

# Preparation and Characterizations of Natural Rubber from Natural Rubber Latex by Using Thermal Drying Method

## การศึกษาการเตรียมและสมบัติของยางธรรมชาติที่เตรียมจากน้ำยางสดด้วยวิธีการระเหยน้ำออกโดยการใช้ความร้อน

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### ABSTRACT

Natural rubber prepared from thermal drying technique was achieved. The range of drying temperature covered from 70 °C to 150 °C using local-made machine. The cure characteristic at 155 °C, mooney viscosity, mechanical properties (before and after aging) were investigated as compared to control rubber (derived from acid coagulation). It was found that mooney viscosity of thermal dried natural rubbers (TNR) was higher than that of acid coagulated rubber (ANR).  $T_2$  and  $T_{90}$  of TNR for all vulcanizing system were shorter than that of ANR. The average mechanical properties of TNR showed slightly lower tensile strength as compared to ANR. The differences in obtained properties should be claimed by the content of non rubber in rubber composition.

### บทคัดย่อ

งานวิจัยนี้ได้ศึกษาเทคนิคการเตรียมยางธรรมชาติด้วยวิธีการระเหยน้ำออกจากน้ำยางสดโดยใช้ความร้อนในช่วงอุณหภูมิ 70 °C ถึง 150 °C โดยพบว่ายางแห้งที่เตรียมด้วยวิธีการดังกล่าวจะให้ค่าความหนืดมูนนี่สูงกว่ายางธรรมชาติที่เตรียมด้วยวิธีการจับตัวด้วยกรด ในขณะที่ค่า scorch time, cure time และ tensile strength ของยางดังกล่าวนั้นมีค่าน้อยกว่า ยางที่จับตัวด้วยกรดเล็กน้อย โดยสมบัติที่แตกต่างกันของยางทั้งสองประเภทนั้นเป็นผลเนื่องมาจากการประกอบด้วย non rubber ซึ่งมีปริมาณแตกต่างกันออกไปตามเทคนิคที่ใช้ในการเตรียมยาง

**Key Words :** Drying process, NR properties, NR vulcanizate

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## Introduction

Natural rubber (NR) is a high molecular weight hydrocarbon polymer consisting mainly of the isoprene unit,  $C_5H_8$ . It made of Hevea tree with mostly cis configuration, having high green strength and good tack. However, NR products face the problem of inconsistency in its properties. These are major disadvantages of NR as compared to synthetic rubbers.

In previous works, Srichantamit and co-worker investigated the effects of non-rubber constituents on curing behaviour and properties of NR vulcanisates. They compared 3 types of NR, acid coagulated NR (ANR), NR containing non rubber constituents (WNR) and constant viscosity NR (CVNR). They found that vulcanization properties of WNR and CVNR are rather consistent with respect to seasonal variation and clones. They concluded that NR containing whole non rubbers exhibited no variation in cure time and scorch time (Srichantamit, 1991). Suchiva and co-worker studied the effect of non-rubber constituents on the network structures and properties of NR vulcanizates, by comparing the mechanical properties of purified NR (PNR) with WNR. They found that non-rubber constituents affect highly the curing of NR. PNR exhibited long delay in vulcanization compared with WNR, resulting in much longer vulcanization times. For PNR, their tensile and tear properties were generally smaller than those of WNR (Suchiva, 2000). Othman and co-worker studies the cure behavior of mixed PNR containing non rubber. They found that free fatty acid which derived from the hydrolysis of phospholipids and glycolipids, also affect the curing behavior of rubber. Torque

modulus increased while cure rate was slowed down. Surprisingly scorch time was not affected (Othman, 1988). Another previous work has reported that the processing and vulcanizing properties of NR are highly dominated by coagulation method, dilution effect and maturation periods. This is attributable to the variation in non rubber content. Non rubbers which have been extracted from differently processed rubbers gave different accelerating effect on the properties of NR (Boucher, 1964).

In the present work, the preparation of NR under thermal drying method was performed. The purpose focused on the production of NR with novel technology without using acid for coagulation. The properties of obtained NR and vulcanizate were evaluated and discussed comparing to ANR as used as control in this study.

## Experimental

### *Preparation of acid coagulated rubber (ANR)*

Natural rubber latex was diluted with an equal volume of distilled water, then added with 2% formic acid for coagulation. After that, the coagulum was sheeted and dried at 60 °C until clear color was observed.

### *Preparation of thermal dried natural rubber (TNR)*

TNR used as a raw material was prepared by casting NR latex on conveyer belt at given temperature (70 °C, 90 °C, 110 °C, 130 °C and 150 °C). Output rate of dried NR was set in the range of 4 to 20 g/min depending on quality of obtained NR. Both ANR and TNR were kept in desiccator for further use.

### **Preparation of vulcanized samples**

For sulfur vulcanization, the compound formulation is given in table 1. All sulfur curing systems have the same basic ingredients, except for the amounts of sulfur and accelerator that will be varied to prepare the samples of conventional vulcanization system (CV), semi - efficient system (semi-EV) and efficient system (EV). The ratios of sulfur : accelerator were also shown in Table 2.

### **Compounding**

The compounds were prepared in a laboratory two-roll mill, NR was first masticated for 1 min before charging the stearic acid and zinc oxide.

**Table 1** Compound formulation

Ingredients	Quantity (phr)		
	CV system	Semi-EV system	EV system
Natural rubber	100	100	100
ZnO	8	8	8
Steric acid	1	1	1
CBS	0.5	2	2.7
Sulphur	3	1.5	0.8

**Table 2** Compositions of vulcanization systems

Type	A/S
CV	0.1-0.6
Semi-EV	0.7-2.5
EV	2.5-12

Mixing was further carried out for 2.5 min. Finally, sulfur and accelerator were added and continued for 2 min.

### **Cure characteristics**

Scorch time ( $t_2$ ) and vulcanization time ( $t_{90}$ ) were measured by using the Monsanto Moving Die Rheometer (MDR 2000) according to ASTM D 2084-93. About 4 g samples of sulfur compounds were tested at 155°C.

### **Vulcanization process**

The compounds were sheeted in hydraulic press at 155°C. The optimal pressing time of sulfur vulcanizing compound were set following  $t_{90}$  obtained from MDR. Sample sheets were kept in desiccator before use.

### **Mooney viscosity**

For measuring the viscosity of TNR and ANR, mooney viscometer was used for measuring Mooney viscosity. About 20g. samples of sample were test at 100°C. Mooney viscosity at 4 min was recorded.

### **Mechanical properties**

The measurements of mechanical properties were carried out in accordance to ASTM D-412, using an Instron tensile testing machine model 5566, USA. Samples were cut into dumbbell shape. Cross head speed of 500 mm/min was used. Mechanical values were collected from average of 5 samples.

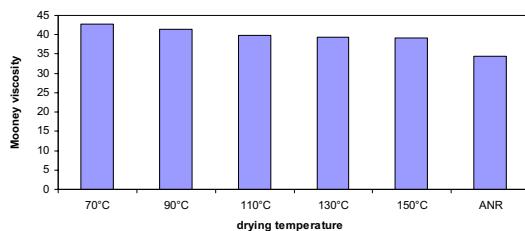
### **Ageing properties**

The dumbbell at same dimension were aged in an air oven for 22 h at 100°C. The aged samples were tested by tensile strength measurements. The strength values were compared with unaged samples.

## Results and discussion

### Effect of drying temperature on Mooney viscosity

The mooney unit value of TNR samples are exhibited in Fig.1. It is seen that the mooney viscosities of TNR are higher than that of ANR. For TNR, viscosity slightly decreases with drying temperature. This could be related to thermal degradation (chain scission) of NR molecules during preparation method (Chindaprasert, 1997).

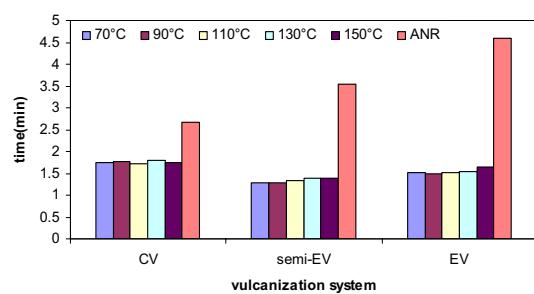


**Figure 1** The comparision of mooney viscosity between TNR and ANR

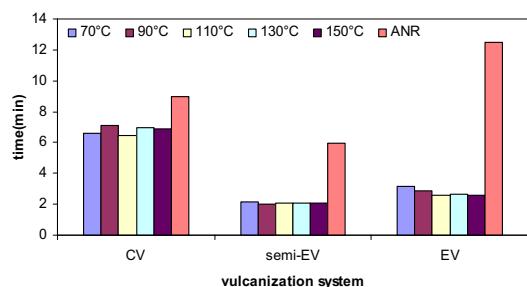
The higher mooney viscosity value observed with TNR was known to be amino acids and some inorganic constituents causing the formation of physical crosslinking (Jadsadaphongphaibool, 1993).

### Effects of curing system on cure characteristic of TNR

From Fig. 2-3, it is seen that drying temperatures do not significantly affect on  $t_2$  and  $t_{90}$  of TNR. On the other hand, in comparison between TNR and ANR, it is found that TNR exhibited faster  $t_{90}$  and  $t_2$  due to the non rubber constituents that accelerate the sulfur vulcanization rate and network structure formation (Jadsadaphongphaibool, 1993). Properties of vulcanized NR could be improved by controlling the amount of non rubbers in NR sample.



**Figure 2**  $t_2$  of sulfur vulcanization at 155 °C curing temperature



**Figure 3**  $t_{90}$  of sulfur vulcanization at 155 °C curing temperature

**Table 3** Tensile properties of sulfur cured

	Drying temperature(°C)	CV system		semi-EV system		EV system	
		no aging	aging	no aging	aging	no aging	aging
stress at break (MPa)	70	25.33	16.65	29.95	22.72	20.27	19.64
	90	25.15	18.86	30.46	24.51	20.94	22.99
	110	24.35	19.65	32.14	29.57	22.10	23.48
	130	24.97	19.30	28.79	26.66	23.83	26.17
	150	23.89	18.74	29.07	26.84	23.81	23.20
	ANR	28.19	21.81	30.02	26.06	21.48	22.40
modulus at 100% (MPa)	70	0.90	0.82	0.86	0.95	0.68	0.80
	90	0.90	0.83	0.86	0.99	0.68	0.80
	110	0.89	0.81	0.88	1.00	0.68	0.82
	130	0.92	0.84	0.91	1.01	0.69	0.86
	150	0.93	0.87	0.88	1.00	0.69	0.84
	ANR	0.95	0.95	0.90	0.95	0.73	0.72
%elongation	70	739.45	641.64	714.32	592.23	740.46	662.88
	90	734.06	667.44	718.36	594.86	762.58	685.27
	110	725.84	671.63	728.45	632.58	760.18	661.79
	130	728.81	691.50	683.47	609.38	774.70	654.93
	150	724.69	658.21	707.48	615.63	775.86	641.68
	ANR	713.35	620.04	689.37	605.42	720.10	686.04

#### **Effects of curing system on mechanical properties and aging properties of TNR**

Table 3 show tensile strength, modulus at 100% elongation and % elongation of sample prepared with different drying temperature. It is found that they were not so different. The results indicate that mechanical properties of TNR are slightly inferior as compared to ANR. It could be attributed to inorganic constituents acting as pro-oxidants in TNR. Previous studies have been reported that copper in fresh latex might form a complex with proteins and amino acids remaining in thermal dried natural rubber, could deteriorate

the NR vulcanize, especially under ageing condition of TNR (Boucher, 1964).

#### **Conclusions**

It could be concluded that natural rubber prepared by thermal drying method could be effectively performed and developed to be novel technology for NR production. The obtained properties are acceptable with more consistency as compared to conventional acid coagulation. The consistency in properties of vulcanized rubbers is obtained by controlling the amount of non-rubber in NR under thermal drying technique.

## References

Boucher, M., and Carlier, G. 1964. Rev.Gen.Cauch, 41, 1297.

Chindaprasert, S. 1997. Further Study to Apply Phase Transfer Technique for Surface Characterisation of Non-Crosslinked Natural Rubber Latex. Thesis in Polymer Science Faculty of Graduate Studies, Mahidol University.

Jadsadaphongphaibool, B. 1993. A Study of Preparation of High Quality Natural Rubber. Thesis in Polymer Science Faculty of Graduate Studies, Mahidol University.

Othman, AB., and Hasma, H. 1988. Proceedings International Rubber Technology Conference, 166-177.

Srichantamit, L. 1991. Effect of Nonrubber Constituent on Behaviour and Properties of Natural Rubber Vulcanisates. Thesis in Polymer Science Faculty of Graduate Studies, Mahidol University.

Suchiva, K., Kowitteerawut, T., and Srichantamit, L. 2000. Structure Properties of Purified natural Rubber. Journal of Applied Polymer Science, 78, 1495-1504.