



EXTRACTION AND MODIFICATION OF LIGNIN RESIN FOR RUBBER COMPOUNDS

By

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Abstract

The aim of this work was related to development of economically viable method of extraction of lignin from sawdust in order to produce additive capable to improve physical mechanical and aging properties of rubber compounds. Five methods were developed and performed to isolate lignin from sawdust. They were delignification, acidolysis with 5M Hydrochloric acid, acidolysis with 10M Hydrochloric acid, ethanolysis and extraction with acetone. Chemical structure of produced lignin samples was identified with Fourier Transform Infra Red spectrometer (FTIR). Analysis of FTIR spectra showed good conformity of all wave numbers at which specified peaks of experimented and reference lignin samples were observed. Acidolysis of sawdust with 10M hydrochloric acid was found the most efficient for lignin extraction. This lignin was proceeded as a filler for rubber compounds. Properties of rubber compounds extended with produced lignin samples were determined. The test results showed that lignin was capable to improve properties of rubber compounds to the level achieved with carbon black and imported lignin filler samples, as well as resistance of rubber to prolong heat aging.

An attempt was made to synthesize commercial antioxidants for rubber from extracted lignin sample. Methodology was developed and performed. Synthesized products were 2,4,6 three-tert-butyl phenol and 2,6 di-tert butyl paracresol. Alcoholic and methoxyl groups present earlier in extracted lignin samples were replaced with methyl and tertiary butyl groups. These groups are particularly capable to donate hydrogen to an alkyl peroxy radical. The effects of substituent groups in the transition state involved not only electron transfer to the peroxy radical but electron delocalization in the aromatic ring. So synthesized products were expected to act as effective antioxidants in rubber compounds. Rubber compounds with experimented antioxidants were prepared and their properties carefully investigated. Test results showed excellent resistance of rubber compounds to heat aging, that proved with high level of confidence antioxidation activity of synthesized products.

I certify that this thesis does not incorporate without acknowledgement any material previously submitted for a Degree or Diploma in any university and to the best of my knowledge and belief it does not contain any material previously published, written or orally communicated by another person except where due reference is made in the text.

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LIST OF ABBREVIATION

DTA – Differential Thermal Analysis

DGA - Differential Gravimetric Analysis

FTIR – Fourier Transform Infra Red

MPa - Mega Pascal

M_w - Average Molecular Weight

NaOH- Sodium Hydroxide

NMR – Nuclear Magnetic Resonance

O₂ - Oxygen

phr - Parts Per Hundred

RSS - Ribbed Smoked Sheet

R[·] - Alkyl Radical

RH - Hydrocarbon Rubber

ROO[·] – Alkyl Peroxy Radical

HCl - Hydrochloric acid

MBTS –2,2 Dithio bis benzothiozole

UV - Ultra Violet

ZnO - Zinc Oxide

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GLOSSARY

Absorption – The physical mechanism by which one substance takes up another substance (liquid, gas or vapour) into its interior

Abrader - Abrasion testing machine.

Abrasion resistance – The ability of a rubber compound to resist mechanical wear.

Antioxidant – A compounding ingredient used to retard deterioration caused by oxygen.

Aging – To undergo changes in physical properties with age or lapse of time.

Accelerated Aging – Tests run on various rubbers to find out in as short period as possible the destructive influence of light, oxygen, heat and ozone.

Batch – The product of one mixing operation.

Compounding – Intimate mixing of a polymer with all the ingredients necessary for the finished article.

Compound – A term applied to a mixture of polymers and other ingredients to produce a useable rubber material.

Durometer – An arbitrary numerical value that measures the resistance to indentation of blunt indenter point of the durometer.

Dumbbell Specimen – A flat specimen having a narrow straight central portion of essentially uniform cross section with enlarged ends.

Evaporation – The direct conversion from liquid to vapour state of a given fluid.

Elongation at Break – General means ultimate elongation or percent increase in original length of specimen when it breaks/Extension produced by a tensile stress.

ISO – International Organization for Standard.

Internal mixer – Mixing unit for the production of rubber mixtures.

Mixing mill/ Two-roll mill – Driven pair rollers for the production of rubber mixtures as well as for the plasticizing, homogenizing, preheating and rough crushing of rubber.

Modulus – Tensile stress at specific elongation

PH – Determines the concentration of either an acid or a base.

phr – parts per hundred (by weight).

Plasticity – When subject to sufficient shearing stress, any given body will be deformed. After stress is removed, if there is no recovery, the body is completely plastic. If recovery is complete and instantaneous, the body is completely elastic. A balance between two is required.

Plastometer – An instrument for measuring the plasticity of a unvulcanized rubber compounds.

RSS – (Ribbed Smoke Sheet Rubber) a type of rubber available in the form of sheet that is ~~is~~ ^{dried} with smoke.

Reinforcement agent – Material dispersed in an elastomer to improve abrasion and tensile properties. Sri Lanka.

Scorch – Premature vulcanization of a rubber

Shore A – Unit of Hardness for soft rubber

Strain – Deflection due to force.

Stress – Force per unit of original cross section area.

Stress relaxation – Decreasing stress with constant strain over a given time interval (viscoelastic response).

Swell – Increased volume of a specimen caused by immersion in a fluid

Tensometer – a tensile testing machine.

Vulcanization – An irreversible process during which a rubber compound achieves its designed properties through a change in its chemical structure.

Vulcanizate – main product of vulcanization.

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