



**LABORATORY WASTEWATER TREATMENT USING
CLAY BIOCHAR COMPOSITES IN
BIO-GEO FILTERS**

Vinitha Pathinayaka

(168419L)

Master of Science in Building Services Engineering

Department of Mechanical Engineering

University of Moratuwa

Sri Lanka

August 2021

**LABORATORY WASTEWATER TREATMENT USING
CLAY BIOCHAR COMPOSITES IN
BIO-GEO FILTERS**

Vinitha Pathinayaka

(168419)

Thesis submitted in partial fulfillment of the requirements for the degree
Master of Science in Building Services Engineering

Department of Mechanical Engineering

University of Moratuwa

Sri Lanka

August 2021

DECLARATION

"I declare that this is my own work, and that this thesis does not incorporate without acknowledgement any material previously submitted for a Degree or Diploma at any other University or institute of higher learning, and that it does not contain any material previously published or written by another person to the best of my knowledge and belief, except where acknowledgement is made in the text." In addition, I grant the University of Moratuwa the non-exclusive right to reproduce and disseminate my thesis in whole or in part in print, electronic, or other media. I reserve the right to use this content in whole or in part in future works (for example, articles or books)."

Signature:

Date:

Under our direction, the aforesaid applicant conducted research for her master's thesis.

Name of the supervisor:

Name of the supervisors:	Prof. Bandunee Athapattu	Dr.M.M. Inoka D. Manthilake
Signature of the supervisor:		
Date :		

ABSTRACT

Laboratory waste is overlooked because of its low flow rate, despite the fact that it has a negative influence on the human and environmental systems. As a result, the focus of this research is on using clay biochar composites in horizontal flow bio geo filters to treat university laboratory wastewater in an environmentally acceptable manner.

The composite was made with a 1:5, 1:3, 1:1, 3:1, and 5:1 mass ratio of Cinnamon biochar and Neem biomass to clay from Giant tank, Murunkan, Mannar for laboratory research experiments, and then treated with slow pyrolysis at 400°C. To determine the hydraulic retention period, adsorption kinetic studies and isotherms were performed, followed by the m/v ratio and COD test. For both Neem and Cinnamon, the Clay: Biochar mass ratios, 1:1 ratio composite exhibits superior efficacy in COD elimination.

To ensure the presence of Montmorillonite, clay samples were analyzed by Laser diffraction particle size analysis. As a result, clay samples taken from the Giant tank contain 3.42% and 4.99% nano clay, respectively. The existence of MMT in Murunkan clay is confirmed by FTIR readings of clay samples from Murunkan, which reveal a distinct and strong band at 998.03 cm^{-1} and 3620 cm^{-1} . XRF analysis was used to assess the chemical composition of biochar samples. The use of biochar with a greater K content supports heavy metal sorption and phosphorus retention. H/C ratios of Cinnamon and Neem biochars were 0.06 and 0.02 respectively, according to CHN analyses. BET study revealed that the specific surface area (SSA) of gasified Cinnamon Biochar was 563 m^2/g . As a result, it has a higher adsorption affinity. Kinetic model parameters for COD adsorption onto Neem - BC and Cinnamon - BC composites were determined using the most commonly utilized adsorption kinetic mathematical models. The removal effectiveness of adsorbent constructed of Neem biochar composite is better than that of Cinnamon biochar composite.

The Bio Geo Filter was created using Subsurface Flow Constructed Wetlands design principles. For the treatment system, a composite sample of on-site stored wastewater was diluted to 1:100. The system is based on a mix of physical, chemical, and biological processes that occur naturally in wetlands and are linked to vegetation, sediments, and the microbial populations that live there. The removal effectiveness of the system for heavy metals (Cd, Cr, Hg, and Mn) was investigated, and all heavy metal concentrations in effluent were much lower than in influent. The effluent quality was assessed and compared to CEA criteria for inland surface discharge. When water travels through the systems and into the tanks, only phosphate levels increase. As a result, methods for using this by-product (Phosphate) as fertilizer must be developed. As a result, our newly designed cost-effective bio-geo filter treatment system is highly recommended for laboratory wastewater purification.

Keywords: *Laboratory wastewater, Murunkan Clay, Biochar, Constructed wetland, Bio-geo composite, Kinetic models & Isotherms*

ACKNOWLEDGEMENT

First and foremost, I want to thank my advisors, Dr. Inoka Manthilaka, Course Coordinator, MSc in Building Engineering Services, Department of Mechanical Engineering, University of Moratuwa, and Prof. Bandunee Athapattu, Professor in Environmental Engineering, Department of Civil Engineering, The Open University of Sri Lanka, for their unwavering patience, motivation, enthusiasm, and immense support throughout my MSc study and research. Their advice aided me in finishing my research and producing this thesis on deadline.

Prof. T. Mangaleswaran, Vice Chancellor, University of Vauniya, and the Dean and Academic Coordinators of Faculty of Sciences (formerly Vauniya Campus, University of Jaffna) deserve special thanks for their support and sponsorship in putting this treatment system in place to treat their laboratory wastewater.

My sincere thanks goes to lectures of MSc in Building Services Engineering Prof. R.A.Athalage, Prof. Mahesh Jayaweera, Dr. Asanka Rodrigo, Dr. Anusha Wejewardana, Dr. Chatura Ranasinghe, Dr. Narein Perera, Eng. Samantha Gunawardana, Eng. J.P. Premarathna, Eng. Indradeva Mendis and Eng. Prasanna Narangoda for guiding me to improve the subjects relevant to MSc in Building Services Engineering which leads me working on diverse exciting project.

I'd like to thank the OUSL undergraduate students who helped me with this project, Ms. Karthika, Mr. Nimantha, Mr. Niruparan, and Ms. Sashika. Ms. Pradeepa Rajaguru, Senior Technical Officer, Department of Civil Engineering, OUSL, who assisted with this project also deserves my gratitude.

I am grateful to the Engineering Testing Laboratories and the employees of the Sri Lanka Institute of Nanotechnology, Homagama, and the Industrial Technology Institute for providing correct testing data in order to evaluate this research.

TABLE OF CONTENTS

DECLARATION	i
ABSTRACT	ii
ACKNOWLEDGEMENT	iii
TABLE OF CONTENTS	iv
LIST OF FIGURES	viii
LIST OF TABLES	xi
LIST OF ABBREVIATIONS	xiii
Chapter 1 INTRODUCTION	1
1.1 Introduction	1
1.2 Background	2
1.3 Problem Statement	4
1.4 Aim	5
1.5 Objectives	6
1.6 Methodology	6
Chapter 2 LITERATURE REVIEW	8
2.1 Laboratory Wastewater	8
2.1.1 Research Area of Concern	8
2.1.2 Pollutants in Laboratory Wastewater	8
2.1.3 Laboratory Wastewater Generation in Sri Lanka	9
2.1.4 Present Practice of University Laboratories	9
2.1.5 Roles of Analytical Laboratories	10
2.1.6 Effects of Heavy Metals in Laboratory Wastewater	11
2.1.7 Laboratory Wastewater Disposal Practices in Sri Lanka	12
2.2 Methods of Wastewater Treatment	12
2.3 Pollutant Removal Wetland Plants	15
2.4 Study of Biochar as Pollutant Remover	21
2.4.1 Properties of Biochars	21
2.4.2 Biochar Aromaticity and Elemental Ratios	22
2.4.3 Removal Mechanism of Heavy Metal by Biochar	24
2.4.4. Preparation Methods of Biochar	25
2.4.5. Characteristics	26
2.5 Study of Clay as Filter Media	28
2.5.1 Montmorillonite (MMT)	29
2.5.2 Characteristic of Murunkan Clay	30
2.6 Clay –Biochar Composite	31
2.6.1 Clay-Biochar Composite	31
2.6.2 Isotherm	32

2.6.3 Kinetics	33
2.6.4 Modifying Biochar	34
2.7 Constructed Wetlands	35
2.7.1 Constructed Wetland Systems	35
2.7.2 Classification of Constructed Wetland Systems	36
2.7.2.1 Surface Flow Systems	37
2.7.2.2 Subsurface Flow Systems	39
2.7.3 Metal Removal Mechanism in Wetlands	41
2.7.4 Design Approach for Horizontal Flow Constructed Wetland	42
2.8 Bio-Geo Filters	44
2.9 Conclusion of Literature	44
Chapter 3 MATERIALS AND METHODS	46
3.1 Methodology	46
3.2 Sample Collection of Raw Materials	47
3.2.1 Clay	47
3.2.2 Cinnamon Biochar	48
3.2.3 Neem Biomass	48
3.2.4 Calicut Tile	49
3.2.5 Wetland Plants	49
3.2.6 Aggregates	51
3.2.7 Data Collection of Laboratory Wastewater	51
3.3 Preparation of Sample	51
3.3.1 Preparation of Clay Biochar Composite	51
3.3.2 Adsorption Isotherm and Kinetic Studies	51
3.4 Characterization of Materials	52
3.4.1 Characterization of Clay	52
3.4.2 Characterization of Biochar	52
3.4.3 Characterization of Wastewater	52
3.4.4 Characterization of Filter Media	53
3.5 Methods	53
3.6. Design Approach	53
3.6.1 Hydraulic Design of Filter Media	54
3.6.2 Vegetation in Constructed Wetland	54
Chapter 4 RESULTS AND DISCUSSION	58
4.1 Characterization of Material	58
4.1.1. Characterization of Murunkan Clay	58
4.1.1.1. Laser Diffraction Particle Size Analysis	58
4.1.1.2 Fourier Transform Infrared Spectroscopy (FTIR) Analysis	58
4.1.2. Characterization of Biochar	60

4.1.2.1. X-Ray Florescence Analysis (XRF)	60
4.1.2.2. Carbon Hydrogen and Nitrogen (CHN) Analysis	61
4.1.2.3. Brunauer-Emmett-Teller (BET) Analysis	62
4.1.3. Characterization of Laboratory Wastewater	62
4.2. Design of the Treatment System	64
4.2.1. Design of the Filter Media	64
4.2.1.1. COD Removal Efficiency of Clay Biochar Composite	64
4.2.1.2. Adsorption Studies	65
4.2.1.3. Phosphate Releasement	70
4.2.2. Hydraulic Design of Prototype Treatment System	71
4.2.2.1. Subsurface Flow (SSF) Constructed Wetland System	71
4.2.2.2. Hydraulic Detention Time	71
4.3 Checking the Efficiency of the System	72
4.3.1 COD removal efficiency in the treatment system	72
4.3.2. Characterization of Treated Laboratory Wastewater	73
4.4 Checking the Efficiency of the System for Heavy metal Removal	73
Chapter 5 DESIGN AND IMPLEMENTATION OF BGF	76
5.1 Location and Area	76
5.2 Existing Condition of the Site	76
5.3 Design of Treatment System	79
5.4 Implementation	83
5.4.1 Preparation of Filter Media	83
5.4.2 Clay Biochar Composite Tiles Preparation	84
5.4.3 Preparation of Absorption Unit	84
5.4.4 Progress of the Implementation Work	85
5.4 5 Wetland plants	85
5.5 Monitoring of the performance	87
5.6 The implemented System	87
5.5 performance of the Treatment System	88
5.6 Cost Estimation	90
Chapter 6 CONCLUSIONS AND RECOMMENDATIONS	91
6.1 Conclusions	91
6.2. Recommendation	92
REFERENCES	93

ANNEXURES	102
Annex.1 General Standard Criteria (CEA)	102
Annex.2 Biochars' Basic Utility Properties (Test Category A)	103
Annex.3 Under the EBC Specification Following Parameters Have to be Tested for Biochar	104
Annex.4 Usage of Chemicals in Laboratory in University of Vauniya	105
Annex.5 Design Calculation for laboratory wastewater – University of Vauniya	111
Annex.6 Construction Drawings	115
Annex.7 Test Results	117
Annex.8 Cost Estimation	119

LIST OF FIGURES

Figure 1.1 Location Map of State Universities of Sri Lanka	3
Figure 2.1 A Typical Waste Chemical Neutralization System's Process Flow Diagram	13
Figure 2.2 Types of phytoremediation	16
Figure 2.3 Van Krevelen Diagram for Biochar and Biomass	23
Figure 2.4 Based on the Oxygen to Carbon Ratio in the Residual Solid Product, a Spectrum of Black C Products	24
Figure 2.5 Heavy metal removal using conventional methods	24
Figure 2.6 Schematic representation of the MMT structure	29
Figure 2.7 3D view molecular structure of MMT	30
Figure 2.8 Scanning Electron Microscope image of MMT	30
Figure 2.9 Biochar modification processes are classified using classification systems	35
Figure 2.10 Classification of Constructed Wetlands	37
Figure 2.11 Constructed Wetlands with Free Water Surface	38
Figure 2.12 Floating Treatment Wetlands	38
Figure 2.13 Constructed Wetlands with Horizontal Subsurface Flow	39
Figure 2.14 Constructed Wetlands with Vertical Flow	40
Figure 2.15 Hybrid CWs made up of a variety of different CW types	41
Figure 3.1 Flow Diagram of Research Methodology	46
Figure 3.2 Google Image of Sample Collected Location	48
Figure 3.3 Gasifier of the Heritage Kandalama	49
Figure 3.4 Image of <i>Cyperus corymbosus</i> (Gal ehi)	49
Figure 3.5 Image of <i>Vetiveria zizanioides</i> (Savandara)	50
Figure 3.6 Image of Cannas	50
Figure 3.7 Image of <i>Cosmos Sulphureus</i>	50
Figure 3.8 Image of Mustard	50
Figure 3.9 Image of Cattail	50
Figure 3.10 General Arrangement of Wastewater Treatment Tank	56
Figure 3.11 Image of Arrangements to Treatment	57
Figure 3.12 Treatment System Flow Diagram	57
Figure 4.1 Sample 01 Particle Size Analysis Graph	59
Figure 4.2 Particle Size Analysis Graph for Sample 02	59

Figure 4.3 Clay Sample from the Murunkan Area FTIR Spectroscopy	60
Figure 4.4 ppm Concentration there are fewer hazardous components	63
Figure 4.5 ppm Harmful and Toxic Elements Found in Lab Wastewater Concentration	63
Figure 4.6 Heavy Metal Concentration in the Laboratory Wastewater in ppm	64
Figure 4.7 Different Composites' Influence and Effluent Concentration on Cinnamon Biochar Composites	65
Figure 4.8 Different Composites' Influence and Effluent Concentration on Neem Biochar Composites	65
Figure 4.9 COD adsorption kinetic model parameters on Neem-BC and Cinnamon-BC composites at 320°C	66
Figure 4.10 For Neem – BC and Cinnamon –BC composites Pseudo First Order Kinetic Model was developed	67
Figure 4.11 For Neem – BC and Cinnamon –BC composites, a pseudo second order kinetic model was developed	67
Figure 4.12 For Neem – BC and Cinnamon –BC composites, the Freundlich Isotherm model was used	68
Figure 4.13 The Variation in COD Concentration over Time	69
Figure 4.14 COD Removal Efficiency with Time	70
Figure 4.15 Dissolved Phosphate Variation over Time in Various pH Conditions	70
Figure 4.16 The Treatment System in Cross Section	71
Figure 4.17 Percentage of removal effectiveness with tanks	72
Figure 4.18 Batch 15 & 16 Effluent	73
Figure 4.19 The Treatment System in Cross Section	74
Figure 5.1 Images of the Laboratory Building and Available land	77
Figure 5.2 Layout Plan of Ground Floor in Chemistry Laboratory	77
Figure 5.3 Layout Plan of 1 st Floor in Chemistry Laboratory	78
Figure 5.4 Layout Plan of Existing Sewerage System	78
Figure 5.5 Layout Plan of the Treatment Plant	81
Figure 5.6 Details of Elements of the treatment system	82
Figure 5.7 Cross section of absorption unit	83
Figure 5.8 Biochar prepared at the site	84
Figure 5.9 Preparation of composite tiles	84
Figure 5.10 R/F work for the absorption unit	85
Figure 5.11 Formwork for the absorption unit	85

Figure 5.12 Completed structure for the absorption unit	85
Figure 5.13 Laying of filter media	86
Figure 5.14 Before implementation of the treatment system	87
Figure 5.15 After implementation of the treatment system	87
Figure 5.16 After implementation of the treatment system at the rear	87
Figure A.5.1 Longitudinal section of absorption unit	115
Figure A.5.2 Cross section 5-5 of Absorption unit	116
Figure A.5.3 Cross section 4-4 of Absorption unit	116

LIST OF TABLES

Table 2.1. Chemicals Used in Analytical Laboratories	10
Table 2.2. Different Types of Technology for Wastewater Treatment in the Laboratory	14
Table 2.3 Minimization Programs for Laboratory Wastewater	15
Table 2.4 List of research that have been undertaken to remove H/M from contaminated soil	17
Table 2.5 Different types of adsorption	25
Table 2.6 Removal Mechanisms for Biochar-Based Adsorbents	25
Table 2.7. Comparison of Biochar Production Techniques	26
Table 2.8 Previous Studies of Biochar Preparation Method and Removed Heavy Metals	27
Table 2.9 Heavy Metal Removal Using Clay-Biochar Composites in the Previous era	34
Table 2.10 Heavy Metal Removal Mechanisms in Constructed Wetlands (Summary)	43
Table 2.11 Heavy Metal Concentration in Roots and Shoots of <i>Vetiveria zizanioides</i>	44
Table 3.1: Coordinates of Extracted Place	47
Table 4.1 Chemical Composition of Biochar Sample in Relation to Prominent Elements' Mass Percentage	61
Table 4.2 Elemental Concentrations of Carbon, Hydrogen, and Nitrogen in Relation to Mass Percentage	61
Table 4.3 Important Parameters of the Influent Wastewater	62
Table 4.4 Adsorption of COD onto Neem-BC and Cinnamon-BC composites kinetic model parameters	68
Table 4.5 Influence Sample Test Results	74
Table 4.6 Effluent Sample Test Results	74
Table 4.7 Tolerance limits for treated wastewater effluents under the Central Environmental Authority's General Standards	75
Table 5.1 Existing Fittings	79
Table.5.2 Additional Facilities for the Bio Chemistry Building	79
Table. A.1 General Standard Criteria for the Discharge of Industrial Effluent into Inland Surface Waters	102
Table. A.2 Test Category A: Basic Utility Properties (Required for All Biochars)	103
Table. A.3 Under the EBC Specification Following Parameters Have to be Tested for	

Biochar.	104
Table. A.4 Usage of Chemicals - Laboratory in Vavuniya University	105
Table. A.7 Test Results	117
Table. A.8 Cost Estimation	119

LIST OF ABBREVIATIONS

UGC	-	University Grant Commission
SLSI	-	Sri Lanka Standard Institute
NBRO	-	National Building Research Organization
NWS&DB	-	National Water Supply and Drainage Board
ITI	-	Industrial Technology Institute
CEA	-	Central Environmental Authority
SLLDLC	-	Sri Lanka Land Development Corporation
CECB	-	Central Engineering Consultancy Bureau
NERD	-	National Engineering Research and Development
OUSL	-	Open University of Sri Lanka
COD	-	Chemical Oxygen Demand
BOD	-	Bio Chemical Oxygen Demand
TDS	-	Total Dissolved Solids
TSS	-	Total Suspended Solids
SSA	-	Specific Surface Area
BC	-	BioChar
WHO	-	World Health Organization
UV	-	Ultra Violet
HDPE	-	High Density Polyethylene
IBI	-	International Biochar Initiative
MMT	-	Montmorillonite
XRD	-	X-ray diffraction
DTA	-	Differential thermal analysis
TGA	-	Thermogravimetric Analysis
FTIR	-	Fourier transform infrared spectroscopy
BET	-	Brunauer-Emmett-Teller (BET)
XRF	-	X-Ray Florescence Analysis
SEM	-	Scanning Electron Microscopy
CEC	-	Cation Exchange Capacity
FWS CW	-	Free water surface constructed wetlands
FTWs	-	Floating Treatment Wetlands
HSF CW	-	Horizontal Subsurface Flow Constructed Wetland
VF CWs	-	Vertical Flow Constructed Wetlands
HFSS	-	Horizontal flow subsurface