

การเตรียมขงธรรมชาติ/คาร์บอนแบล็กคอมโพสิต โดยการจับตัวเชิงกลของลาเท็กซ์

นางสาว อริสรา ชะนะมา

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

สาขาวิชาปิโตรเคมีและวิทยาศาสตร์พอลิเมอร์

คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

ปีการศึกษา 2549

ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย



4 7 7 3 4 3 3 9 2 3

PREPARATION OF NATURAL RUBBER/CARBON BLACK COMPOSITE
BY MECHANICAL COAGULATION OF LATEX

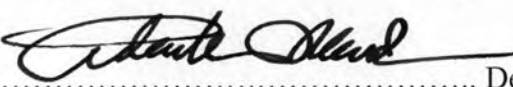
Miss Arisara Chanama

A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Petrochemistry and Polymer Science
Faculty of Science
Chulalongkorn University
Academic Year 2006
Copyright of Chulalongkorn University

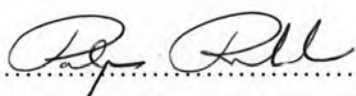
492276

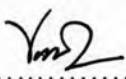
Thesis Title PREPARATION OF NATURAL RUBBER/CARBON BLACK
COMPOSITE BY MECHANICAL COAGULATION OF LATEX
By Miss Arisara Chanama
Field of study Petrochemistry and Polymer Science
Thesis Advisor Assistant Professor Varawut Tangpasuthadol, Ph.D.

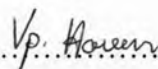
Accepted by the Faculty of Science, Chulalongkorn University in Partial
Fulfillment of the Requirements for the Master's Degree

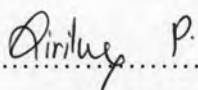

..... Dean of the Faculty of Science
(Professor Piamsak Menasveta, Ph.D.)

THESIS COMMITTEE


..... Chairman
(Professor Pattarapan Prasassarakich, Ph.D.)


..... Thesis Advisor
(Assistant Professor Varawut Tangpasuthadol, Ph.D.)


..... Member
(Assistant Professor Voravee P. Hoven, Ph.D.)


..... Member
(Sirilux Poompradub, Ph.D.)

อริสรา ชะนะมา: การเตรียมยางธรรมชาติ/คาร์บอนแบล็กคอมโพสิตโดยการจับตัวเชิงกลของลาเท็กซ์. (PREPARATION OF NATURAL RUBBER/CARBON BLACK COMPOSITE BY MECHANICAL COAGULATION) อ. ที่ปรึกษา: ผศ. ดร. วราวุฒิจึงพสุธาตล, 56 หน้า.

ได้เตรียมยางธรรมชาติ/คาร์บอนแบล็กคอมโพสิต ที่เตรียมโดยการจับตัวด้วยกระบวนการทางกลของน้ำยาง โดยใช้เครื่องผสมความเร็วรอบสูงแบบปิด สามารถทำให้เกิดการจับก้อนได้สำเร็จโดยใช้ความเร็ว 2,800 รอบต่อนาที ระยะเวลา 3 นาที ได้คอมโพสิตที่มีการกระจายตัวของคาร์บอนแบล็กในเมทริกซ์ยางไม่สม่ำเสมอ หลังจากนั้นของผสมยาง/คาร์บอนแบล็กที่จับตัวมาผสมกับสารเคียวซัลเฟอร์ด้วยเครื่องบดผสมสองลูกกลิ้ง พบว่าอนุภาคคาร์บอนแบล็กมีการกระจายตัวดีขึ้นในเมทริกซ์ยางความสามารถในการจับตัวด้วยกระบวนการทางกลขึ้นกับชนิดและปริมาณของคาร์บอนแบล็ก ซึ่งพบว่าคาร์บอนแบล็กชนิด N330 และ N550 ที่ปริมาณ 30 ส่วนต่อยางร้อยละ โดยน้ำหนัก ให้สมบัติเชิงกลด้านความต้านแรงดึงสูงที่สุด เมื่อเปรียบเทียบสมบัติเชิงกลระหว่างยางธรรมชาติ/คาร์บอนแบล็กหลังการวัลคาไนซ์ที่เตรียมจากการจับตัวเชิงกล และจากการผสมแบบปกติทั่วไปพบว่า ยางที่ได้จากการจับตัวเชิงกลมีสมบัติเชิงกลต่ำกว่าวิธีการผสมแบบปกติเล็กน้อย

สาขาวิชา.....ปิโตรเคมีและวิทยาศาสตร์พอลิเมอร์.....ลายมือชื่อนิสิต.....อริสรา ชะนะมา.....
ปีการศึกษา.....2549.....ลายมือชื่ออาจารย์ที่ปรึกษา.....

4773433923: MAJOR PETROCHEMISTRY AND POLYMER SCIENCE

KEY WORD: NATURUL RUBBER/CARBON BLACK/LATEX/COMPOSITE/
MECHANICAL COAGULATION

ARISARA CHANAMA: PREPARATION OF NATURAL RUBBER/CARBON
BLACK COMPOSITE BY MECHANICAL COAGULATION OF LATEX.

THESIS ADVISOR: ASST. PROF. VARAWUT TANGPASUTHADOL, Ph.D.,

56 pp.

Preparation of natural rubber/carbon black composites by mechanical coagulation of latex was carried out by using a high speed internal mixer. The coagulation was successfully obtained from the mixing speed of 2,800 rpm for 3 minutes. After coagulation, the carbon black particles were poorly dispersed in the rubber matrix. Better dispersion was later obtained when the coagulated NR/CB mixtures was mixed with sulfur curing chemical in a two-roll mill. Coagulation of the NR/CB mixtures was found to depend on types and quantities of CB. The CB content in the shear-induced vulcanizate was lower than the feed amount as analyzed by specific gravity. The vulcanizates containing 30 phr of N330 or N550 had the highest mechanical properties such as tensile strength. A comparison between NR/CB vulcanizates from both methods in terms of mechanical properties revealed that the shear-induced vulcanizates had slightly interior mechanical properties than the conventional ones.

Field of study Petrochemistry and Polymer Science Student's signature Arisara Chanama
Academic year 2006 Advisor's signature VS

ACKNOWLEDGEMENTS

I would like to express gratitude and deep appreciation to my thesis advisor, Assistant Professor Varawut Tangpasuthadol for his invaluable suggestion, guidance and kindness throughout this work.

I am sincerely greatly grateful to Professor Pattarapan Prasassarakich, Assistant Professor Voravee Hoven and Dr. Sirilux Poompradub for their invaluable comment and suggestions as the committee members.

I am thankful for my boss, Banja Junhasawasdikul, Distinguished Scholar, for financial support throughout master program. I also thank Sutin Chamulitrat and Chalermphol Wongdithnan for encouragement and gave me a chance to create this research, including the supports of experimental testing facilities at the R&D laboratory of Innovation Group. I also appreciate the technical assistance by Pachara Pongsaprakop and Rubber team students of Polymer Summer Camp 2006 (S. Srivimol, J. Suvimon, L. Satima, P. Kewalin, K. Ekwipoo, M. Arunsri, C. Phanutchakorn, K. Kanungnuch, C. Wanlapha, C. Veeraporn and D. Chokchai) This research would not have been possible without their support.

I am also thankful for supports from all my colleagues at Innovation group and members of Tangpasuthadol group in Organic Synthesis Research Unit for helping and warm friendship, staff of Science-Center for Analytical Service (Sci-CAS) for assistance with SEM.

Finally, I would like to thank my family and friends for their love and encouragement throughout my entire study.

CONTENTS

	Page
ABSTRACT IN THAI.....	iv
ABSTRACT IN ENGLISH.....	v
ACKNOWLEDGEMENT.....	vi
LIST OF FIGURES.....	x
LIST OF TABELS.....	xii
LIST OF ABBREVIATIONS.....	xiv
CHAPTER I INTRODUCTION.....	1
1.1 Statement of problems.....	1
1.2 Objective.....	2
1.3 Scope of the investigation.....	2
CHAPTER II THEORY AND LITERATURE REVIEW.....	3
2.1 Natural rubber latex	3
2.2 Fillers.....	5
2.2.1 Reinforcement of filler.....	5
2.3 Carbon black.....	6
2.3.1 Carbon black characteristics.....	6
2.4 Rubber to filler linkage mechanism.....	11
2.5 Mixing technology.....	12
2.6 Testing of unvulcanized samples.....	13
2.6.1 Mooney viscosity.....	13
2.6.2 Dispersion of filler.....	14
2.6.3 Cure characteristic.....	15
2.7 Science and Technology of NR/CB compound.....	15

	Page
2.7.1 NR/CB solid compound.....	15
2.7.2 NR/CB latex compound.....	16
2.8 Literature review.....	17
CHAPTER III EXPERIMENTS.....	19
3.1 Materials.....	19
3.2 Procedure.....	19
3.2.1 Preparation of NR/CB compound from shear-induced coagulation process.....	20
3.2.2 Vulcanization of NR/CB composite.....	20
3.2.3 Characterization of NR /CB compound.....	21
3.2.4 Characterization of NR/CB vulcanizates.....	22
CHAPTER IV RESULTS AND DISCUSSION.....	23
4.1 Shear-induced coagulation process	23
4.1.1 Distribution of CB in NR matrix.....	24
4.1.2 Mooney viscosity.....	26
4.2 Effect of mill-mixed shear on distribution in NR matrix.....	27
4.3 Curing of NR/CB composites.....	32
4.4 Specific gravity of compound.....	35
4.5 Mechanical testing of NR/CB vulcanizates.....	36
CHAPTER V CONCLUSION AND FUTURE DIRECTION.....	40
5.1 Conclusions.....	40
5.2 Future direction.....	40
REFERENCES.....	41

	Page
APPENDICES.....	45
APPENDIX A.....	46
APPENDIX B.....	48
APPENDIX C.....	50
APPENDIX D.....	51
VITAE.....	56

LIST OF FIGURES

Figure		Page
2.1	Classification of filler according to average particle size.....	6
2.2	Relevant carbon black dimensions.....	8
2.3	TEM of carbon black in many forms.....	9
2.4	Surface chemistry of CB.....	11
2.5	Molecular slippage model of reinforced mechanism based on rubber to filler linkage.....	12
2.6	Die and rotor of Mooney viscometer.....	14
2.7	Scanning process of SEM.....	14
2.8	Cure characteristic.....	15
2.9	Batch process for NR/CB masterbatch preparation.....	16
2.10	A continuous process for NR/CB masterbatch preparation.....	17
4.1	SEM micrographs of NR/N330 compound obtained from shear-induced coagulation after passing through the two-roll mill 10 times.....	25
4.2	SEM micrographs of NR/N550 compound obtained from shear-induced coagulation after passing through the two-roll mill 10 times.....	26
4.3	Mooney viscosity; ML1+4 (100°C) of NR/CB composites before vulcanization.....	27
4.4	SEM micrographs of vulcanized NR/N330 composites from solid NR (a-d), and from shear-induced coagulation (e-h).....	28

	Page
4.5-1 SEM micrographs of vulcanized NR/N550 composites from solid NR (a-d), and from shear-induced coagulation (e-g) at 5,000x.....	30
4.5-2 SEM micrographs of vulcanized NR/N550 composites from solid NR (a-d), and from shear-induced coagulation (e-g) at 10,000x.....	31
4.6-a Change in ML as a function of CB content in NR/CB.....	33
4.6-b Change in MH as a function of CB content in NR/CB.....	33
4.6-c Change in TS2 as a function of CB content in NR/CB.....	34
4.6-d Change in TC90 as a function of CB content in NR/CB.....	34
4.7-a Change in hardness as a function of CB content in NR/CB vulcanizates.....	37
4.7-b Change in tensile strength as a function of CB content in NR/CB vulcanizates.....	37
4.7-c Change in M300 as a function of CB content in NR/CB vulcanizates.....	38
4.7-d Change in tear strength as a function of CB content in NR/CB vulcanizates.....	38
4.7-e Change in elongation at break as a function of CB content in NR/CB vulcanizates.....	39

LIST OF TABLES

Table		Page
2.1	Composition of typical latex.....	4
2.2	Type of preservation system used in centrifuged concentrate...	4
2.3	ISO 2004 requirement for the centrifuged and cream concentrate latex.....	5
2.4	Carbon black Grades.....	10
3.1	Compound formulation for NR/CB compound from shear- induced coagulation.....	21
3.2	Compound formulation for solid NR.....	21
4.1	Coagulation results of latex mixed with different grades and quantities of carbon black.....	23
4.2	Curing characteristic of NR/CB compound before vulcanization.....	32
4.3	Specific gravity from experimental VS theory.....	35
A-1	Specification of CB.....	46
A-2	Specification of natural rubber latex.....	47
A-3	Specification of lab high speed intensive mixer model LMX5	47
B-1	Cure curve Entry 1-4.....	48
B-2	Cure curve Entry 5-8.....	48
B-3	Cure curve Entry 9-12.....	49
B-4	Cure curve Entry 14-16.....	49
C-1	Specific gravity calculation of rubber compound.....	50
D-1	The hardness of NR/CB vulcanizates.....	51

		Page
D-2	The tensile strength of NR/CB vulcanizates.....	52
D-3	The M300 of NR/CB vulcanizates.....	53
D-4	The tear strength of NR/CB vulcanizates.....	54
D-5	The elongation at break of NR/CB vulcanizates.....	55

LIST OF ABBREVIATIONS

NR	:	Natural rubber
CB	:	Carbon black
°C	:	Degrees Celsius
S	:	Sulphur
ZnO	:	Zinc oxide
phr	:	Part per 100 grams of rubber
rpm	:	Round per minute
HA	:	High ammonia
LA	:	Low ammonia
mm	:	Millimeter
nm	:	Nanometer
MPa	:	Mega Pascal
TEM	:	Transmission electron microscopy
ML	:	Mooney viscosity measured with large rotor
TS2	:	Scorch time

TC90	:	Cure time to 90% of max. Torque
g	:	Gram
kg	:	Kilogram
DSC	:	Dry solid content
TSC	:	Total solid content
VFA	:	Volatile fatty acid number