

**THIOACETATE- AND MERCAPTO-MODIFIED HYDROGENATED NATURAL
RUBBER AS COMPATIBILIZERS FOR RUBBER BLENDS**



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A Thesis Submitted in Partial Fulfillment of the Requirements
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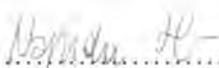
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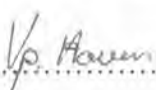
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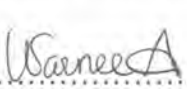

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ธีรัช พฤตศิริกุล : ยางธรรมชาติไฮโดรจิเนตคัดแปรด้วยหมู่ไทโอเอซีเทตและเมอร์แคปโตเป็นสารเสริมความเข้ากันได้สำหรับยางผสม. (THIOACETATE- AND MERCAPTO-MODIFIED HYDROGENATED NATURAL RUBBER AS COMPATIBILIZERS FOR RUBBER BLENDS) อ.ที่ปรึกษา : ผศ.ดร.นพิตา วิทยุธีระนันท์, 89 หน้า

ยางธรรมชาติอ่อนไวต่อการสลายตัวด้วยความร้อนและปฏิกิริยาออกซิเดชันเนื่องจากยางธรรมชาติมีพันธะคู่อยู่มาก ไฮโดรจิเนชันเป็นวิธีลดความไม่อิ่มตัวของยางธรรมชาติเพื่อให้มีความต้านทานต่อความร้อนและปฏิกิริยาออกซิเดชันได้สูงขึ้น ดังนั้นจึงคาดว่า การนำยางธรรมชาติไฮโดรจิเนตมาผสมกับยางที่ไม่อิ่มตัวสามารถปรับปรุงความต้านทานต่อความร้อนและปฏิกิริยาออกซิเดชันของยางผสมได้ อย่างไรก็ตามสมบัติเชิงกลและความเข้ากันได้ของยางผสมมีค่าต่ำ เนื่องจากความแตกต่างกันของระดับความไม่อิ่มตัวระหว่างยางเหล่านี้ ดังนั้นงานวิจัยนี้จึงศึกษาการคัดแปรโครงสร้างยางธรรมชาติไฮโดรจิเนตด้วยหมู่ไทโอเอซีเทตและเมอร์แคปโตเพื่อใช้เป็นสารเสริมความเข้ากันได้ ในยางผสมระหว่างยางธรรมชาติและยางธรรมชาติไฮโดรจิเนต ศึกษาผลของความเข้มข้นกรดไทโอเอซีติก ความเข้มข้นตัวริเริ่มปฏิกิริยา และเวลาที่ใช้ในการทำปฏิกิริยาต่อการคัดแปรยางธรรมชาติไฮโดรจิเนตด้วยหมู่ไทโอเอซีเทต (HNRTA) จากนั้นนำยางที่สังเคราะห์ได้มาทำปฏิกิริยาเมทาโนไลซิสในสารละลายผสมโซเดียมไฮดรอกไซด์และเมทานอล โดยปรับเปลี่ยนความเข้มข้นและเวลาเพื่อผลิตยางธรรมชาติไฮโดรจิเนตที่มีหมู่เมอร์แคปโต (HNRSR) วิเคราะห์โครงสร้างและปริมาณของหมู่ฟังก์ชันที่เกิดบนโครงสร้างด้วยเทคนิคอินฟราเรดสเปกโทรสโกปีและนิวเคลียร์แมกเนติกเรโซแนนซ์สเปกโทรสโกปี การเติม HNRTA หรือ HNRSR เพื่อเป็นสารเสริมความเข้ากันได้ ในยางผสมระหว่างยางธรรมชาติไฮโดรจิเนตและยางธรรมชาติสามารถเร่งการวัลคาไนเซชันและเพิ่มความทนทานต่อแรงดึงของยางผสมได้ ลักษณะทางสัญญาณวิทยาแสดงว่ายางที่มีหมู่ฟังก์ชันสามารถทำให้เกิดวัฏภาคต่อเนื่องบนพื้นผิวที่ได้จากการทดสอบความทนทานต่อแรงดึงของยางผสม ความเข้ากันได้แบบอ่อนโยนขึ้นได้จากการเพิ่มขึ้นของความหนาแน่นในการเชื่อมโยงและการลดลงของค่าการกระจายพลังงานจากการวิเคราะห์ความร้อนพลวัต นอกจากนี้ยางธรรมชาติไฮโดรจิเนตที่มีการเติมหมู่ฟังก์ชันยังสามารถปรับปรุงความต้านทานต่อการเสื่อมสภาพต่อความร้อนและโอโซนของยางผสมได้อีกด้วย

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THEERACHAI PRUTTISIRIKUL: THIOACETATE- AND MERCAPTO-MODIFIED HYDROGENATED NATURAL RUBBER AS COMPATIBILIZERS FOR RUBBER BLENDS. ADVISOR: ASSISTANT PROFESSOR NAPIDA HINCHIRANAN, Ph.D., 89 pp.

Natural rubber (NR) is deteriorated by thermal and oxidative degradation due to its high level of unsaturated carbon double bond. Hydrogenation is the technique that can reduce the unsaturation of NR and thus improve thermal and oxidative resistance. Therefore, the hydrogenated natural rubber (HNR) is expected to improve the thermal and oxidative resistance of rubber blends containing diene-based elastomers. However, the mechanical performance and the compatibility of the blends are poor due to the difference of the unsaturation level between these rubbers. Thus, the modification of the HNR structure with thioacetate and mercapto groups to be used as the compatibilizer for of HNR/NR blends was the aim of this work. The effect of thioacetic acid and initiator concentration including reaction time on the extent of reaction was investigated for the functionalized thioacetate in HNR structure. The methanolysis of thioacetate-modified HNR (HNRTA) in NaOH methanolic solution using varied concentration and reaction time provided mercapto-modified HNR (HNRSH). The structure and the content of additional functional groups on the resulting product were analyzed by FT-IR spectroscopy and $^1\text{H-NMR}$ spectroscopy. The addition of HNRTA or HNRSH as the compatibilizers in HNR/NR blends could accelerate the vulcanization and increased the tensile strength of the blends. A scanning electron micrographs demonstrated that these functionalized HNRs led a co-continuous morphology in the tensile fracture surface of the blends. The reactive compatibilization was also confirmed by the enhancement of crosslink density and the reduction of damping values characterized by dynamic mechanical analysis. Moreover, the functionalized HNRs improved the thermal and ozone resistance of the blends.

Field of Study: Petrochemistry and Polymer Science

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NOMENCLATURES

ACCN	:	1,1'-Azobis(Cyclohexanecarbonitrile)
CBS	:	<i>N</i> -Cyclohexylbenzthiazylsulphenamide
3-CPA	:	3-Chloropropionic acid
CV	:	Conventional Vulcanization
DCP	:	Dicumyl peroxide
DMA	:	Dynamic Mechanical Analysis
DSC	:	Differential Scanning Calorimetry
E'	:	Storage or Elastic Modulus
E''	:	Loss or viscous Modulus
EPDM	:	Ethylene – Propylene Copolymer
EPV	:	Efficient Vulcanization with Peroxide
FTIR	:	Fourier Transform Infrared Spectroscopy
HNR	:	Hydrogenated Natural Rubber
HNRTA	:	Thioacetate-modified HNR
HNRSH	:	Mercapto-modified HNR
MDR	:	Moving Die Rheometer
MH	:	Maximum Torque
ML	:	Minimum Torque
NMR	:	Nuclear Magnetic Resonance Spectroscopy
NR	:	Natural Rubber
phr	:	Parts per hundred of rubber by weight
TAA	:	Thioacetic acid

t_{c90}	:	Optimum cure time
T_g	:	Glass Transition Temperature
T_{id}	:	Initial Decomposition Temperature
T_{max}	:	Maximum Decomposition Temperature
t_{s2}	:	Scorch time
$\tan \delta$:	Loss Angle or Ratio of E'' to E'
TGA	:	Thermogravimetric Analysis
TMTD	:	Tetramethyl thiuram disulphide
ZnO	:	Zinc Oxide