EFFECT OF REPLACEMENT OF CALCIUM CARBONATE BY FLY ASH ON PROCESSABILITY AND THERMO-MECHANICAL PROPERTIES OF SOLID TIRE MIDDLE COMPOUNDS

A. H. W. O. Sandaruwan

(158517C)

Thesis submitted in partial fulfilment of the requirements for the degree Master of Science in Polymer Technology

Department of Chemical and Process Engineering

University of Moratuwa

Sri Lanka

February 2020

DECLARATION OF THE CANDIDATE & SUPERVISOR

I declare that this is my own work and this thesis does not incorporate without acknowledgment any material 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 the University of Moratuwa the non-exclusive right to reproduce and distribute my thesis dissertation, in whole or in part in print, electronic or any other medium. I retain the right to use this content in whole or part in future works (such as articles or books).

Signature: Date:17/02/2020

The above candidate has carried out research for the master's dissertation under my supervision

Name of the supervisor: Dr. Susantha Siriwardhana

Signature of the supervisor: Date:17/02/2020

Name of the Co-supervisor:Dr. (Mrs.) Shantha Egodage

Signature of the Co-supervisor: Date: 17/02/2020

ACKNOWLEDGMENT

First, I would like to thank my supervisor Dr. Susantha Siriwardhana (Deputy Director-Research (Technology), Rubber Research Institute of Sri Lanka) who gave tremendous support and aligns me in the correct direction. His encouragement and support were one of the pillars which led me to complete the study. With his scientific and industrial background, he always guided me to complete the research in a scientific and holistic approach.

Also, my sincere thanks go to Dr. (Mrs.) Shantha Egodage (Senior Lecturer, Department of Chemical and Process Engineering, University of Moratuwa, Sri Lanka) for her excellent supervision and guidance were given to me throughout the project as the co-supervisor.

I would like to thank prof. Jagath Premachandra who was the course coordinator of Master of Science in Polymer Technology degree for his encouragement and guidance throughout the research to make it a success and complete.

Laboratories in the Materials Sciences and Engineering Department, University of Moratuwa were great pillars in the testing and analytical role of the research. So, I would like to thank the Head of the Department Mr. Sampath Weragoda for providing permission to conduct testing. Also, I would like to thank Mr. Bandu Samarasekara who was supporting the arrangement of the test environment and who is a senior lecturer in the same department for support and guidance.

Other all testings were conducted in Camso Loadstar (Pvt) Ltd. I would like to thank all the staff who helped to conduct the testings in the Research & Development Center and Ancillary Product, Division.

Finally, I would like to thank family members and friends for the support, devotion, and understanding throughout the research period.

ABSTRACT

In this study, as the first step, the effect of partial replacement of $CaCO_3$ with unmodified fly ash on the curing characteristics of the solid tire middle compound and the properties of its vulcanizate was investigated. Fly ash and $CaCO_3$ were first characterized. A series of solid tyre middle compounds with varying $CaCO_3$ / fly ash loading were prepared in a laboratory-scale internal mixer. The total content of non-reinforcement filler was kept constant. Fly ash loading was increased from 0 to 60 pphr intervals with replacing $CaCO_3$. The dispersion level was evaluated using the α view and SEM studies. Curing characteristics of the compounds were evaluated. Filler dispersion levels of the compounds and mechanical, rheological and physical properties of the respective vulcanizates were then focused. The unmodified fly ash filled rubber compound with optimum filler loading was selected based on the above properties.

In the second step of the study, the same studies were carried out with a smaller particle size (modified) fly ash filled NR compounds loaded with the selected filer loading.

It was shown that dispersion level was reduced with the addition of fly ash. Dispersion was improved with the incorporation of fly ash with reduced particle size and narrow particle size distribution. SEM studies showed a higher tendency of particle agglomeration with increasing loading of fly ash. It was found that M_H and M_L values of the rubber compounds were deteriorated with the increase of unmodified fly ash loading and the values were improved when fly ash with smaller particle size was used. However, no impact was observed on curing characteristics either with the replacement of $CaCO_3$ with fly ash, it's loading nor their sizes.

The hardness of the vulcanizates of the unmodified fly ash incorporated compounds was increased with the increase of fly ash percentage. Tensile strength, tearing strength, elongation at break and modulus showed a general reduction with the increase of unmodified fly ash except for tear strength, which had reduced only up to 45 pphr and shown a slight increase in 60 pphr of fly ash added sample. When the overall performance was considered, rubber compounds and vulcanizates prepared with 30 pphr fly ash loading showed the most comparable properties.

Studies carried out in the second stage, it was found that physical properties were improved with the use of fly ash with smaller particle size and narrow particle size distribution (0-53 μm) compared to unmodified fly ash incorporated compounds.

Rebound resilience values were reduced with the increase of the fly ash percentage. Dynamic mechanical properties such as heat build-up and blowout time were deteriorated with the addition of unmodified fly ash and results were improved with the use of small particles of fly ash. Tan delta value which depends on loss modulus and storage modulus shows a neutral role.

Based on the study, it was concluded that there is a potential to replace 50% w/w (i.e. 30 pphr) of the $CaCO_3$ loading from the fly ash with smaller size particles without a significant adverse effect on the curing, physical, mechanical and rheological properties.

Keywords: rubber compounds, fly ash, calcium carbonate, compound properties, dispersion, solid tyre middle compound

TABLE OF CONTENT

Page

LIST OF FIGURES

Page

LIST OF TABLES

Page

LIST OF ABBREVIATIONS

Abbreviation	Description
6PPD	N-(1,3-Dimethylbutyl)-N'-phenyl-p-phenylenediamine
ASTM	American Society for Testing and Materials
BO	Blow Out
CTP	N-(cyclohexyl-thio)phthalimide
DMA	Dynamic Mechanical Analyzer
et al	and others
HBU	Heat build-up
MDR	Moving Die Rheometer
M_H	Maximum Torque
M_L	Minimum Torque
NR	Natural Rubber
pphr	parts per hundred of rubber
PVI	Pre vulcanization inhibitor
Rpm	Revolutions per minute
SEM	Scanning Electron Microscope
SG	Specific Gravity
SIR	Standard Indonesia Rubber
SMR	Standard Malaysia Rubber
STR	Standard Thai Rubber
t_{90}	Optimum Cure Time
TBBS	N-tert-butyl-2-benzothiazolesulphenamide
TGA	Thermal Gravimetric Analysis
TMQ	Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline
t_{s2}	Induction Time
TSR	Technically Specified Rubber

LIST OF APPENDICES