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THE INFLUENCE OF MASTICATION TO CURING CHARACTERISTIC OF NATURAL RUBBER AND PHYSICAL PROPERTIES OF ITS VULCANIZATES

Abu Hasan[1], Rochmadi[2], Hary Sulistyo[2] and Suharto Honggokusumo[3]
[1]Chemical Engineering Department-State Polytechnic of Sriwijaya
Jl. Srijaya Negara Bukit Besar, Palembang 30139, Indonesia
[2]Chemical Engineering Department, Faculty of Engineering-Gadjah Mada University
Jl. Grafika No. 2, Yogyakarta 55281, Indonesia
[3]The Indonesian Rubber Association (Gapkindo)
Jl. Cideng Barat No. 62A, Jakarta 10150, Indonesia
e-mail : abu_hasan@polisriwijaya.ac.id

ABSTRACT

THE INFLUENCE OF MASTICATION TO CURING CHARACTERISTIC OF NATURAL RUBBER AND PHYSICAL PROPERTIES OF ITS VULCANIZATES. The research on the mastication that influences the curing characteristic of natural rubber and physical properties of its vulcanizates has been conducted. The research started from rubber mastication with variation of carbon black filler addition into rubber compound. Rubber compound was then measured to identify its curing characteristic using rheometer and tested its viscosity using Mooney viscosity tester. Physical properties of rubber vulcanizates such as abrasion resistance, tear strength, and elongation at break were also measured. The results indicated that curing characteristic tended to decrease, while only scorch time tended to increase from B1, B2, B3 and B4. Elongation at break tended to decrease, while tear resistance and abrasion resistance tended to increase from B1, B2, B3 and B4. Mooney viscosity of the compound also tended to decrease. It can be concluded that B1 is better than B2, B3 and B4. However, if rubber vulcanizates require high tear resistance and abrasion resistance, such as for shoes sole and tread of tire, then B4 is the most appropriate vulcanizate.

Key words : Carbon black, Curing characteristic, Mastication, Natural rubber

ABSTRAK

PENGARUH MASTIKASI TERHADAP KARAKTERISTIK PROSES *CURING* DARI KARET ALAM DAN SIFAT FISIK DARI HASIL VULKANISASINYA. Studi ini mempelajari pengaruh dari proses mastikasi terhadap karakteristik proses *curing* dari karet alam dan sifat fisis dari hasil vulkanisasinya. Percobaan diawali dengan proses mastikasi dari karet alam dengan memvariasikan *carbon black* sebagai material pengisi (*filler*). Material yang dihasilkan kemudian dianalisis untuk mengukur karakteristik dari proses *curing* dengan menggunakan *rheometer* serta diuji dengan *Mooney Viscosity tester*. Sifat fisis dari hasil vulkanisasi yang diuji termasuk ketahanan abrasi, ketahanan sobek, serta perpanjangan putus. Hasil uji menunjukkan bahwa *curing characteristic* cenderung menurun, sementara waktu pemanasan cenderung naik dari B1, B2, B3 dan B4. Perpanjangan putus cenderung menurun, sementara ketahanan sobek dan abrasi cenderung naik dari B1, B2, B3 dan B4. Viskositas *Mooney* dari campuran juga cenderung menurun. Dapat disimpulkan bahwa B1 lebih baik dari B2, B3, dan B4. Meskipun demikian, untuk hasil vulkanisasi yang memerlukan ketahanan sobek dan ketahanan abrasi yang tinggi, seperti untuk alas sepatu atau ban, maka B4 merupakan produk yang paling tepat.

Kata kunci : *Carbon black, Curing characteristic,* Mastikasi, Karet alam

INTRODUCTION

The use of filler on rubber compound is very important in determining the physical properties of vulcanizates and economical consideration of the products. In terms of economical aspect, filler reduces the price of rubber compound without decreasing the physical characteristics, and even there are synergies of reinforcement of certain physical properties of rubber vulcanizates. Particle size and structure of carbon black filler determine the physical properties of rubber vulcanizates. To function carbon black as reinforcing filler, it requires its total area and specific surface activity [1]. Filler addition into rubber compound in the form of carbon black, silica, and carbon-silica influenced dynamical properties of rubber vulcanizates [2,3], as well as rubber mastication procedure, i.e. the way to add carbon black filler into rubber. For filler added first to the rubber and oil was added after carbon black had been incorporated, the abrasion resistance was higher than that of carbon black and rubber mixed by oil and carbon black addition together [4].

Mixing sequence of ingredients and mixing sequence of coupling agent influencing the dynamical properties have been also studied [5]. The effort to know good filler dispersion was conducted by measuring carbon black flocculation. Flocculation was evaluated by the variation of the electrical resistivity of rubber samples aged under different conditions. There was a correlation between flocculation and bound rubber. Bound rubber influenced curing characteristic [6]. The filler dispersion of carbon black obtained by electrical measurement was found to be more dependent on the particle size [7, 8].

Here, carbon black type was used which had different structure N 326, N 330, and N 351 [9]. Carbon black type N 220 and N 550 was used to study curing characteristic of rubber. Types of carbon black influenced to the curing rate and curing time [10]. The existence of carbon black filler decreased the scorch time and functioned as a catalyst of the crosslink formation [11]. From this point of view, carbon black filler addition into rubber, type and structure of filler, and sequence of rubber mixing (mastication) were the most important factors to make the expected curing characteristic and physical and dynamical properties of rubber vulcanizates. However there are no thorough consideration concerning the method of carbon black addition into rubber.

Rubber mastication was the main purpose in this research, with the aim to study the filler addition into rubber and variation of mastication procedure. The filler used is carbon black type N 330.

EXPERIMENTAL METHOD

Materials

The main raw materials used in the experiment were natural rubber Ribbed Smoked Sheet (RRS-1) and carbon black filler type N 330. The chemical ingredients were sulfur, Tert Butyl Benzothiazole Sulfenamide (TBBS), Tetra Methyl Quinone (TMQ), ZnO, stearic acid and oil.

Equipment

The equipment used in the experiment are open roll mill, rheometer MDR 2000, temperature thermocouple, tensometer, Mooney viscosity equipment, tear strength tester and abrasion resistance tester.

Table 1. Natural rubber formula RSS-1

No	Ingredients	Phr
1.	RSS-1	100
2.	ZnO	5
3.	Stearic acid	2
4.	Carbon Black N 330	50
5.	Paraffinic oil	5
6.	TMQ	2
7.	TBBS	0,5
8.	Sulfur	2,5

Table 2. Sequence of rubber mastication RSS-1

No	Ingredients	Time, minutes
1	RSS-1	1
2	Stearic acid	1
3	Sulfur	2
4	ZnO	
5	CB_1	3
6	CB_2 + oil	7
7	TBBS	6
8	TMQ	
	Total	20

Procedure

Rubber mixture with the formula listed in Table 1 was masticated using open roll mill with sequence and time taken for each sequence of carbon black filler addition and other chemical ingredients shown on Table 2. Mooney viscosity of rubber compound was then measured.

Curing characteristic of rubber compound was also measured using rheometer at temperature 150 ^{o}C. The rheograph indicated the relation between vulcanization time and torque change on curing region. Based on this vulcanization time, the specimen can be cured to measure its physical properties.

Rubber formula in Table 1, especially Carbon Black-1 (CB-1) and Carbon Black-2 (CB-2) added on rubber mastication process were varied. There are four variation of carbon black addition into rubber: B1, B2, B3 and B4. B1 refers to the amount of carbon black filler 10 phr (part per hundred rubber) from the total of 50 phr

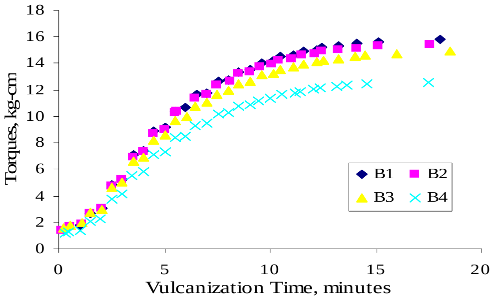

Figure 1. Torque as a function of vulcanization time of natural rubber compound RSS-1 on vulcanization temperature 150 ^{o}C

carbon black, added in the beginning of mastication process. Similarly, B2 refers to 20 phr the carbon black filler added in the beginning of mastication process from the total 50 phr, and the remaining carbon black is added afterward. The procedure of B3 and B4 are similar to B1 and B2 with 30 phr and 40 phr carbon black filler first addition, respectively.

Refer to rheograph on Figure 1, vulcanization time and temperature determined from the Figure 1 were used to produce and measure the physical properties of specimen namely elongation at break, abrasion resistance and tear resistance.

RESULTS AND DISCUSSION

The observation on the change of torque as the function of vulcanization time using rheometer is presented in Figure 1. Figure 1 shows the increase of torque that was in line with the increase of vulcanization time on curing region [12-17]. This torque change was caused by the increase of rubber stiffness that was proportional with the vulcanization reaction occured.

Sample B1 has the highest torque followed by B2, B3, and B4. It significantly indicated there was an influence of mastication procedure upon the physical properties of rubber vulcanizates. The more carbon black filler was added in the beginning of mastication process, the less its influence on the torque change was. It implied that the large number of carbon black filler in the beginning of mastication did not guarantee carbon black filler to be reinforcing filler.

There was a competition between carbon black particles, aggregates or its cluster to function as reinforcing filler. Only those that are closer to rubber backbone could function as reinforcing filler. If the number of carbon black particle was small, it was highly possible that carbon black filler was closer to rubber backbone so that they could function as reinforcing filler.

The relation between scorch time and mastication procedure was illustrated in Figure 2. Figure 2 showed that scorch time tended to increase from B1 to B4. Scorch time is the time required for rubber compound to flow in order to fill the most difficult part of the mould in the molding process of rubber goods industry. The higher the scorch time the slower the vulcanization reaction was, but the more chances for rubber compound to flow to the most difficult part of mould. Scorch time also influenced the lapse between maximum torque and minimum torque. The higher the scorch time was, the smaller the lapse between maximum torque and minimum torque was. A high scorch time indicates that vulcanization reaction proceeds slowly and insignificantly influence the torque of rubber vulcanizates. The lapse between maximum torque and minimum torque is illustrated in Figure 3.

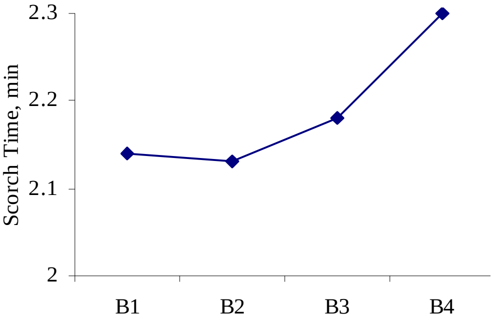

Figure 2. Scorch time as a function of mastication procedure.

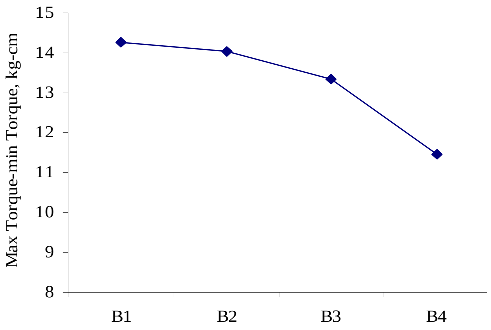

Figure 3. Maximum torque-minimum torque as a function of mastication procedure.

The relation between curing rate and mastication procedure was shown in Figure 4. Curing rate was calculated using Equation (1) as follows [6]:

$$Curing\ rate = \left[\frac{(Torque_{\max} - Torqur_{\min}}{Opt.Curing - ScorchTime} \right] \quad .... \quad (1)$$

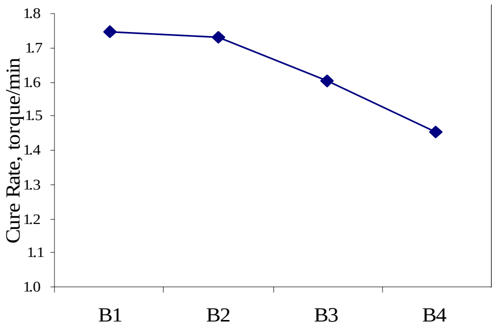

Figure 4. Cure rate as a function of mastication procedure.

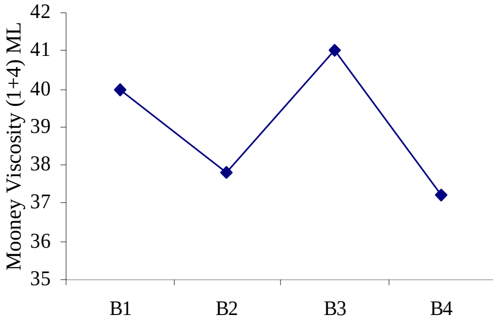

Figure 5. Mooney viscosity as function of mastication procedure.

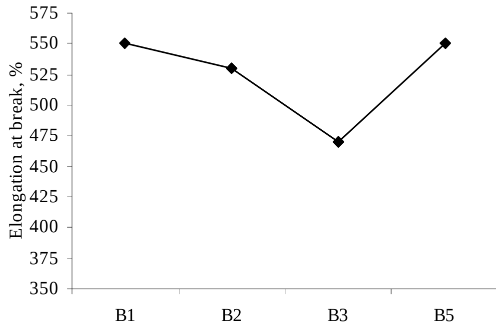

Figure 6. Elongation at break as function of mastication procedure.

Figure 4 indicates the reduction of curing rate starting from B1 to B4. Rubber mastication procedure influenced the curing rate. The more carbon black was added in the beginning process of mastication, the smaller the competition of carbon black filler to function as reinforcing filler was, and the smaller the chance of carbon filler to be closer to rubber backbone. The increase of maximum torque and scorch time caused the low curing rate.

The relation between Mooney viscosity and mastication procedure was illustrated in Figure 5, which showed the tendency of Mooney viscosity to decrease on B1 to B4. Here, the number of carbon black filler plays a significant role in the beginning of mastication process to make rubber softening. Particles of carbon black filler that sheared between rubber backbones in the mastication process can play a role in tearing up the rubber molecule chain besides shearing force of roll mill. Rubber softened and its viscosity decreased.

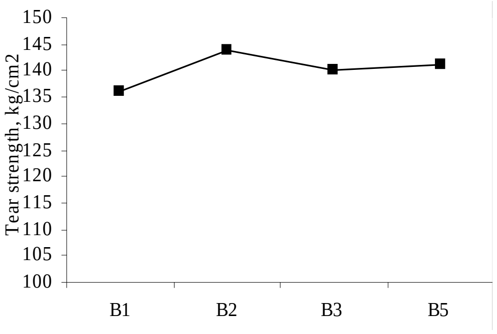

Figure 7. Tear resistance as function of mastication procedure.

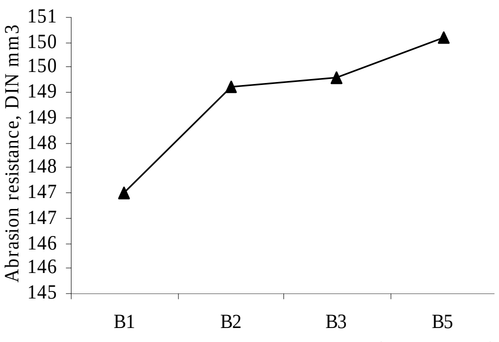

Figure 8. Abrasion resistance as function of mastication procedure

The relation between elongation at break and mastication procedure was illustrated in Figure 6, which showed the decrease of elongation at break starting from B1 to B4. The larger number of carbon black filler added in the beginning process of mastication can cause the spaces between rubber backbones fully filled so that rubber vulcanizates becomes stiffer. The increase of rubber vulcanizates stiffness decreases elongation at break or there are more carbon black filler that cannot function as reinforcing filler on rubber vulcanizates. The increase of stiffness on rubber vulcanizates can increase tear strength and abrasion resistance. High tear resistance and abrasion resistance are required for rubber goods such as shoe sole and tread of tire.

The relation between tear strength and abrasion resistance and mastication procedure are shown in Figure 7 and Figure 8.

CONCLUSION

Mastication procedure of rubber compound B1 to B4 influenced the curing characteristic. Mooney viscosity and the physical properties of rubber vulcanizates. Maximum torque-minimum torque and curing rate tended to decrease while scorch time tended to increase. Mooney viscosity also decreased from B1 to B4. Tear resistance and abrasion resistance tended to increase while elongation at break tended to decrease from B1 to B4. In general, B1 was better than B2, B3, and B4. However, if rubber vulcanizates require high tear resistance and abrasion resistance, such as for shoes sole and tread of tire, B4 is the most appropriate vulcanizate.

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