures under Static and Impact Load Conditions,” *Exp. Mech.*, vol. 53, no. 3, pp. 377–384, 2013.

[158] B. S. Mohammed, M. Adamu, and M. S. Liew, “Evaluating the effect of crumb rubber and nano silica on the properties of high volume fly ash roller compacted concrete pavement using non-destructive techniques,” *Case Stud. Constr. Mater.*, vol. 8, pp. 380–391, 2018.

[159] N. Ganesan, J. Bharati Raj, and A. P. Shashikala, “Flexural fatigue behavior of self compacting rubberized concrete,” *Constr. Build. Mater.*, vol. 44, pp. 7–14, 2013.

[160] A. Yilmaz and N. Degirmenci, “Possibility of using waste tire rubber and fly ash with Portland cement as construction materials,” *Waste Manag.*, vol. 29, no. 5, pp. 1541–1546, 2009.

[161] Y. Park, A. Abolmaali, M. Mohammadagha, and S.-H. Lee, “Flexural Characteristic of Rubberized Hybrid Concrete Reinforced With Steel and Synthetic Fibers,” *Adv. Civ. Eng. Mater.*, vol. 3, no. 1, pp. 495–508, Sep. 2014.

[162] M. Gesoğlu, E. Güneyisi, G. Khoshnaw, and S. İpek, “Abrasion and freezing–thawing resistance of pervious concretes containing waste rubbers,” *Constr. Build. Mater.*, vol. 73, pp. 19–24, 2014.

[163] J. Kang, B. Zhang, and G. Li, “The abrasion-resistance investigation of rubberized concrete,” *J. Wuhan Univ. Technol. Sci. Ed.*, vol. 27, no. 6, pp. 1144–1148, 2012.

[164] Z. Xiong, Q. Cai, F. Liu, L. Li, and Y. Long, “Dynamic performance of RAC-filled double-skin tubular columns subjected to cyclic axial compression,” *Constr. Build. Mater.*, vol. 248, p. 118665, 2020.

[165] H. Sallam, A. . Sherbini, M. Seleem, and M. Balaha, “Impact resistance of rubberized concrete,” vol. 31, 2008.

[166] S. Kaewunruen, D. Li, Y. Chen, and Z. Xiang, “Enhancement of Dynamic Damping in Eco-Friendly Railway Concrete Sleepers Using Waste-Tyre Crumb Rubber,” *Materials* , vol. 11, no. 7. 2018.

[167] M. Adamu, B. S. Mohammed, N. Shafiq, and M. Shahir Liew, “Effect of crumb rubber and nano silica on the fatigue performance of roller compacted concrete pavement,” *Cogent Eng.*, vol. 5, no. 1, p. 1436027, Jan. 2018.

[168] S. Gupta, Trilok and Tiwari, Anshuman and Siddique, Salman and Sharma, Ravi K and Chaudhary, “Response assessment under dynamic loading and microstructural investigations of rubberized concrete,” *J. Mater. Civ. Eng.*, vol. 29, no. 8, p. 04017062, 2017.

[169] R. Pacheco-Torres, E. Cerro-Prada, F. Escolano, and F. Varela, “Fatigue performance of waste rubber concrete for rigid road pavements,” *Constr. Build. Mater.*, vol. 176, pp. 539–548, 2018.

[170] F. Xiao, P. Zhao, and S. Amirkhanian, “Fatigue behavior of rubberized asphalt concrete mixtures containing warm asphalt additives,” *Constr. Build. Mater.*, vol. 23, pp. 3144–3151, Oct. 2009.

[171] S. M. Saeed, M. Y. Aman, K. A. Ahmad, A. Batari, A. T. A. Yero, and A. U. Chinade, “Effect of Crumb Rubber Modifier on the Fatigue Performance of Warm Mix Asphalt,” in *GCEC 2017*, 2019, pp. 1367–1376.

[172] F. Hernández-Olivares, G. Barluenga, B. Parga-Landa, M. Bollati, and B. Witoszek, “Fatigue behaviour of recycled tyre rubber-filled concrete and its implications in the design of rigid pavements,” *Constr. Build. Mater.*, vol. 21, no. 10, pp. 1918–1927, 2007.

[173] P. Sugapriya and R. Ramkrishnan, “Crumb Rubber Recycling in Enhancing Damping Properties of Concrete,” *{IOP} Conf. Ser. Mater. Sci. Eng.*, vol. 310, p. 12013, Feb. 2018.

[174] R. Meesit and S. Kaewunruen, “Vibration Characteristics of Micro-Engineered Crumb Rubber Concrete for Railway Sleeper Applications,” *J. Adv. Concr.*

Technol., vol. 15, no. 2, pp. 55–66, 2017.

[175] M. Ayman and E. M. A., “Strain Rate Effect on Properties of Rubberized Concrete Confined with Glass Fiber–Reinforced Polymers,” *J. Compos. Constr.*, vol. 20, no. 5, p. 4016014, Oct. 2016.

[176] O. Youssf, M. A. ElGawady, and J. E. Mills, “Experimental Investigation of Crumb Rubber Concrete Columns under Seismic Loading,” *Structures*, vol. 3, pp. 13–27, 2015.

[177] M. Ayman, G. Ahmed, and E. M. A., “Shaking-Table Testing of High Energy–Dissipating Rubberized Concrete Columns,” *J. Bridg. Eng.*, vol. 22, no. 8, p. 4017042, Aug. 2017.

[178] N. V. Rao and T. Meena, “A review on carbonation study in concrete,” *{IOP} Conf. Ser. Mater. Sci. Eng.*, vol. 263, p. 32011, Nov. 2017.

[179] A. Marques, J. Akasaki, A. Trigo, and M. Marques, “Influence of the surface treatment of tire rubber residues added in mortars,” *Ibracon Struct. Mater. J.*, vol. 1, 2008.

[180] B. S. Thomas, R. C. Gupta, and V. J. Panicker, “Recycling of waste tire rubber as aggregate in concrete: durability-related performance,” *J. Clean. Prod.*, vol. 112, pp. 504–513, 2016.

[181] T. C. Ling, H. M. Nor, and S. K. Lim, “Using recycled waste tyres in concrete paving blocks,” *Proc. Inst. Civ. Eng. - Waste Resour. Manag.*, vol. 163, no. 1, pp. 37–45, 2010.

[182] T. C. Ling, H. M. Nor, M. R. Hainin, and S. K. Lim, “Long-term strength of rubberised concrete paving blocks,” *Proc. Inst. Civ. Eng. - Constr. Mater.*, vol. 163, no. 1, pp. 19–26, 2010.

[183] S. Zhang and L. Zong, “Evaluation of Relationship between Water Absorption and Durability of Concrete Materials,” *Adv. Mater. Sci. Eng.*, vol. 2014, 2014.

[184] R. Di Mundo, A. Petrella, and M. Notarnicola, “Surface and bulk hydrophobic cement composites by tyre rubber addition,” *Constr. Build. Mater.*, vol. 172, pp. 176–184, 2018.

[185] M. Gesoğlu and E. Güneyisi, “Strength development and chloride penetration in rubberized concretes with and without silica fume,” *Mater. Struct.*, vol. 40, no. 9, pp. 953–964, 2007.

[186] N. Oikonomou and S. Mavridou, “Improvement of chloride ion penetration resistance in cement mortars modified with rubber from worn automobile tires,” *Cem. Concr. Compos.*, vol. 31, no. 6, pp. 403–407, 2009.

[187] R. Si, S. Guo, and Q. Dai, “Durability performance of rubberized mortar and concrete with NaOH-Solution treated rubber particles,” *Constr. Build. Mater.*, vol. 153, pp. 496–505, 2017.

[188] B. S. Thomas, R. C. Gupta, P. Mehra, and S. Kumar, “Performance of high strength rubberized concrete in aggressive environment,” *Constr. Build. Mater.*, vol. 83, pp. 320–326, 2015.

[189] M. Gesoğlu and E. Güneyisi, “Permeability properties of self-compacting rubberized concretes,” *Constr. Build. Mater.*, vol. 25, no. 8, pp. 3319–3326, 2011.

[190] R. K. E., A. A. A., H. A. A. A., and C. Bruce, “Evaluation of Abrasion Resistance of Self-Consolidating Rubberized Concrete by Acoustic Emission Analysis,” *J. Mater. Civ. Eng.*, vol. 30, no. 8, p. 4018196, Aug. 2018.

[191] A. M. Rashad, “A comprehensive overview about recycling rubber as fine aggregate replacement in traditional cementitious materials,” *Int. J. Sustain. Built Environ.*, vol. 5, no. 1, pp. 46–82, 2016.

[192] N. Flores Medina, D. Flores-Medina, and F. Hernández-Olivares, “Influence of fibers partially coated with rubber from tire recycling as aggregate on the acoustical properties of rubberized concrete,” *Constr. Build. Mater.*, vol. 129, pp. 25–36, 2016.

[193] E. Güneyisi, T. Özturan, and M. Gesoğlu, “A study on reinforcement corrosion and related properties of plain and blended cement concretes under different curing conditions,” *Cem. Concr. Compos.*, vol. 27, no. 4, pp. 449–461, 2005.

[194] A. Nadal Gisbert *et al.*, “Analysis behaviour of static and dynamic properties of Ethylene-Propylene-Diene-Methylene crumb rubber mortar,” *Constr. Build.*

Mater., vol. 50, pp. 671–682, 2014.

[195] A. M. Marques, J. R. Correia, and J. de Brito, “Post-fire residual mechanical properties of concrete made with recycled rubber aggregate,” *Fire Saf. J.*, vol. 58, pp. 49–57, 2013.

[196] İ. B. Topçu and T. Bilir, “Experimental investigation of some fresh and hardened properties of rubberized self-compacting concrete,” *Mater. Des.*, vol. 30, no. 8, pp. 3056–3065, 2009.

[197] İ. B. Topçu and T. Bilir, “Effects of slag fineness on durability of mortars,” *J. Zhejiang Univ. A*, vol. 8, no. 11, pp. 1725–1730, 2007.

[198] E. Ünlüoğlu, İ. B. Topçu, and B. Yalaman, “Concrete cover effect on reinforced concrete bars exposed to high temperatures,” *Constr. Build. Mater.*, vol. 21, no. 6, pp. 1155–1160, 2007.

[199] A. Farhad and K. Maryam, “Properties of High-Performance Self-Compacting Rubberized Concrete Exposed to High Temperatures,” *J. Mater. Civ. Eng.*, vol. 31, no. 5, p. 4019040, May 2019.

[200] M. M. A. Kader, S. M. Abdel-wehab, M. A. Helal, and H. H. Hassan, “Evaluation of thermal insulation and mechanical properties of waste rubber/natural rubber composite,” *HBRC J.*, vol. 8, no. 1, pp. 69–74, 2012.

[201] P. Meshgin, Y. Xi, and Y. Li, “Utilization of phase change materials and rubber particles to improve thermal and mechanical properties of mortar,” *Constr. Build. Mater.*, vol. 28, no. 1, pp. 713–721, 2012.

[202] T. Gupta, S. Siddique, R. K. Sharma, and S. Chaudhary, “Effect of elevated temperature and cooling regimes on mechanical and durability properties of concrete containing waste rubber fiber,” *Constr. Build. Mater.*, vol. 137, pp. 35–45, 2017.

[203] M. Olivia, “Durability related properties of low calcium fly ash based geopolymer concrete,” Curtin University, 2011.

[204] S. Raffoul, R. Garcia, K. Pilakoutas, M. Guadagnini, and N. F. Medina, “Optimisation of rubberised concrete with high rubber content: An experimental investigation,” *Constr. Build. Mater.*, vol. 124, pp. 391–404,

2016.

[205] N. M. Al-Akhras and M. M. Smadi, “Properties of tire rubber ash mortar,” *Cem. Concr. Compos.*, vol. 26, no. 7, pp. 821–826, 2004.

[206] CEN - EN 206-1, *Concrete - Part 1: Specification, performance, production and conformity*. 2000.

[207] ACI Committee 213, “Guide for Structural Lightweight-Aggregate Concrete,” 1999.

[208] A. Abbas and H. Abdulzahra, “Studying the Properties of Lightweight Concrete Using Construction Materials Waste,” *J. Environ. Sci. Eng. A*, vol. 4, 2015.

[209] W. K. Akinyele, Joseph Olawale and Salim, Ramhadhan Wanjala and Kupolati, “Production of lightweight concrete from waste tire rubber crumb,” *Eng. Struct. Technol.*, vol. 8, pp. 108--116, 2016.

[210] T. M. Pham, M. Elchalakani, A. Karrech, and H. Hao, “Axial impact behavior and energy absorption of rubberized concrete with/without fiber-reinforced polymer confinement,” *Int. J. Prot. Struct.*, vol. 10, no. 2, pp. 154–173, Sep. 2018.

[211] W. Cheewapattananuwong and P. Chaloeywares, “The Innovation of Natural Rubber Applied with Concrete Road Barriers in Thailand,” *Adv. Sci. Technol.*, vol. 103, pp. 55–61, 2020.

[212] H. Reza, Y. Osama, V. Tom, M. J. E., M. Allan, and G. Rebecca, “Experimental Study on Compressive Behavior of FRP-Confined Expansive Rubberized Concrete,” *J. Compos. Constr.*, vol. 24, no. 4, p. 4020034, Aug. 2020.

[213] M. Elchalakani, M. F. Hassanein, A. Karrech, and B. Yang, “Experimental investigation of rubberised concrete-filled double skin square tubular columns under axial compression,” *Eng. Struct.*, vol. 171, pp. 730–746, 2018.

[214] K. Strukar, T. Kalman Šipoš, I. Miličević, and R. Bušić, “Potential use of rubber as aggregate in structural reinforced concrete element – A review,” *Eng. Struct.*, vol. 188, pp. 452–468, 2019.

[215] H. Reza, Y. Osama, and M. J. E., “Seismic Performance of Precast

Posttensioned Segmental FRP-Confined and Unconfined Crumb Rubber Concrete Columns," *J. Compos. Constr.*, vol. 21, no. 4, p. 4017006, Aug. 2017.

[216] I. M. K. and H. A. A. A., "Ductility and Cracking Behavior of Reinforced Self-Consolidating Rubberized Concrete Beams," *J. Mater. Civ. Eng.*, vol. 29, no. 1, p. 4016174, Jan. 2017.

[217] A. S. M. Mendis, S. Al-Deen, and M. Ashraf, "Effect of rubber particles on the flexural behaviour of reinforced crumbed rubber concrete beams," *Constr. Build. Mater.*, vol. 154, pp. 644–657, 2017.

[218] R. Hassanli, O. Youssf, and J. E. Mills, "Experimental investigations of reinforced rubberized concrete structural members," *J. Build. Eng.*, vol. 10, pp. 149–165, 2017.

[219] O. Youssf, M. A. ElGawady, and J. E. Mills, "Static cyclic behaviour of FRP-confined crumb rubber concrete columns," *Eng. Struct.*, vol. 113, pp. 371–387, 2016.

[220] Y. Liu, Feng and Chen, Guixuan and Li, Lijuan and Guo, "Study of impact performance of rubber reinforced concrete," *Constr. Build. Mater.*, vol. 36, pp. 604–616, 2012.

[221] I. Abdulkadir, B. S. Mohammed, M. S. Liew, and M. M. A. Wahab, "Modelling and optimization of the mechanical properties of engineered cementitious composite containing crumb rubber pretreated with graphene oxide using response surface methodology," *Constr. Build. Mater.*, vol. 310, no. December, p. 125259, 2021.

[222] American Society for Testing and Materials, "Astm, C 136. Standard test method for sieve analysis of fine and coarse aggregates," in *Standard test method for sieve analysis of fine and coarse aggregates*, American Society for Testing and Materials, Philadelphia, PA, 2005.

[223] BSI 1097-6, "BSI 1097-6: 2013 Tests for mechanical and physical properties of aggregates. Determination of particle density and water absorption," in *Tests for mechanical and physical properties of aggregates. Determination of particle density and water absorption*, 2013.

[224] BSI 1097-3, "Tests for mechanical and physical properties of aggregates: Determination of loose bulk density and voids," in *Tests for mechanical and physical properties of aggregates: Determination of loose bulk density and voids*, 1998.

[225] M. Rabehi, B. Mezghiche, and S. Guettala, "Correlation between initial absorption of the cover concrete, the compressive strength and carbonation depth," *Constr. Build. Mater.*, vol. 45, pp. 123–129, 2013.

[226] J. XU, Z. Yao, G. Yang, and Q. Han, "Research on crumb rubber concrete: From a multi-scale review," *Constr. Build. Mater.*, vol. 232, no. Complete, 2020.

[227] R. Assaggaf, M. Rizwan Ali, S. Al-Dulaijan, and M. Maslehuddin, "Properties of concrete with untreated and treated crumb rubber – A review," *J. Mater. Res. Technol.*, vol. 11, Feb. 2021.

[228] M. Ismail, M. Sherir, H. Siad, A. Hassan, and M. Lachemi, "Properties of Self-Consolidating Engineered Cementitious Composite Modified with Rubber," *J. Mater. Civ. Eng.*, vol. 30, Jan. 2018.

[229] Y. Wang, Z. Zhang, J. Yu, J. Xiao, and Q. Xu, "Using Green Supplementary Materials to Achieve More Ductile ECC," *Materials* , vol. 12, no. 6. 2019.

[230] O. Youssf *et al.*, "Influence of Mixing Procedures, Rubber Treatment, and Fibre Additives on Rubcrete Performance," *J. Compos. Mater.*, vol. 3, Apr. 2019.

[231] E. Abdelaal *et al.*, "Novel Approach to Improve Crumb Rubber Concrete Strength Using Thermal Treatment," *Constr. Build. Mater.*, vol. 229, Sep. 2019.

[232] L. He, Y. Ma, Q. Liu, and Y. Mu, "Surface modification of crumb rubber and its influence on the mechanical properties of rubber-cement concrete," *Constr. Build. Mater.*, vol. 120, pp. 403–407, Sep. 2016.

[233] S. binti Salehuddin, N. L. Rahim, N. M. Ibrahim, S. A. N. Tajri, and M. Z. Zainol Abidin, "The Behaviour of Rubber Tyre as Fine Aggregate in Concrete Mix," *Appl. Mech. Mater.*, vol. 754–755, pp. 427–431, 2015.

[234] F. M. Z. Hossain, M. Shahjalal, K. Islam, M. Tiznobaik, and M. S. Alam, "Mechanical properties of recycled aggregate concrete containing crumb rubber

and polypropylene fiber," *Constr. Build. Mater.*, vol. 225, pp. 983–996, 2019.

[235] G. Li *et al.*, "Properties of rubberized concrete modified by using silane coupling agent and carboxylated SBR," *J. Clean. Prod.*, vol. 112, pp. 797–807, 2016.

[236] M. K. Ismail and A. A. A. Hassan, "Use of metakaolin on enhancing the mechanical properties of self-consolidating concrete containing high percentages of crumb rubber," *J. Clean. Prod.*, vol. 125, pp. 282–295, 2016.

[237] H. Zhong, E. W. Poon, K. Chen, and M. Zhang, "Engineering properties of crumb rubber alkali-activated mortar reinforced with recycled steel fibres," *J. Clean. Prod.*, vol. 238, p. 117950, 2019.

[238] F. Jokar, M. Khorram, G. Karimi, and N. Hataf, "Experimental investigation of mechanical properties of crumbed rubber concrete containing natural zeolite," *Constr. Build. Mater.*, vol. 208, pp. 651–658, 2019.

[239] Q. Han, G. Yang, J. Xu, Z. Fu, G. Lacidogna, and A. Carpinteri, "Acoustic emission data analyses based on crumb rubber concrete beam bending tests," *Eng. Fract. Mech.*, vol. 210, pp. 189–202, 2019.

[240] K. Bisht and P. V Ramana, "Evaluation of mechanical and durability properties of crumb rubber concrete," *Constr. Build. Mater.*, vol. 155, pp. 811–817, 2017.

[241] B. S. Thomas, R. C. Gupta, P. Kalla, and L. Cseteneyi, "Strength, abrasion and permeation characteristics of cement concrete containing discarded rubber fine aggregates," *Constr. Build. Mater.*, vol. 59, pp. 204–212, 2014.

[242] Z. Zhang, H. Ma, and S. Qian, "Investigation on Properties of ECC Incorporating Crumb Rubber of Different Sizes," *J. Adv. Concr. Technol.*, vol. 13, no. 5, pp. 241–251, 2015.

[243] I. Mohammadi, H. Khabbaz, and K. Vessalas, "Enhancing mechanical performance of rubberised concrete pavements with sodium hydroxide treatment," *Mater. Struct.*, vol. 49, no. 3, pp. 813–827, 2016.

[244] A. Kashani, T. D. Ngo, P. Hemachandra, and A. Hajimohammadi, "Effects of surface treatments of recycled tyre crumb on cement-rubber bonding in concrete composite foam," *Constr. Build. Mater.*, vol. 171, pp. 467–473, 2018.

[245] H. L. Chou, C. K. Lu, J. R. Chang, and T. M. Lee, "Use of waste rubber as concrete additive," *Waste Manag. Res.*, vol. 25, no. 1, pp. 68–76, 2007.

[246] R. A. Assaggaf, M. R. Ali, S. U. Al-Dulaijan, and M. Maslehuddin, "Properties of concrete with untreated and treated crumb rubber – A review," *J. Mater. Res. Technol.*, vol. 11, pp. 1753–1798, 2021.

[247] X. Wang, J. Xia, O. Nanayakkara, and Y. Li, "Properties of high-performance cementitious composites containing recycled rubber crumb," *Constr. Build. Mater.*, vol. 156, pp. 1127–1136, 2017.

[248] O. Youssf, J. E. Mills, and R. Hassanli, "Assessment of the mechanical performance of crumb rubber concrete," *Constr. Build. Mater.*, vol. 125, pp. 175–183, 2016.

[249] J. Wang, Q. Dai, R. Si, and S. Guo, "Mechanical, durability, and microstructural properties of macro synthetic polypropylene (PP) fiber-reinforced rubber concrete," *J. Clean. Prod.*, vol. 234, pp. 1351–1364, 2019.

[250] N. Segre and I. Joekes, "Use of tire rubber particles as addition to cement paste," *Cem. Concr. Res.*, vol. 30, no. 9, pp. 1421–1425, 2000.

[251] M. Safan, F. M. Eid, and M. Awad, "Enhanced properties of crumb rubber and its application in rubberized concrete," *Int. J. Curr. Eng. Technol.*, vol. 7, no. 5, pp. 1784–1790, 2017.

[252] S. Sgobba, M. Borsa, M. Molfetta, and G. C. Marano, "Mechanical performance and medium-term degradation of rubberised concrete," *Constr. Build. Mater.*, vol. 98, pp. 820–831, 2015.

[253] X. Colom *et al.*, "Structural and mechanical studies on modified reused tyres composites," *Eur. Polym. J.*, vol. 42, no. 10, pp. 2369–2378, 2006.

[254] X. Colom, F. Carrillo, and J. Cañavate, "Composites reinforced with reused tyres: Surface oxidant treatment to improve the interfacial compatibility," *Compos. Part A Appl. Sci. Manuf.*, vol. 38, no. 1, pp. 44–50, 2007.

[255] N. F. Medina, R. Garcia, I. Hajirasouliha, K. Pilakoutas, M. Guadagnini, and S. Raffoul, "Composites with recycled rubber aggregates: Properties and opportunities in construction," *Constr. Build. Mater.*, vol. 188, pp. 884–897,

2018.

[256] H. Zhang, M. Gou, X. Liu, and X. Guan, "Effect of rubber particle modification on properties of rubberized concrete," *J. Wuhan Univ. Technol. Sci. Ed.*, vol. 29, no. 4, pp. 763–768, 2014.

[257] C. K. Leung and Z. C. Grasley, "Effect of micrometric and nanometric viscoelastic inclusions on mechanical damping behavior of cementitious composites," *Constr. Build. Mater.*, vol. 35, pp. 444–451, 2012.

[258] A. Ibrahim Abdulla and S. Hadi Ahmed, "Effect of Rubber Treated by Acidic Solution On Some Mechanical Properties of Rubberize Cement Mortar," *Eng. Technol. J.*, vol. 29, no. 13, pp. 2793–2806, Sep. 2011.

[259] C. Chen, H. Qian, W. Chen, Z. Zheng, and H. Zhu, "Auto-weighted multi-view constrained spectral clustering," *Neurocomputing*, vol. 366, pp. 1–11, 2019.

[260] C.-Y. Chen and M.-T. Lee, "Application of Crumb Rubber in Cement-Matrix Composite," *Materials* , vol. 12, no. 3. 2019.

[261] M. Iorio, M. L. Santarelli, G. González-Gaitano, and J. González-Benito, "Surface modification and characterization of basalt fibers as potential reinforcement of concretes," *Appl. Surf. Sci.*, vol. 427, pp. 1248–1256, 2018.

[262] S.-J. Park and K.-S. Cho, "Filler–elastomer interactions: influence of silane coupling agent on crosslink density and thermal stability of silica/rubber composites," *J. Colloid Interface Sci.*, vol. 267, no. 1, pp. 86–91, 2003.

[263] A. Stewart, B. Schlosser, and E. P. Douglas, "Surface Modification of Cured Cement Pastes by Silane Coupling Agents," *ACS Appl. Mater. Interfaces*, vol. 5, no. 4, pp. 1218–1225, Feb. 2013.

[264] G. Xiong, B. Luo, X. Wu, G. Li, and L. Chen, "Influence of silane coupling agent on quality of interfacial transition zone between concrete substrate and repair materials," *Cem. Concr. Compos.*, vol. 28, no. 1, pp. 97–101, 2006.

[265] Y. Li, M. Wang, and Z. Li, "Physical and mechanical properties of Crumb Rubber Mortar(CRM)with interfacial modifiers," *J. Wuhan Univ. Technol. Sci. Ed.*, vol. 25, no. 5, pp. 845–848, 2010.

[266] L. Yu, Q. Yu, and L. Liu, “Hybrid modified rubber powder and its application in cement mortar,” *J. Wuhan Univ. Technol. Sci. Ed.*, vol. 25, no. 6, pp. 1033–1037, 2010.

[267] R. H. Ghedan and D. M. Hamza, “Effect Of Rubber Treatment On Compressive Strength And Thermal Conductivity Of Modified Rubberized Concrete,” *J. Eng. Dev.*, vol. 15, no. 4, pp. 21–29, 2011.

[268] C. Xiaowei, H. Sheng, G. Xiaoyang, and D. Wenhui, “Crumb waste tire rubber surface modification by plasma polymerization of ethanol and its application on oil-well cement,” *Appl. Surf. Sci.*, vol. 409, pp. 325–342, 2017.

[269] D. Hegemann, H. Brunner, and C. Oehr, “Plasma treatment of polymers for surface and adhesion improvement,” *Nucl. Instruments Methods Phys. Res. Sect. B Beam Interact. with Mater. Atoms*, vol. 208, pp. 281–286, 2003.

[270] U. Lommatzsch, D. Pasedag, A. Baalmann, G. Ellinghorst, and H.-E. Wagner, “Atmospheric Pressure Plasma Jet Treatment of Polyethylene Surfaces for Adhesion Improvement,” *Plasma Process. Polym.*, vol. 4, no. S1, pp. S1041–S1045, Apr. 2007.

[271] M. Noeske, J. Degenhardt, S. Strudthoff, and U. Lommatzsch, “Plasma jet treatment of five polymers at atmospheric pressure: surface modifications and the relevance for adhesion,” *Int. J. Adhes. Adhes.*, vol. 24, no. 2, pp. 171–177, 2004.

[272] J. Li, F. Xiao, and S. N. Amirkhanian, “High temperature rheological characteristics of plasma-treated crumb rubber modified binders,” *Constr. Build. Mater.*, vol. 236, p. 117614, 2020.

[273] J. F. Rabek, *Photodegradation of polymers: physical characteristics and applications*. Springer Science \& Business Media, 2012.

[274] I. Mathieson and R. H. Bradley, “Improved adhesion to polymers by UV/ozone surface oxidation,” *Int. J. Adhes. Adhes.*, vol. 16, no. 1, pp. 29–31, 1996.

[275] A. M. Shanmugharaj, J. K. Kim, and S. H. Ryu, “Modification of rubber surface by UV surface grafting,” *Appl. Surf. Sci.*, vol. 252, no. 16, pp. 5714–5722, 2006.

[276] O. Onuaguluchi and N. Banthia, “Durability performance of polymeric scrap

tire fibers and its reinforced cement mortar," *Mater. Struct.*, vol. 50, no. 2, p. 158, 2017.

[277] E. S. Abd-Elaal *et al.*, "Novel approach to improve crumb rubber concrete strength using thermal treatment," *Constr. Build. Mater.*, vol. 229, p. 116901, 2019.

[278] H. Liu *et al.*, "Characterization of a Bacillus subtilis surfactin synthetase knockout and antimicrobial activity analysis," *J. Biotechnol.*, vol. 237, pp. 1–12, 2016.

[279] C. Albano, N. Camacho, J. Reyes, J. L. Feliu, and M. Hernández, "Influence of scrap rubber addition to Portland I concrete composites: Destructive and non-destructive testing," *Compos. Struct.*, vol. 71, no. 3–4, pp. 439–446, 2005.

[280] O. Youssf, R. Hassanli, J. E. Mills, and M. Abd Elrahman, "An experimental investigation of the mechanical performance and structural application of LECA-Rubcrete," *Constr. Build. Mater.*, vol. 175, pp. 239–253, 2018.

[281] N.-P. Pham, A. Toumi, and A. Turatsinze, "Effect of an enhanced rubber-cement matrix interface on freeze-thaw resistance of the cement-based composite," *Constr. Build. Mater.*, vol. 207, pp. 528–534, 2019.

[282] O. Onuaguluchi, "Effects of surface pre-coating and silica fume on crumb rubber-cement matrix interface and cement mortar properties," *J. Clean. Prod.*, vol. 104, pp. 339–345, 2015.

[283] A. Kashani, T. D. Ngo, P. Mendis, J. R. Black, and A. Hajimohammadi, "A sustainable application of recycled tyre crumbs as insulator in lightweight cellular concrete," *J. Clean. Prod.*, vol. 149, pp. 925–935, 2017.

[284] H. Su, J. Yang, G. S. Ghataora, and S. Dirar, "Surface modified used rubber tyre aggregates: effect on recycled concrete performance," *Mag. Concr. Res.*, vol. 67, no. 12, pp. 680–691, 2015.

[285] A. Zandiatashbar *et al.*, "Effect of defects on the intrinsic strength and stiffness of graphene," *Nat. Commun.*, vol. 5, no. 1, p. 3186, 2014.

[286] B. C. Brodie, "XIII. On the atomic weight of graphite," *Philos. Trans. R. Soc. London*, vol. 149, pp. 249–259, Jan. 1859.

[287] H. V Kumar, S. J. Woltornist, and D. H. Adamson, “Fractionation and characterization of graphene oxide by oxidation extent through emulsion stabilization,” *Carbon N. Y.*, vol. 98, pp. 491–495, 2016.

[288] T. Tong, Z. Fan, Q. Liu, S. Wang, S. Tan, and Q. Yu, “Investigation of the effects of graphene and graphene oxide nanoplatelets on the micro- and macro-properties of cementitious materials,” *Constr. Build. Mater.*, vol. 106, pp. 102–114, 2016.

[289] L. Lu, P. Zhao, and Z. Lu, “A short discussion on how to effectively use graphene oxide to reinforce cementitious composites,” *Constr. Build. Mater.*, vol. 189, pp. 33–41, 2018.

[290] R. Hu, Y. He, M. Huang, G. Zhao, and H. Zhu, “Strong Adhesion of Graphene Oxide Coating on Polymer Separation Membranes,” *Langmuir*, vol. 34, no. 36, pp. 10569–10579, Sep. 2018.

[291] B. Mensah, S. Kang, W. Wang, and C. Nah, “Effect of graphene on polar and nonpolar rubber matrices,” *Mech. Adv. Mater. Mod. Process.*, vol. 4, Feb. 2018.

[292] Y. Wang *et al.*, “Room-temperature ferromagnetism of graphene.,” *Nano Lett.*, vol. 9, no. 1, pp. 220–224, Jan. 2009.

[293] K. Gong *et al.*, “Reinforcing Effects of Graphene Oxide on Portland Cement Paste,” *J. Mater. Civ. Eng.*, vol. 27, p. A4014010, Feb. 2015.

[294] Z. Pan *et al.*, “Mechanical properties and microstructure of a graphene oxide-cement composite,” *Cem. Concr. Compos.*, vol. 58, Feb. 2015.

[295] X. Li *et al.*, “Incorporation of graphene oxide and silica fume into cement paste: A study of dispersion and compressive strength,” *Constr. Build. Mater.*, vol. 123, pp. 327–335, Oct. 2016.

[296] Y. Shang, D. Zhang, C. Yang, Y. Liu, and Y. Liu, “Effect of graphene oxide on the rheological properties of cement pastes,” *Constr. Build. Mater.*, vol. 96, pp. 20–28, Oct. 2015.

[297] S. C. Devi and R. A. Khan, “Effect of graphene oxide on mechanical and durability performance of concrete,” *J. Build. Eng.*, vol. 27, no. November 2018, p. 101007, 2020.

[298] E. Garcia Taengua, M. Sonebi, K. Hossain, M. Lachemi, and J. Khatib, "Effects of the addition of nanosilica on the rheology, hydration and development of the compressive strength of cement mortars," *Compos. Part B Eng.*, vol. 81, Nov. 2015.

[299] M. Sonebi, E. Garcia Taengua, K. Hossain, J. Khatib, and M. Lachemi, "Effect of nanosilica addition on the fresh properties and shrinkage of mortars with fly ash and superplasticizer," *Constr. Build. Mater.*, vol. 84, pp. 269–276, Jun. 2015.

[300] S. Lv, Y. Ma, C. Qiu, T. Sun, J. Liu, and Q. Zhou, "Effect of graphene oxide nanosheets of microstructure and mechanical properties of cement composites," *Constr. Build. Mater.*, vol. 49, pp. 121–127, Dec. 2013.

[301] H. Peng, Y. Ge, C. S. Cai, Y. Zhang, and Z. Liu, "Mechanical properties and microstructure of graphene oxide cement-based composites," *Constr. Build. Mater.*, vol. 194, pp. 102–109, Jan. 2019.

[302] X. Li, S. Chuah, W. Li, Y. Liu, W. Duan, and Z. Li, "Effects of graphene oxide aggregates on hydration degree, sorptivity, and tensile splitting strength of cement paste," *Compos. Part A Appl. Sci. Manuf.*, vol. 100, May 2017.

[303] D. Dikin *et al.*, "Preparation and characterization of graphene oxide paper," *Nature*, vol. 448, pp. 457–460, Aug. 2007.

[304] D. Zahin and A. Rizalman, "Properties of cement mortar containing NaOH-treated Crumb rubber as fine aggregate replacement," *IOP Conf. Ser. Earth Environ. Sci.*, vol. 476, p. 12030, Jun. 2020.

[305] A. Adesina and O. Atoyebi, "Effect of crumb rubber aggregate on the performance of cementitious composites: A review," *IOP Conf. Ser. Earth Environ. Sci.*, vol. 445, Mar. 2020.

[306] T. M. Pham, Thong M and Chen, Wensu and Khan, Abdul M and Hao, Hong and Elchalakani, Mohamed and Tran, "Dynamic compressive properties of lightweight rubberized concrete," *Constr. Build. Mater.*, vol. 238, p. 117705, 2020.

[307] B. S. Mohammed, S. Haruna, M. M. A. Wahab, M. S. Liew, and A. Haruna,

"Mechanical and microstructural properties of high calcium fly ash one-part geopolymer cement made with granular activator," *Heliyon*, vol. 5, no. 9, p. e02255, 2019.

[308] Z. U. Rehman, M. A. Raza, F. A. Ghauri, R. Kanwal, A. Ahmad, and A. Inam, "Graphene Oxide Coatings Deposited on Steel Substrate Using Electrophoretic Deposition and Electrochemical Evaluation of Coatings in Saline Media," *Key Eng. Mater.*, vol. 778, pp. 111–117, 2018.

[309] B. S. Mohammed, L. W. Xian, S. Haruna, M. S. Liew, I. Abdulkadir, and N. A. W. A. Zawawi, "Deformation Properties of Rubberized Engineered Cementitious Composites Using Response Surface Methodology," *Iran. J. Sci. Technol. Trans. Civ. Eng.*, vol. 45, no. 2, pp. 729–740, 2021.

[310] V. K. Arachchi, J. Gamage, and K. Selvaranjan, "Investigation of replacing aggregate with non-homogeneous waste tire rubber aggregate in concrete," in *2022 Moratuwa Engineering Research Conference (MERCon)*, 2022, pp. 1–6.

[311] Q. Wang, J. Wang, C. Lv, X. Cui, S. Li, and X. Wang, "Rheological behavior of fresh cement pastes with a graphene oxide additive," *New Carbon Mater.*, vol. 31, no. 6, pp. 574–584, 2016.

[312] ACI, "Standard Practice for Selecting Proportions for Normal , Heavyweight , and Mass Concrete," 2002.

[313] K. Vallurupalli, W. Meng, J. Liu, and K. H. Khayat, "Effect of graphene oxide on rheology, hydration and strength development of cement paste," *Constr. Build. Mater.*, vol. 265, p. 120311, 2020.

[314] M. Saatcioglu, "Concrete structures," *Encycl. Earth Sci. Ser.*, vol. 2018, p. 118, 2013.

[315] M. Heikal, S. Abd El Aleem, and W. M. Morsi, "Characteristics of blended cements containing nano-silica," *HBRC J.*, vol. 9, no. 3, pp. 243–255, Dec. 2013.

[316] D. Mohotti, P. Mendis, K. Wijesooriya, I. Fonseka, D. Weerasinghe, and C. K. Lee, "Abrasion and Strength of high percentage Graphene Oxide (GO) Incorporated Concrete," *Electron. J. Struct. Eng.*, vol. 22, no. 1, pp. 37–43,

2022.

[317] C. Liu *et al.*, “Research progress on individual effect of graphene oxide in cement-based materials and its synergistic effect with other nanomaterials,” vol. 10, no. 1, pp. 1208–1235, 2021.

[318] C. S. R. Indukuri, R. Nerella, and S. R. C. Madduru, “Workability, microstructure, strength properties and durability properties of graphene oxide reinforced cement paste,” *Aust. J. Civ. Eng.*, vol. 18, no. 1, pp. 73–81, Jan. 2020.

[319] N. Azline *et al.*, “Permeation-durability properties of metakaolin blended concrete containing rubber,” *Eur. J. Environ. Civ. Eng.*, vol. 0, no. 0, pp. 1–16, 2021.

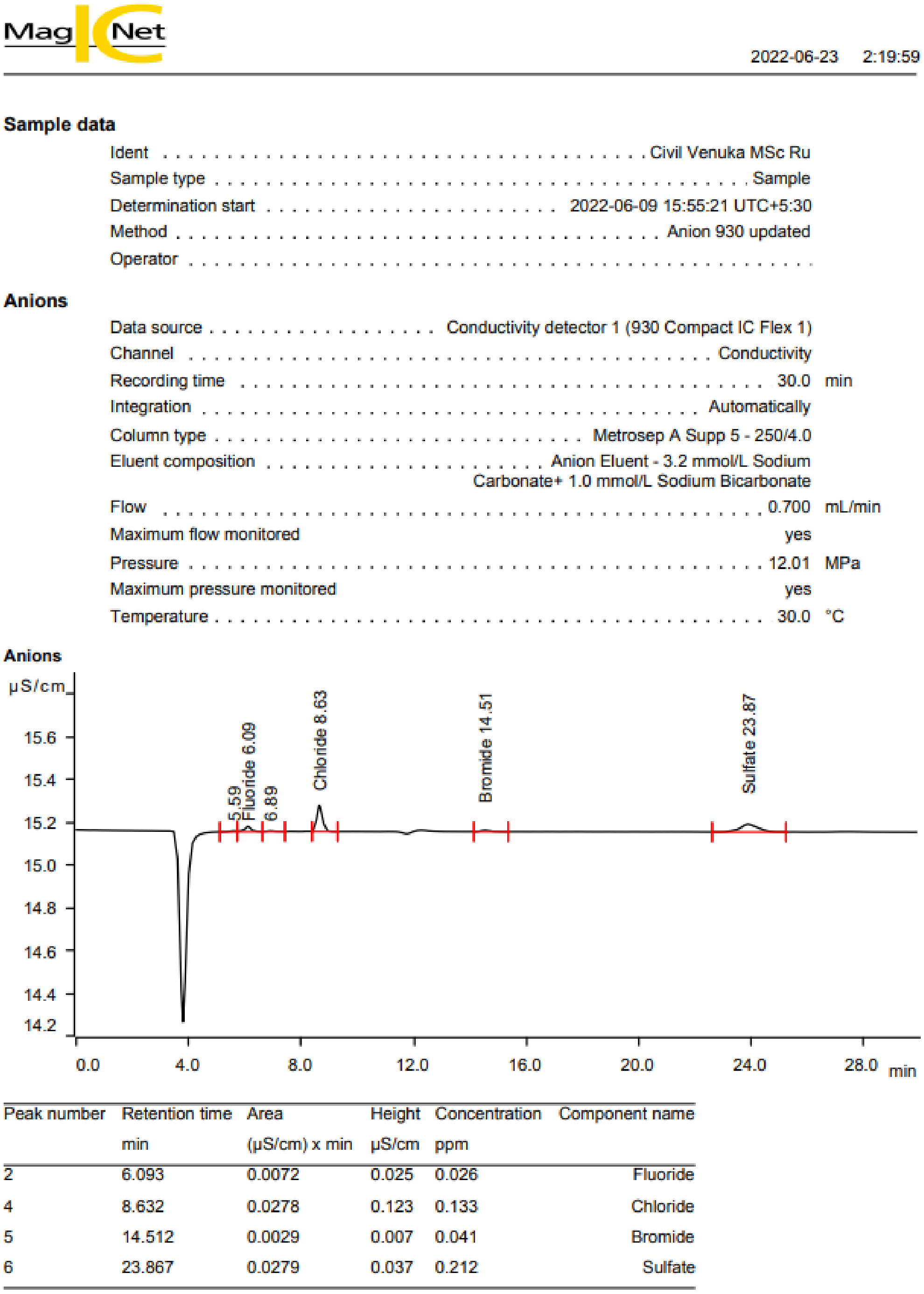
[320] L. Y. Feng, A. J. Chen, and H. D. Liu, “Effect of Waste Tire Rubber Particles on Concrete Abrasion Resistance Under High - Speed Water Flow,” *Int. J. Concr. Struct. Mater.*, 2021.

[321] ASTM, “Standard Test Method for Electrical Indication of Concrete ’ s Ability to Resist Chloride,” 2012, pp. 1–8.

[322] B. Standard, “Testing hardened concrete Ð Part 8 : Depth of penetration of water,” 2000.

APPENDICES

Appendix A – IC test results



MagIC Net

2022-06-23 2:19:59

Sample data

Ident	Civil Venuka MSc Ru
Sample type	Sample
Determination start	2022-06-09 15:55:21 UTC+5:30
Method	Anion 930 updated
Operator	

Anions

Data source	Conductivity detector 1 (930 Compact IC Flex 1)	
Channel	Conductivity	
Recording time	30.0	min
Integration	Automatically	
Column type	Metrosep A Supp 5 - 250/4.0	
Eluent composition	Anion Eluent - 3.2 mmol/L Sodium Carbonate+ 1.0 mmol/L Sodium Bicarbonate	
Flow	0.700	mL/min
Maximum flow monitored	yes	
Pressure	12.01	MPa
Maximum pressure monitored	yes	
Temperature	30.0	°C

Anions

Peak number	Retention time min	Area (µS/cm) x min	Height µS/cm	Concentration ppm	Component name
2	6.093	0.0072	0.025	0.026	Fluoride
4	8.632	0.0278	0.123	0.133	Chloride
5	14.512	0.0029	0.007	0.041	Bromide
6	23.867	0.0279	0.037	0.212	Sulfate

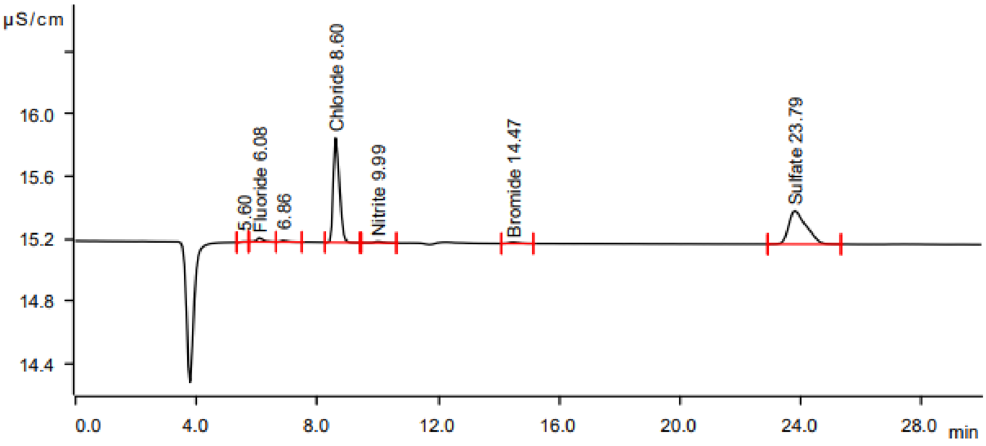
Sample data

Ident	Civil Venuka MSc GO	
Sample type	Sample	
Determination start	2022-06-09 14:47:12 UTC+5:30	
Method	Anion 930 updated	
Operator		

Anions

Data source	Conductivity detector 1 (930 Compact IC Flex 1)	
Channel	Conductivity	
Recording time	30.0	min
Integration	Automatically	
Column type	Metrosep A Supp 5 - 250/4.0	
Eluent composition	Anion Eluent - 3.2 mmol/L Sodium Carbonate+ 1.0 mmol/L Sodium Bicarbonate	
Flow	0.700	mL/min
Maximum flow monitored	yes	
Pressure	11.95	MPa
Maximum pressure monitored	yes	
Temperature	30.0	°C

Anions



Peak number	Retention time min	Area (µS/cm) x min	Height µS/cm	Concentration ppm	Component name
2	6.077	0.0063	0.025	0.023	Fluoride
4	8.603	0.1564	0.672	0.823	Chloride
5	9.990	0.0028	0.008	invalid	Nitrite
6	14.465	0.0034	0.009	0.047	Bromide
7	23.787	0.1438	0.216	1.129	Sulfate

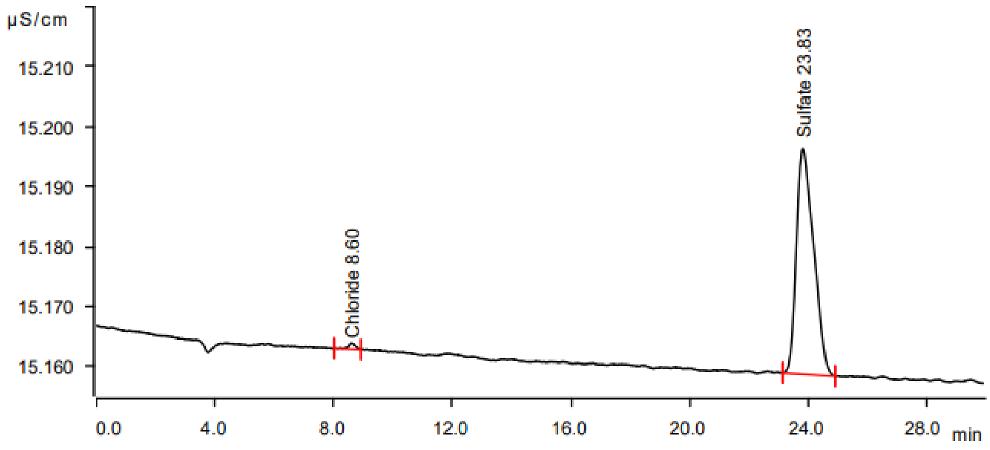
Sample data

Ident	Civil Venuka MSc GO+Ru
Sample type	Sample
Determination start	2022-06-09 15:24:51 UTC+5:30
Method	Anion 930 updated
Operator	

Anions

Data source	Conductivity detector 1 (930 Compact IC Flex 1)	
Channel	Conductivity	
Recording time	30.0	min
Integration	Automatically	
Column type	Metrosep A Supp 5 - 250/4.0	
Eluent composition	Anion Eluent - 3.2 mmol/L Sodium Carbonate+ 1.0 mmol/L Sodium Bicarbonate	
Flow	0.700	mL/min
Maximum flow monitored	yes	
Pressure	11.95	MPa
Maximum pressure monitored	yes	
Temperature	30.0	°C

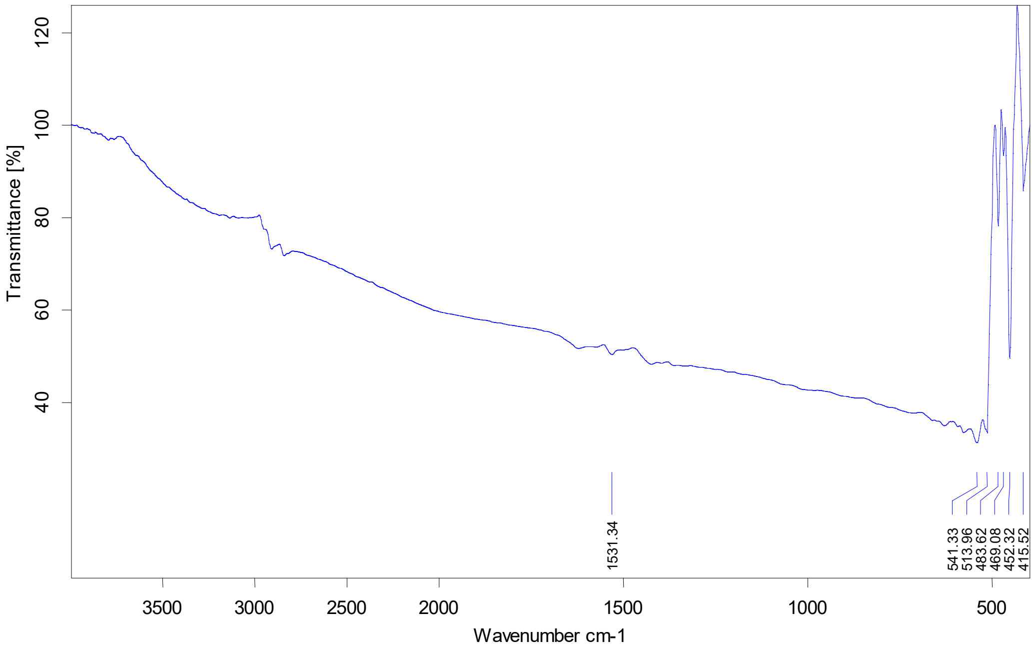
Anions



Peak number	Retention time min	Area (µS/cm) x min	Height µS/cm	Concentration ppm	Component name
1	8.603	0.0003	0.001	-0.014	Chloride
2	23.825	0.0251	0.038	0.191	Sulfate

Appendix B – FTIR test results

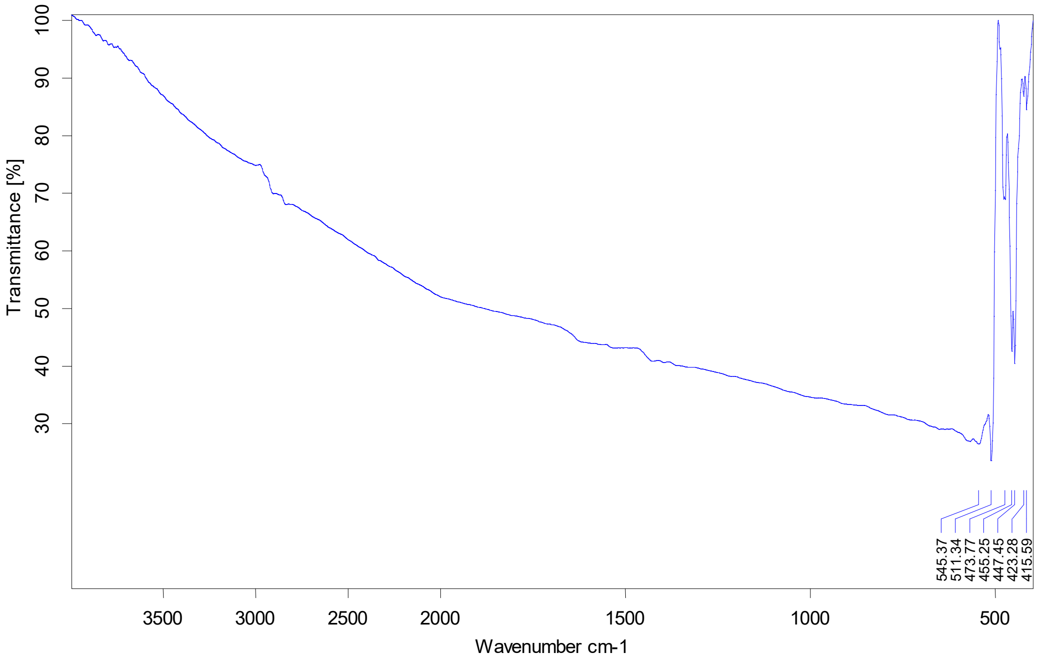
Sample 01 – As received rubber aggregate.



C:\Program Files\OPUS_65\MEAS\VE SAM 01.0 VE SAM 01 Instrument type and / or accessory	08/07/2022

Page 1/1

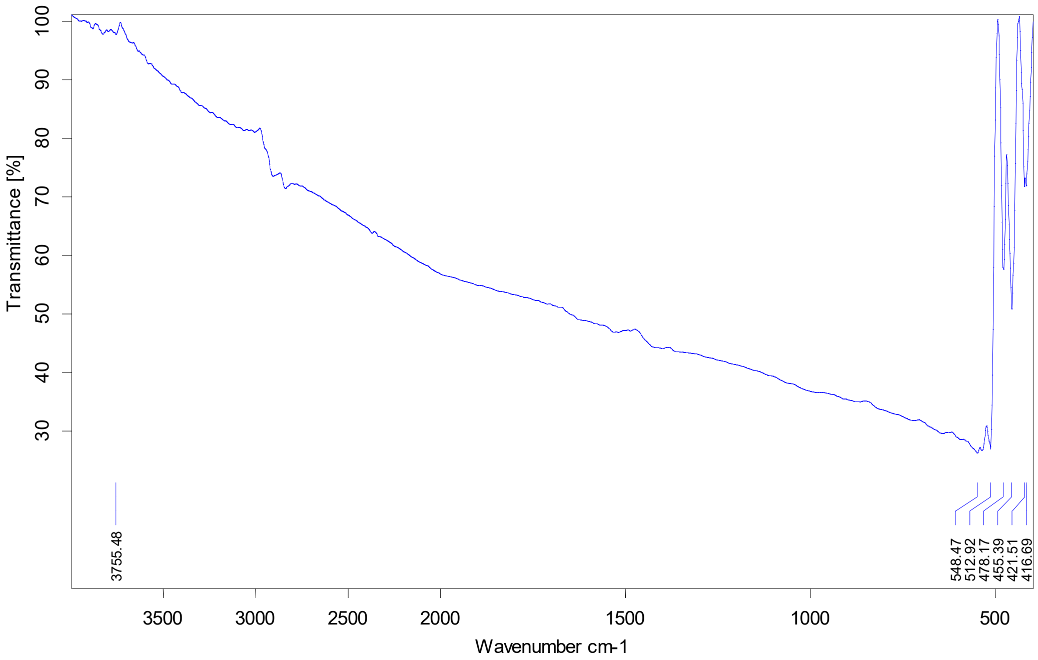
Sample 02 – Pretreated rubber samples with GO 0.5 g/l



C:\Program Files\OPUS_65\MEAS\VE SAM 02.0 VE SAM 02 Instrument type and / or accessory	08/07/2022

Page 1/1

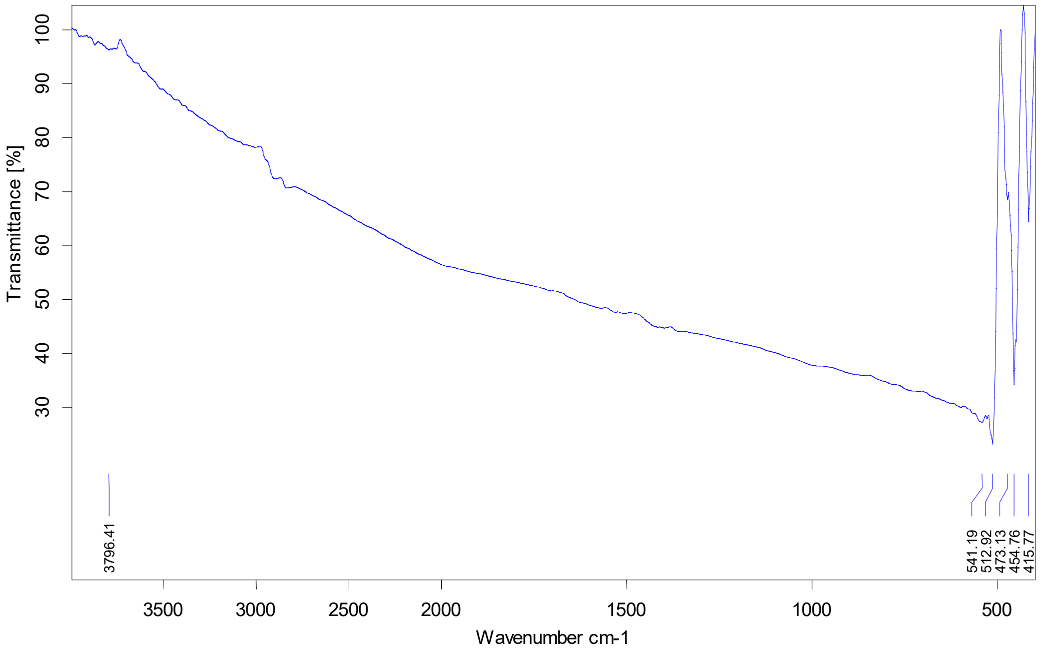
Sample 03 - Pretreated rubber samples with GO 1 g/l



C:\Program Files\OPUS_65\MEAS\VE SAM 03.0	VE SAM 03	Instrument type and / or accessory		08/07/2022

Page 1/1

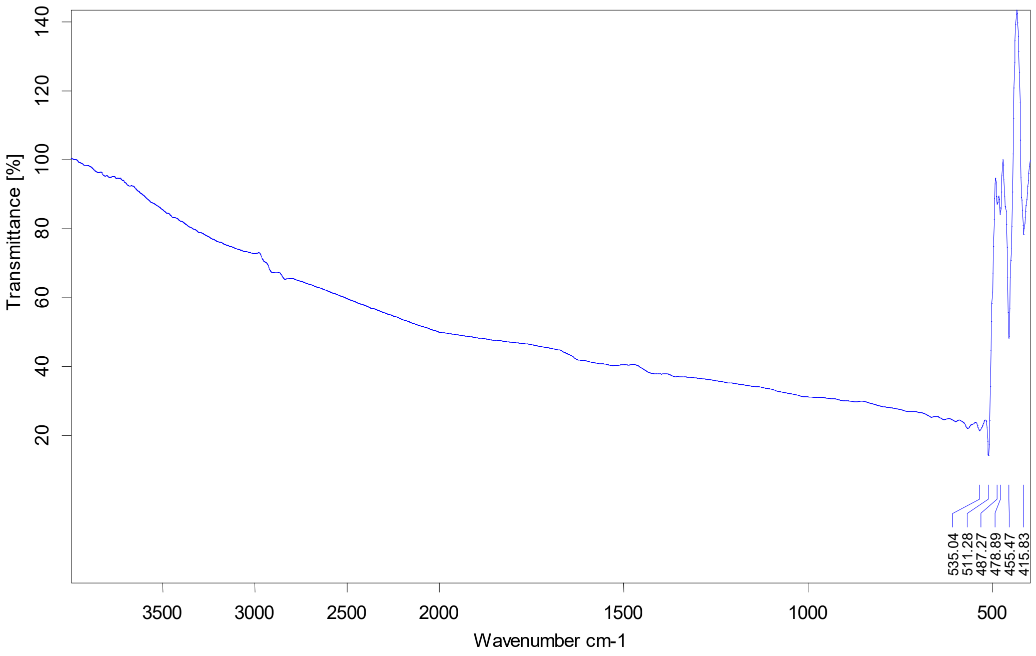
Sample 04 - Pretreated rubber samples with GO 2 g/l



C:\Program Files\OPUS_65\MEAS\VE SAM 04.0 VE SAM 04 Instrument type and / or accessory	08/07/2022

Page 1/1

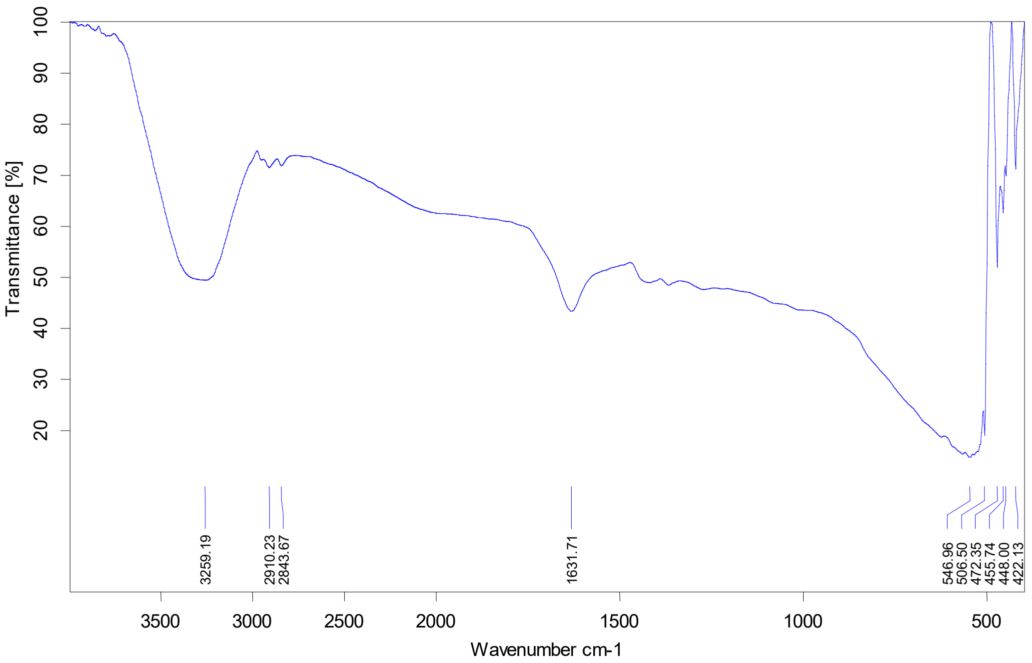
Sample 05 - Pretreated rubber samples with GO 3 g/l



C:\Program Files\OPUS_65\MEAS\VE SAM 05.0 VE SAM 05 Instrument type and / or accessory	08/07/2022

Page 1/1

Sample 06 - Pretreated rubber samples with Acetic acid (5%)



C:\Program Files\OPUS_65\MEAS\VE SAM 06.0 VE SAM 06 Instrument type and / or accessory	08/07/2022

Page 1/1

Appendix C – Rapid chloride ion penetration test

Project description – Research project – Venuka – UOM
Name: i2i con 661 56d
Date: 2023/02/21 - 10:25:06 AM
Test Method: ASTM C1202
Operator: Mr. Indika
Sample ID: I2I CON 661 01, 02, 03

		S1			S2			S3-3		
		Channels			Channels			Channels		
		1	2	3	1	2	3	1	2	3
Row ID	Relative Time (min)	Current (mA)	Current (mA)	Current (mA)	Current (mA)	Current (mA)	Current (mA)	Current (mA)	Current (mA)	Current (mA)
1	0	22.2	16.9	29.2	27.6	9.4	24.4	25.2	24.9	22.5
2	1	25.4	21.7	27.6	35.9	23.6	23.1	25	26.9	20.8
3	2	25.5	21.9	27	35.7	23.6	22.8	25	27.9	20.5
4	3	25.5	21.9	26.5	35.6	23.5	22.4	24.9	27.7	20.3
5	4	25.4	22	26	35.8	23	22.2	24.9	27.6	20
6	5	25.5	22.4	25.4	35.7	23.4	21.8	25.1	27.8	19.7
7	6	25.7	22.1	25	36.3	23.5	21.7	25.3	27.4	19.6
8	7	25.8	22.1	24.4	36	23.6	21.5	25.3	27.5	19.4
9	8	25.8	22.1	24	36.2	23.7	21.5	25.3	27.7	19.2
10	9	25.9	22.2	23.6	36.1	23.6	21.3	25.4	28.4	19
11	10	25.9	22.2	23.1	36	24	21.2	25.4	27.4	18.9
12	11	25.9	22.2	22.8	36.4	24	21	25.4	27.9	18.8
13	12	25.9	22.3	22.3	36.2	24.1	20.8	25.4	27.9	18.7
14	13	26	22.2	22.2	36.3	23.9	20.7	25.5	28.3	18.6
15	14	26.1	22.3	21.8	36.5	24	20.5	25.5	27.9	18.5
16	15	26.1	22.3	21.5	36.5	23.9	20.5	25.4	27.4	18.3
17	16	26.1	22.4	21.4	36.5	23.7	20.5	25.5	28	18.2
18	17	26	22.4	21.2	36.2	24.1	20.6	25.5	28.2	18.2
19	18	26	22.5	21.1	36.5	24.2	20.3	25.5	29	18.1
20	19	26.1	22.5	20.8	36.4	24.2	20.2	25.5	28.8	18.1
21	20	26.1	22.5	20.7	36.5	24	20.1	25.5	28.8	18.1
22	21	26.2	22.6	20.5	36.5	24.1	20	25.5	29	18
23	22	26.2	22.8	20.2	36.6	24.2	19.7	25.6	29	17.9
24	23	26.3	22.8	20.2	36.1	24.2	19.6	25.6	29.2	17.9
25	24	26.2	22.6	20	36.5	24.2	19.5	25.6	29.1	17.8
26	25	26.2	22.6	19.7	36.5	24	19.5	25.6	29.1	17.8
27	26	26.2	22.8	19.7	36.5	24.1	19.5	25.6	29	17.8
28	27	26.3	22.7	19.5	36.6	24.1	19.4	25.6	28.5	17.8
29	28	26.4	22.7	19.4	36.6	24.2	19.4	25.7	28.8	17.9
30	29	26.2	22.7	19.2	36.8	24.2	19.3	25.7	29.1	17.8
31	30	26.2	23	19.1	36.6	24.3	19.3	25.7	29.2	17.8

32	31	26.5	22.5	19.1	36.5	24.3	19.4	25.7	29.2	17.9
33	32	26.3	22.7	19	36.8	24.4	19.4	25.8	29.2	17.8
34	33	26.4	22.8	19.1	36.4	24.3	19.3	25.8	29.1	17.9
35	34	26.3	22.5	19.2	36.5	24.3	19.4	25.7	29.1	18.2
36	35	26.5	23	19.1	36.5	24.2	19.4	24.5	29.2	18.1
37	36	26.5	22.9	19	36.9	24.4	19.4	25	29.1	18.1
38	37	26.5	22.7	19.2	36.5	24.4	19.4	25.4	29.2	18.3
39	38	26.6	22.7	19.2	36.2	24.5	19.6	25.3	29.2	18.3
40	39	26.6	22.3	19.2	37.1	24.6	19.6	25.8	29.1	18.7
41	40	26.6	22.5	19.2	37	24.7	19.8	25.7	29	18.8
42	41	26.5	22.6	19.2	37.1	24.6	19.8	25.3	29	18.9
43	42	26.5	22.7	19.1	37.1	24.8	20	25.7	28.6	18.8
44	43	26.6	23.1	19.1	37.1	24.7	20.2	25.6	28.6	18.9
45	44	26.6	22.8	19.2	37.1	24.8	20.2	25.9	28.3	19.1
46	45	26.7	22.8	19.3	37.1	24.7	20.2	25.9	28.6	19.3
47	46	26.7	22.8	19.3	37.1	24.7	20.1	25.9	28.4	19.7
48	47	26.7	22.6	19.4	36.5	24.8	20.4	25.7	28.2	19.7
49	48	26.7	22.7	19.5	36.5	24.8	20.3	25.9	27.9	19.6
50	49	26.7	22.6	19.5	36.5	24.8	20.3	25.8	28.3	19.8
51	50	26.8	22.7	19.7	36.6	24.7	20.3	25.8	27.8	20
52	51	26.8	22.7	19.6	36.5	24.2	20.5	25.9	27.4	19.9
53	52	26.8	22.7	19.6	36.5	24.1	20.3	26	27.9	20
54	53	26.8	22.8	19.5	36.9	24.3	20.5	26.1	27.8	20.2
55	54	26.8	22.8	19.4	36.8	24	20.5	25.8	27.5	20.2
56	55	26.8	22.8	19.6	36.8	23.8	20.8	25.9	27.7	20.3
57	56	26.9	22.7	19.6	36.7	24	20.8	25.9	27.5	20.1
58	57	26.9	22.8	19.6	36.9	24.1	20.9	26	28.9	20.2
59	58	26.9	22.7	19.7	36.9	23.8	21	26.1	28.8	20.2
60	59	26.9	22.7	19.7	37	23.4	21	26.1	29	20.1
61	60	26.9	22.7	19.6	36.9	23	21	26	28.8	20
62	61	26.9	22.7	19.7	37	23.4	21.1	26	26.9	19.8
63	62	27	22.7	19.6	36.9	23.7	21.3	26	28.4	19.8
64	63	27	22.6	19.6	37	24.3	21.5	26.2	27.9	19.6
65	64	27	22.6	19.7	37	24.4	21.4	26.4	27.7	19.4
66	65	26.9	22.6	19.6	37.2	23.7	21.3	26.4	27.4	19.3
67	66	27	22.5	19.5	37.1	23.9	21.4	26.4	27.2	19.3
68	67	27	23	19.6	37.1	22.9	21.5	26.1	27.9	19.4
69	68	27.1	23	19.5	37.1	24.1	21.5	26.5	27.7	19.2
70	69	27.1	23	19.5	37.2	24.2	21.6	26.4	27.1	19.1
71	70	27.1	22.3	19.4	37.2	23.8	21.5	26.3	27.5	19
72	71	27.1	22.5	19.5	37.2	23.2	21.5	26.3	27.6	19.2
73	72	27.1	22.3	19.4	37.2	23.4	21.6	26.3	27.3	19.4
74	73	27.2	22.2	19.5	37.2	23.2	21.8	26.3	27.2	19.2
75	74	27.2	22.4	19.5	37.2	23.1	21.6	26.3	27.1	19.1
76	75	27.2	22.5	19.4	37.3	23.6	21.7	26.5	27.6	19
77	76	27.2	22.4	25.1	37.2	24	21.2	26.5	27.2	19.1

78	77	27.1	22.3	23.3	37.6	22.9	21.2	26.5	27.5	18.9
79	78	27.2	21.9	21.6	37.3	22.4	21.2	26.4	27.4	19.1
80	79	27.2	22.2	21.2	37.2	23	21.3	26.6	27.1	19
81	80	27.3	21.9	20.5	37.3	22.9	21.3	26.5	27.4	19
82	81	27.2	22.3	20.6	37.8	23.3	21.4	26.5	27.4	19.2
83	82	27.2	21.7	20.8	37.8	23.4	21.3	26.6	27.9	19.4
84	83	27.3	22.1	20.4	37.7	22.8	21.4	26.6	27.7	19.4
85	84	27.4	21.8	20.3	37.7	23.5	21.4	26.6	27.6	19.7
86	85	27.3	21.6	20.2	37.6	23.6	21.4	26.6	26.7	19.7
87	86	27.4	21.7	20.2	37.7	23.4	21.6	26.7	28.5	19.6
88	87	27.5	22.1	20.2	37.7	23.1	21.6	26.7	28.7	19.8
89	88	27.4	21.6	20.3	37.8	23.1	21.5	26.7	28.6	19.8
90	89	27.5	22	20.3	37.8	23.8	21.9	26.7	27	19.8
91	90	27.5	21.4	20.4	37.8	23.9	21.8	26.7	26.7	19.9
92	91	27.4	21.9	20.4	37.7	24	21.8	26.7	26.2	19.8
93	92	27.5	21.9	20.4	37.9	23.9	21.8	26.7	27.5	20
94	93	27.5	22.1	20.5	37.8	23.1	21.9	26.8	27.8	20
95	94	27.5	21.6	20.5	37.3	23.9	22.1	26.8	27.9	20
96	95	27.5	21.5	20.4	37.6	24.1	22.3	26.7	28.5	20.1
97	96	27.6	21.6	20.5	37.2	24.3	22.5	26.8	28.9	20.1
98	97	27.6	21.8	20.5	37.5	23	22.5	26.8	29.8	20.3
99	98	27.6	21.8	20.6	37.5	23.6	22.6	26.7	28.4	20.1
100	99	27.6	21.8	20.5	37.8	23.4	22.8	26.8	28.4	20.1
101	100	27.6	21.9	20.5	37.9	23.2	23.1	26.8	28.9	20.2
102	101	27.6	21.9	20.6	37.7	24.1	23	26.8	27.8	20.2
103	102	27.7	22.1	20.7	37.8	24.1	23.1	26.9	27.7	20.3
104	103	27.7	21.3	20.8	37.9	23.5	23	26.8	28	20.3
105	104	27.7	21.2	20.8	38	23.7	23	26.9	27.6	20.3
106	105	27.7	22.7	20.9	37.8	24.1	23	26.9	27	20.4
107	106	27.8	22.8	20.9	37.9	24.2	23.1	26.9	28	20.3
108	107	27.8	22.7	21.1	37.7	23.7	23.5	26.9	26.9	20.4
109	108	27.7	22.4	21.1	37.8	24.2	23.4	26.9	27.7	20.5
110	109	27.8	23.1	21.1	38	24.4	23.2	26.9	27.3	20.5
111	110	27.9	22.7	21.2	37.9	24.5	23.2	26.2	27.2	20.5
112	111	27.8	22.7	21.3	38.2	24.5	23.2	27	28.1	20.5
113	112	27.8	23.7	21.5	38.2	23.3	23.1	27	28.1	20.6
114	113	27.9	23.6	21.5	38.2	23.6	23.1	27.1	27.9	20.7
115	114	27.8	23	21.5	38.2	22.9	23.1	27.3	27.3	20.5
116	115	28	23.5	21.6	38.2	23.1	23	27.3	27.4	20.5
117	116	28	24	21.6	38.3	23.3	23.1	27.3	28.4	20.5
118	117	28	24.1	21.7	38.3	23.4	23.1	27.3	26.3	20.5
119	118	28	24.2	21.7	38.3	23	23.1	27	27.2	20.7
120	119	28.1	24.1	21.7	38.3	23.4	23	27	27.3	20.5
121	120	28	24.2	21.8	38.3	23.6	23.2	27.2	27.9	20.4
122	121	28	24.1	21.7	38.3	23.5	23.2	27.4	27.7	20.4
123	122	28	24.2	21.8	38.4	23.5	23.2	27.2	27	20.5

124	123	28	24.2	21.8	38.4	22.4	23.3	27	26.6	20.4
125	124	28.1	24.2	21.9	38.4	23	23.1	27.3	27.7	20.3
126	125	28.1	24.1	21.9	38.4	23.1	23.1	27.1	26.7	20.2
127	126	28	24.2	21.9	38.4	23.4	23.1	27.5	26.7	20.1
128	127	28	24.2	21.9	38.5	23.8	23.1	27.4	26.4	20
129	128	28.1	23.7	21.9	38.5	23.9	23.2	27.5	27.9	20
130	129	28.1	24.2	22	38.5	24.3	23.3	27.6	28.4	20
131	130	28.1	24.2	22.1	38.5	24.2	23.2	27.1	27.9	19.9
132	131	28.1	24.2	22.1	38.5	24	23.2	27.3	28.8	20
133	132	28.2	24.2	22.1	38.5	24.5	23.3	27.6	27.7	20.1
134	133	28.2	24.3	22.2	38.6	24.6	23.3	27.3	28.1	20.1
135	134	28.2	24.3	22.2	38.6	24.6	23.2	27.1	28.8	20.1
136	135	28.2	24.4	22.4	38.6	24.3	23.3	27.6	28.4	20.2
137	136	28.2	24.4	22.3	38.6	24.4	23.2	27.4	27.9	20.2
138	137	28.3	24.4	26.1	38.6	24.4	23.3	27.5	28.8	20.3
139	138	28.3	24.4	25.3	38.7	24.3	23.4	27.6	27.6	20.3
140	139	28.3	24.4	23.5	38.6	24.4	23.3	27.5	28.2	20.3
141	140	28.3	24.4	23.3	38.6	24.1	23.3	27.6	28.7	20.3
142	141	28.3	24.4	27.3	38.6	24.2	23.3	27.7	27.9	20.3
143	142	28.3	24.3	25.6	38.6	24.4	23.4	27.6	27.8	20.4
144	143	28.3	24.3	25	38.7	24.6	23.4	27.6	28.2	20.3
145	144	28.3	24.3	24.6	38.5	24.6	23.3	27.8	28.3	20.4
146	145	28.4	24.4	24.4	38.4	24.5	23.4	27.9	28.6	20.4
147	146	28.4	24.4	24	38.5	24.6	23.3	27.9	27.9	20.5
148	147	28.4	24.5	23.8	38.8	24.3	23.3	27.9	29.4	20.6
149	148	28.5	24.5	23.8	38.6	24.1	23.3	27.6	28.8	20.6
150	149	28.5	24.3	24	38.7	24	23.2	27.8	29.1	20.7
151	150	28.5	24.2	24	38.6	23.8	23.3	27.9	29.1	20.8
152	151	28.4	24.4	23.9	38.7	24.2	23.2	27.8	28.7	20.7
153	152	28.4	24.3	24.6	38.7	24.1	23.1	27.6	28.9	20.7
154	153	28.5	24.4	27.1	38.5	24.1	23.2	27.6	27.7	20.8
155	154	28.5	24.2	25.8	38.4	23.8	23	28	27.7	20.8
156	155	28.4	24	25.6	39	24.1	23.1	27.5	28.4	20.8
157	156	28.5	23.9	25.3	38.9	24.4	23.1	27.4	28.4	20.8
158	157	28.6	24	24.8	38.8	24.8	23.2	27.6	26.6	20.9
159	158	28.6	23.9	24.5	38.8	24.8	23.3	27.4	28.7	20.9
160	159	28.5	24.1	24.4	38.7	24.7	23.3	28.1	28.9	20.9
161	160	28.6	24.4	25.2	38.8	24.7	23.4	28.1	25.7	21
162	161	28.6	23.7	25.3	38.6	24.7	23.4	28.2	28.3	20.9
163	162	28.6	24	25.2	38.9	24.7	23.5	28.2	28.2	21
164	163	28.6	24.7	25.2	38.9	24.6	23.5	28.2	28.9	21
165	164	28.6	24.4	24.9	38.8	24.8	23.5	28.2	25.5	21.1
166	165	28.7	23.8	24.9	39	24.8	23.7	28.2	26.5	21.1
167	166	28.7	24.1	24.8	38.7	24.7	23.8	28.3	27.6	21.1
168	167	28.7	24	25.4	38.7	24.5	23.8	28.3	28.2	21.2
169	168	28.7	24.2	27	39.1	24.6	23.8	28.3	27.7	21.2

170	169	28.7	24.3	26.2	38.7	24.7	23.6	28.3	26.8	21.2
171	170	28.7	24.6	25.7	39.1	24.7	23.6	28.3	28.5	21.3
172	171	28.7	24.6	25.5	38.9	24.8	23.7	28.3	28.4	21.3
173	172	28.7	24.7	25.3	38.9	24.7	23.7	28.2	28.1	21.3
174	173	28.7	24.7	25.1	39	25.1	23.8	28.3	27.2	21.3
175	174	28.7	24.6	25	39.1	25.2	24	28.3	27.1	21.4
176	175	28.7	24.1	25	39	25.1	24	28.3	26.8	21.4
177	176	28.8	24.1	27.1	39.1	25.2	24	28.4	26.5	21.4
178	177	28.8	23.9	26.2	39.1	25.1	24	28.4	29.7	21.6
179	178	28.8	24.4	26	39.1	24.9	24.1	28.3	26.5	21.7
180	179	28.8	24.6	25.8	39.2	24.6	24	28.3	27.6	21.7
181	180	28.8	24.1	25.7	39.2	24.7	24.1	28.3	27.6	21.8
182	181	28.9	23.7	25.6	39.1	24.4	24.2	28.3	27.4	21.8
183	182	28.9	23.7	25.7	39	24.5	24.3	28.4	29.3	21.9
184	183	28.9	23.8	25.7	39.1	25.2	24.4	28.4	29.4	22
185	184	28.9	23.8	25.6	38.9	25.3	24.4	28.4	29.6	21.9
186	185	28.9	24	25.6	39.2	25.2	24.5	28.4	29	21.8
187	186	29	24	25.7	39.1	25.3	24.5	28.4	29.3	21.8
188	187	28.9	24.4	25.5	39.3	25.2	24.4	28.5	28.4	21.8
189	188	28.9	24.2	25.3	39.3	25.4	24.3	28.5	28.3	21.9
190	189	29	24.1	25.3	39.3	25.4	24.4	28.4	27.8	21.8
191	190	29	24	25.3	39.4	25.4	24.6	28.5	27.7	22
192	191	29	24.2	25.3	39.4	25.4	24.7	28.5	28.2	21.9
193	192	28.9	24.1	25.2	39.5	25.5	24.6	28.5	27.8	22.1
194	193	29	23.9	26.9	39.4	25.4	24.8	28.5	28.4	22.1
195	194	29	24.2	26.5	39.5	25.4	24.9	28.6	28.1	22.1
196	195	29	24.2	26.5	39.5	25.4	24.9	28.5	27.7	22.3
197	196	29.1	24.2	26.4	39.5	25.5	24.9	28.5	27.9	22.3
198	197	29	24.2	26.4	39.5	25.5	24.8	28.2	27.3	22.3
199	198	29	24	26.3	39.5	25.4	24.8	28.1	28	22.4
200	199	29	24.2	26.3	39.6	25.4	24.8	28.1	27.9	22.3
201	200	29.1	23.3	26.5	39.6	25.3	24.8	28.6	27.7	22.3
202	201	29.1	23.6	26.7	39.5	25.2	24.9	28.5	27.8	22.2
203	202	29.1	23.6	26.7	39.6	25	24.8	28.4	27.9	22.3
204	203	29.2	23.2	26.8	39.6	24.9	24.9	28.6	27.7	22.4
205	204	29.1	23.6	26.7	39.6	25.2	25	28.6	27.3	22.4
206	205	29.2	23.5	26.9	39.6	24.6	25.1	28.5	27.8	22.6
207	206	29.2	23	26.7	39.7	24.5	25.1	28.6	27.1	22.5
208	207	29.2	22.9	26.7	39.7	24.7	25.1	28.6	27.1	22.6
209	208	29.2	24.3	26.8	39.5	24.8	25.1	28.8	29.6	22.6
210	209	29.2	24.6	28.6	39.5	24.8	25	28.8	29.7	22.5
211	210	29.2	24.7	27.8	39.5	24.8	25.1	28.8	29.1	22.6
212	211	29.3	24.2	27.6	39.3	24.9	25.1	28.8	28.2	22.7
213	212	29.3	24.3	27.3	39.2	24.9	25	28.9	29	22.7
214	213	29.2	24.3	27.1	39.7	25.2	25	28.9	27.4	22.7
215	214	29.2	24.2	27.3	39.6	25.2	25.1	28.9	27.5	22.8

216	215	29.2	24.2	27.3	39.7	25.2	25.1	28.9	28.7	22.8
217	216	29.3	24.4	27.5	39.7	25.2	25.1	28.9	27	22.8
218	217	29.3	24.2	27.7	39.8	25.4	25.2	28.9	28.6	22.8
219	218	29.3	24.3	27.6	39.7	25.3	25.1	28.9	28	22.8
220	219	29.4	24.4	27.8	39.7	25.1	25.1	29	28.5	22.8
221	220	29.3	24.6	27.7	39.7	25.3	25.1	29	27.7	22.8
222	221	29.3	24.4	27.9	39.8	25.2	25.1	28.9	27.8	22.8
223	222	29.3	24.4	27.9	39.7	25.4	25	28.9	28.6	22.8
224	223	29.4	24.4	27.9	39.8	25.6	25.1	28.9	25.5	22.6
225	224	29.4	24.4	28.1	39.6	25.5	25.1	28.9	26.1	22.8
226	225	29.3	24.4	28.1	39.8	25.4	25.1	28.9	27	22.7
227	226	29.4	24.2	28.1	39.9	25.3	25.1	28.9	26.6	22.7
228	227	29.5	24.1	27.8	39.8	25.4	25.2	28.9	26.2	22.7
229	228	29.5	24.8	27.4	39.7	25.3	25	28.4	27.3	22.7
230	229	29.4	24.7	27.3	39.8	25.4	25.1	28.4	26.4	22.6
231	230	29.4	24.8	28.4	39.8	25.4	25.1	28.3	26.5	22.6
232	231	29.5	24.8	28	39.7	25.5	25.1	29.1	28.9	22.7
233	232	29.5	24.6	28.3	39.8	25.5	25.2	28.9	29.2	22.6
234	233	29.5	24.5	28.6	39.7	25.5	25.3	28.3	27	22.7
235	234	29.5	24.7	28.2	39.8	25.4	25.3	29	27.3	22.7
236	235	29.5	24.6	29	39.7	25.5	25.3	28.3	29	22.6
237	236	29.5	24.7	28.9	39.7	25.4	25.2	29	28	22.7
238	237	29.5	24.7	28.5	39.7	25.4	25.2	29.1	28.6	22.7
239	238	29.5	24.6	28.1	39.8	25.4	25.3	29.2	29.2	22.8
240	239	29.6	24.8	29.2	39.9	25.1	25.3	29.1	28.7	22.7
241	240	29.6	24.4	29	39.8	25.1	25.3	29.2	28.6	22.7
242	241	29.5	24.6	29.1	39.9	25.1	25.3	29.2	28.9	22.8
243	242	29.6	24.6	29.3	39.9	25.1	25.3	29.2	29	22.8
244	243	29.6	24.6	29.1	39.8	25.1	25.2	29.1	28.9	22.9
245	244	29.6	24.7	29.1	39.8	25.1	25.3	29.2	28.9	22.8
246	245	29.6	24.8	28.3	39.9	25.3	25.3	28.5	29.1	22.9
247	246	29.6	24.6	28.3	39.9	25.4	25.4	29.1	29	23
248	247	29.6	24.5	29.6	39.9	25.2	25.3	29.2	28.8	23.1
249	248	29.6	24.5	29.2	40	25.4	25.3	28.5	28.2	23.1
250	249	29.7	25	28.9	40	25.4	25.3	29.3	28.4	23.1
251	250	29.7	24.5	29.8	40	25.4	25.3	29.3	26.4	23.1
252	251	29.7	24.9	29.7	39.9	25.5	25.4	28.6	27.6	23.2
253	252	29.6	24.7	29.9	40.3	25.4	25.4	29.1	28.5	23.2
254	253	29.7	24.7	29.8	40.1	25.5	25.4	29.3	28.5	23.2
255	254	29.7	24.7	29.7	40.1	25.5	25.4	29.1	28.7	23.2
256	255	29.7	24.9	29.3	40.1	25.5	25.4	29.3	28.8	23.2
257	256	29.7	24.9	30.1	40	25.4	25.4	29.3	27.5	23.1
258	257	29.7	24.4	29.9	40.3	25.6	25.4	29.1	28.8	23.2
259	258	29.8	25.1	29.4	40.1	25.4	25.4	29.2	28.1	23.2
260	259	29.7	24.1	29.8	40.1	25.6	25.5	29.3	28.3	23.3
261	260	29.7	25	29.4	40	25.5	25.6	29.3	28.9	23.3

262	261	29.7	24.9	30.3	39.9	25.6	25.5	28.6	29.1	23.3
263	262	29.8	24.9	30.4	39.9	25.6	25.6	29.1	27.3	23.3
264	263	29.8	24.9	30	40.1	25.6	25.7	29.4	27.7	23.2
265	264	29.8	24.9	29.5	39.9	25.6	25.6	29.3	27.3	23.3
266	265	29.8	24.9	29.8	40.1	25.6	25.6	29.1	28	23.4
267	266	29.8	23.6	29.8	40	25.5	25.6	29.3	28	23.4
268	267	29.8	24.6	29.3	40.1	25.1	25.7	29.4	27.6	23.3
269	268	29.8	24.2	30.2	39.9	25.1	25.8	29.2	27.5	23.4
270	269	29.9	24.3	29.5	39.8	25.6	25.9	29.4	28	23.2
271	270	29.9	24.5	29.6	40.1	25.6	26.1	29.2	27.1	23.3
272	271	29.8	24.4	29.4	40	25.8	26	29.4	28.3	23.4
273	272	29.8	25.5	30.1	40.5	25.7	26	29.5	27.4	23.4
274	273	29.8	25.5	29.5	40.4	25.7	26.1	29.4	28.3	23.6
275	274	29.9	25.5	29.7	40.6	25.6	26.2	29.5	28.4	23.4
276	275	29.9	25.6	30.1	40.6	25.5	26.2	29.4	26.7	23.5
277	276	29.9	25.6	29.7	40.4	25.6	26.1	29.6	27.9	23.5
278	277	29.9	25.6	29.4	40.5	25.6	26.2	29.5	27.3	23.4
279	278	29.9	25.6	30.5	40.5	25.8	26.2	29.4	27.8	23.5
280	279	29.9	25.5	30.5	40.5	25.8	26.3	29.4	27.9	23.5
281	280	29.9	25.6	30.6	40.4	25.8	26.5	29.4	28	23.5
282	281	29.9	25.6	30.5	40.3	25.7	26.4	28.9	28.6	23.5
283	282	29.9	25.6	30.4	40.6	25.8	26.4	28.8	27.8	23.5
284	283	29.9	25.6	30.6	40.2	25.6	26.5	29.7	28.1	23.7
285	284	29.9	25.6	30.5	40.2	25.7	26.6	29.8	28.2	23.6
286	285	30	25.6	30.6	40.1	25.5	26.6	29.8	28.2	23.5
287	286	30	24.3	30.6	40.9	25.5	26.5	29.8	29	23.6
288	287	30	24.3	30.4	40	25.7	26.5	29.9	28.6	23.5
289	288	30	25	30.5	40.5	25.5	26.6	29.8	27.3	23.6
290	289	30	24.3	30.3	40.6	25.5	26.7	29.8	28.9	23.5
291	290	30	24.9	30.5	40.5	25.6	26.7	29.8	28.6	23.6
292	291	30	24.3	30.4	40.4	25.6	26.7	29.8	30.2	23.5
293	292	30	25.3	30	40.6	25.2	26.7	29.9	29.8	23.4
294	293	30	24.7	30.7	40.6	25	26.7	29.8	28.8	23.6
295	294	30	25.3	30.5	40.3	24.6	26.7	29.4	29.6	23.4
296	295	30	25.5	30.4	40.5	24.7	26.7	29.9	29.6	23.4
297	296	30	25.2	30.4	40.4	25.2	26.7	29.8	28.3	23.4
298	297	30.1	25.2	30.3	40.4	25	26.8	29.9	29	23.4
299	298	30.1	22.3	29.8	40.7	24.8	26.8	29.8	28.8	23.4
300	299	30.1	25.5	30.9	40.6	25.3	26.6	29.8	29	23.5
301	300	30	24.4	30.7	40.2	25.5	26.7	29.8	28.8	23.5
302	301	30.1	24.6	30.5	40.5	25.4	26.7	29.8	29.2	23.5
303	302	30.2	24.8	30.1	40.4	25.4	26.7	29.7	29.1	23.5
304	303	30.1	21.6	29.8	40.7	25.5	26.7	29.8	29.7	23.5
305	304	30	24.7	30.6	40.6	25.5	26.8	29.7	29.7	23.5
306	305	30.1	24.6	30.6	40.7	25.5	26.9	29.6	29	23.6
307	306	30.1	24.5	30.5	40.5	25.5	27	29.7	29.3	23.5

308	307	30.1	23.9	30.5	40.7	25.5	26.9	29.8	29.6	23.6
309	308	30.2	25.4	30.4	40.4	25.6	26.9	29.7	29.4	23.5
310	309	30.1	25.1	30.5	40.7	25.6	26.9	29	29.3	23.7
311	310	30.2	25.1	30.4	40.5	25.4	26.9	29.9	29.6	23.7
312	311	30.1	25.1	30.4	40.7	25.5	27	30	28.7	23.6
313	312	30.2	24.6	29.8	40.5	25.6	27.1	30.1	28.7	23.7
314	313	30.2	25.7	30.8	40.6	25.4	27	29.9	28.4	23.7
315	314	30.2	25.9	30.8	40.6	25.4	27	30	29.5	23.7
316	315	30.2	25.9	30.7	40.6	25.6	27	30.2	28.4	23.8
317	316	30.2	25.9	30.3	40.5	25.6	27.1	30.2	28.5	23.7
318	317	30.2	25.9	30.4	40.5	25.6	27.2	30.2	28.3	23.9
319	318	30.2	25.9	30.6	40.6	25.6	27	30.2	29.3	23.8
320	319	30.2	25.9	30.4	40.6	25.7	27.2	30.1	30.1	23.7
321	320	30.3	25.9	30	40.6	25.5	27.1	30.2	29	23.8
322	321	30.2	25.9	30.7	40.8	25.7	27.2	30.1	29.5	23.9
323	322	30.2	26	30.4	40.6	25.7	27.1	30.1	29.4	23.9
324	323	30.2	25.9	30.1	40.6	25.8	27	30	28.9	23.8
325	324	30.2	25.9	30.6	40.6	25.6	27	30	28.7	23.8
326	325	30.2	25.9	30.5	40.6	25.6	27	29.3	29.1	23.9
327	326	30.2	25.9	30.3	40.6	25.6	27.2	30.1	26.5	23.8
328	327	30.2	26	30	41	25.5	27.3	29.9	28.5	23.9
329	328	30.2	25.9	30.4	41.1	25.7	27.3	30.2	28.9	23.9
330	329	30.2	25.9	30.3	41	24.9	27.3	30.2	27	24
331	330	30.2	25.9	30.7	40.7	24.8	27.3	30.2	27.9	23.9
332	331	30.3	25.9	30.6	40.9	25.1	27.3	30.2	30.9	24
333	332	30.7	26	30.5	40.9	25.4	27.4	30.3	30.4	24
334	333	30.2	25.9	30.4	41.1	25.7	27.4	30.3	30.5	24
335	334	30.2	25.9	30.1	41.1	25.7	27.4	30.3	30	24.1
336	335	30.3	25.9	30.5	41.1	26	27.4	30.3	30.2	24.1
337	336	30.3	25.9	30.2	41	25.9	27.5	30.3	29.4	24.3
338	337	30.2	25.9	30.8	41	25.8	27.5	30.1	30.4	24.2
339	338	30.3	25.9	30.6	41	25.9	27.6	29.5	31.3	24.2
340	339	30.3	25.9	30.3	41	26	27.6	29.6	30.8	24.3
341	340	30.3	25.9	30.7	40.7	25.9	27.6	30.1	30.1	24.3
342	341	30.4	25.9	30.4	40.6	26.1	27.6	30.1	30.4	24.3
343	342	30.3	25.9	30.8	41	26.1	27.6	30	31.1	24.3
344	343	30.4	25.9	30.6	40.8	26.2	27.7	30.1	31.3	24.4
345	344	30.3	26	30.4	40.9	26.2	27.6	30.1	28	24.5
346	345	30.3	25.9	30.6	40.5	26.1	27.7	29.9	31.6	24.4
347	346	30.3	26	30.2	41.1	26	27.8	30.1	29.9	24.4
348	347	30.4	26	30.8	41	25.9	27.6	30	30	24.5
349	348	30.4	26	30.3	40.8	25.9	27.7	29.9	30.6	24.4
350	349	30.3	26	30.2	41	26	27.7	30.4	29.6	24.5
351	350	30.3	26	30.5	41.2	25.6	27.7	30.3	29.3	24.5
352	351	30.4	26	30.4	40.9	26	27.6	29.9	30.3	24.4
353	352	30.4	26	30.6	40.8	25.6	27.7	30.1	29.1	24.4

354	353	30.4	26	30.5	41	25.5	27.7	30.4	29.8	24.5
355	354	30.4	26	30.2	41.3	25.9	27.8	30.3	28.6	24.5
356	355	30.5	26	30.8	41.3	25.4	27.6	30.3	29.3	24.5
357	356	30.4	26.1	30.5	41.3	25.9	27.6	30.3	29.5	24.5
358	357	30.4	26	30.6	41.1	25.6	27.7	30.3	29.5	24.6
359	358	30.5	26	30.6	41.2	26.2	27.8	30.3	28.1	24.5
360	359	30.4	26.1	30.3	41.2	26.2	27.7	30.4	28.5	24.5
361	360	30.5	26	30.1	41.2	26.2	27.8	30.3	29.6	24.5

Appendix D – Product test report – graphene oxide dispersion

PRODUCT TEST REPORT

Graphene Oxide Dispersion (GO Disp.)

CGT_GO_102

Name of the Client	: University of Moratuwa
Ordered Quantity	: 1 L from each 0.5 g/L, 1 g/L, 2 g/L, 3 g/L dispersions
Batch Number	: CGT-GO-102
Date of Production	:15th February 2022

Equipment used

XRD : BRUKER D8 Focus X-ray diffractometer

RAMAN : BRUKER SENTERRA II -Confocal Raman Microscope

FTIR : BRUKER Vertex80 FTIR microscope (Hyperion)

Sample Details

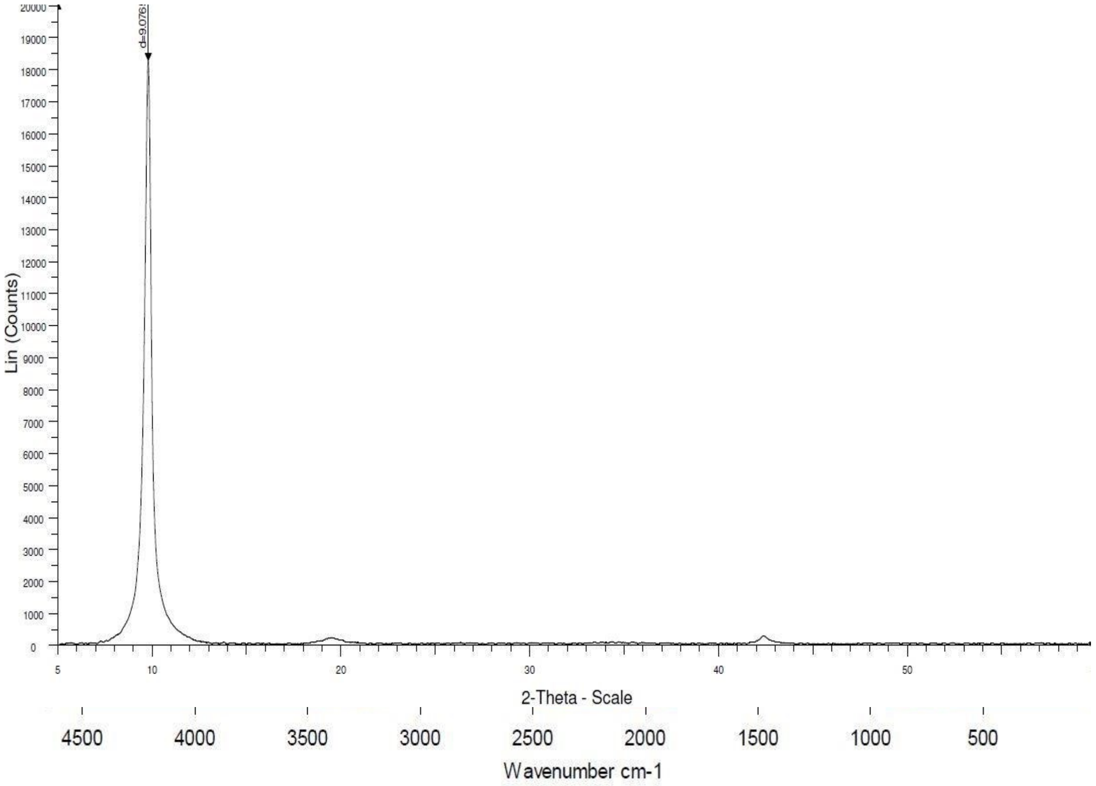
Start-up Graphite : Sri Lanka - C99+ Vein Graphite, Particle size range: 63-90 μm

Appearance : Brown color dry powder

Analysis and Results

X-ray powder diffraction (XRD)

The sample was mounted on sample holder. Parameters were set as follows, Cu Kα radiation (λ= 0.154 nm) over a 2θ range of 5−60° with a step size of 0.02° and a step time of 10 s.



d-spacing 9.0765

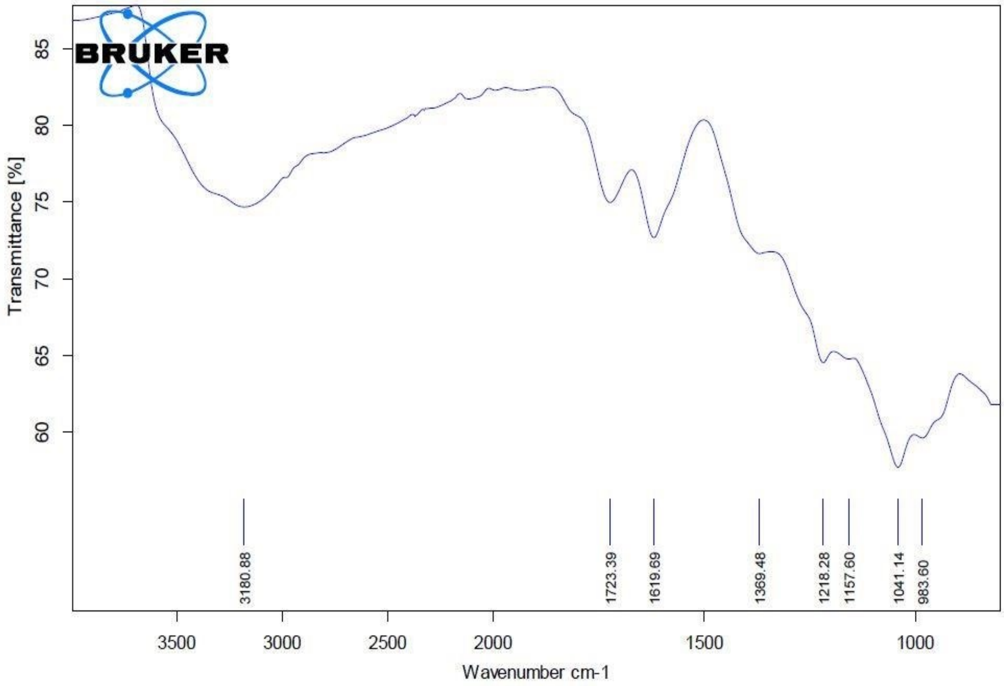
RAMAN Analysis

The sample was drop casted on the microscopic glass slides. Three different spots were analyzed. Parameters were set as follows, 532 nm; green laser was used with 50X optical zooming.

Average I_d/I_g ratio: 1.043

Fourier-transform infrared spectroscopy (FTIR)

Attenuated total reflection Fourier transform infrared (ATR-FTIR) spectra of the graphene oxide were recorded in the region 800 to 4000 cm^{-1} at a resolution of 4 cm^{-1}.



Analysis and Results of GO Dispersion

Form	Aqueous Solution
Color	Dark brown
Dispersibility	Dispersed in Water
Odor	Odorless
Concentration (g/L)	0.5 g/L, 1 g/L, 2g/L, 3 g/L
pH range (at 25 ˚C)	3 - 4

Tested by & for further Information:

Maheshika Premarathna

Quality Assurance Executive

Mob: +94 770835632 Email: maheshikap@lolc.com