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Effect of Carbon Black Types on Curing Behavior of Natural Rubber

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ABSTRACT

This research extensively investigated to improve the processing characteristics of natural rubber NR by insertion various types of carbon black. The average of nitrogen surface area (34 for N660, 76 for N330, 77for N326, and 91for N375) m^2/g and range of particles size (26 -63)nm high abrasive furnace. The curing kinetics was determined using the rheometer with an oscillating disk, in which the network formation process is registered by the torque variation during time. The vulcanization was obtained in a hydraulic press at 185 °C. OSD rheometer results clearly indicate that optimum cure time, scorch time and cure rate (100/t90-ts2) are exponentially decreases with increasing in particles surface area. However, increase in Max., Min. torque of OSD rheometer, reversion period time of cure characteristics and Mooney viscosity ML (1+4) at 100C° with increasing the surface area. This result clearly indicates the improvement of curing characteristics.

Key words: Curing, carbon black, natural rubber, rheometer

INTRODUCTION

Rubber composites have been developed to meet several industrial requirements, such as the need for easier processing and broadening the range of properties, either by varying the type, relative content or the morphology of each component [1].Nowadays, considerable research interest is focused on new enhancement for rubbery materials obtained by addition a various types of Carbon black. The major feature of such process is improve the processing characteristics and the mechanical properties [2]. Use of carbon black reinforced natural rubber is very common in automotive applications especially suspension top cups, cab mounts, suspension bushes, engine mounts etc Carbon black plays key role in the alteration of the rubber compound properties to suit the end product requirements for hysteresis, stiffness, hardness, compression set etc.[3]. Natural rubber is one of the most important agricultural products in Thailand which is currently the world largest natural rubber producer, with the production capacity of 1.8 million tons per year (Rubber Research Institute, 1999) [4]. As an elastomer, natural rubber is widely used in various applications, due to the fact that elastomers possess unique a properties, such as their ability to undergo a large elastic deformation and to absorb energy. Natural rubber products have been used in commodity applications, such as shoes, tires, rubber bands, tubes, etc. Recently, the applications of natural rubber have been expanded into engineering purposes, for example, machine parts, construction parts, automotive parts. For engineering applications, not only mechanical properties, but also dynamic mechanical properties at a sensible range of temperature, must be taken into account. It is of necessary to provide adequate data for engineers in designing rubber products with the required performance at all service temperatures [5].

MATERIALS AND METHODS

Materials

Natural rubber with grade Standard Malaysian Rubber (SMR20) with SP. gravity 0.90, volatile matter 0.5%max, Ash 1%max, Nitrogen content 0.5%max. Carbon black N326 with surface area 77m^2/g, ID No(82),DBP(72) , pure density (446-470)kg/m^3,partical size (26-30nm) ,high abrasive furnace .Carbon black N330 with pure density (370-393)kg/m^3,surface area 76 m^2/g ,ID No(82),DBP(102) ,particle size (26-63) high abrasive furnace . carbon black N375 with surface area 91 m^2/g , ID No(90), DBP(114) ,pure density (345-350) kg/m^3,particle size (26-30) high abrasive furnace . carbon black N660 with surface area 34 m^2/g , ID No(36) ,DBP(90) pure density (432-458)kg/m^3,particle size (49-60) general purpose furnace . ZnO with surface area 62 m /g, purity of zinc oxide 99% Min, SP. gravity 5.5. Stearic acid with SP.gravity0.85, Ash at 550C° 0.1% Max. , volatile matter at 65C° 0.5% Max. Insoluble sulphur with SP.gravity1.57, sulpher content 80±2%, Ash at 550C° 0.2% Max, volatile matter at 80C° 0.5% Max.

Mixing Process

SMR_{20}-masterbatch were prepared in a two roll mill at ~ 50°C according ASTM D15 a Homogeneous mixture and minimize the influence of mixing conditions. The vulcanization systems, including the various grade of particles carbon black with different Nitrogen average surface area are adding to natural rubber compounds were added in two-roll mill. The compounds were sheeted off at a thickness of approximately 2 mm, which was convenient for the subsequent preparation of test specimens. The compositions of SMR20 compounds with various grade of particles carbon black at different nitrogen average surface area are given in Table.1.

Table -1 Rubber compounds model with different average NSA of particles carbon black (amounts in pphr)

Materials Compound	C.B N660 with NSA($34m^2/g$)	C.B N330 with NSA($76m^2/g$)	C.B N326 with NSA($77m^2/g$)	C.B N375with NSA($91m^2/g$)
Standard Malaysian Rubber (SMR20)	100	100	100	100
Carbon Black	45	45	45	45
ZnO	3	3	3	3
Stearic acid	1	1	1	1
Process oil	5	5	5	5
MBT	0.5	0.5	0.5	0.5
TMTD	1	1	1	1
6PPD	1	1	1	1
Sulfur	2	2	2	2

Curing Characteristics

The cure characteristics of the different compounds were measured at 185°C and 6 min with a MV-ODR-PROPERTIES (Micro vision Enterprises-India) according ASTM D2705a type of moving die rheometer. The optimal vulcanization time (t90) and scorch time (t02) of the compounds were determined. The compounds were cure in laboratories of Materials Engineering Department of Nonmetallic Engineering pressing at 140°C and 100 bar, according to the t90 of the specific compounds.

Oscillating Disc Rheometer

The oscillating Disc type Rheometer is efficient, simple and reliable testing equipment. It is quite easy to operate. The Rheometer describes precisely and quickly curing and processing characteristics of vulcanizable rubber compounds. It works on a very simple principle. A test piece of rubber compound is contained in a sealed test cavity under positive pressure and maintained at a specified elevated temperature. A Rotor (bucolicaldisc) is embedded in the test piece and is oscillated through small specified rotary amplitude. This action exerts a shear strain on the test piece and the torque (force) required to oscillate the disc depends upon Stiffness (shear modulus) of the rubber compound.

IS0 3417, ASTM D 2084, BS 903: Part A60: Section 60.2 The oscillating disc cure meter or rheometer (ODR) solves the problem of not being able to make any rheological measurements after the scorch time (as with the Mooney viscometer), by changing the rotor from a rotating mode to an oscillating one. Since cured rubber can stretch to some extent without breaking, the oscillations arekept within this limit. The magnitude of the oscillation is measured in degrees of arc, 1' and 3' are most common, and the rate of oscillation is suggested as 1.7 Hz. The cure meter is an essential piece of equipment and used extensively in the rubber laboratory. The machine plots a graph of torque verses time for any given curing temperature. The full extent of cure and beyond can now be recorded. For example, reversion, the point at which the vulcanized compound breaks down due to prolonged heating can now be measured.

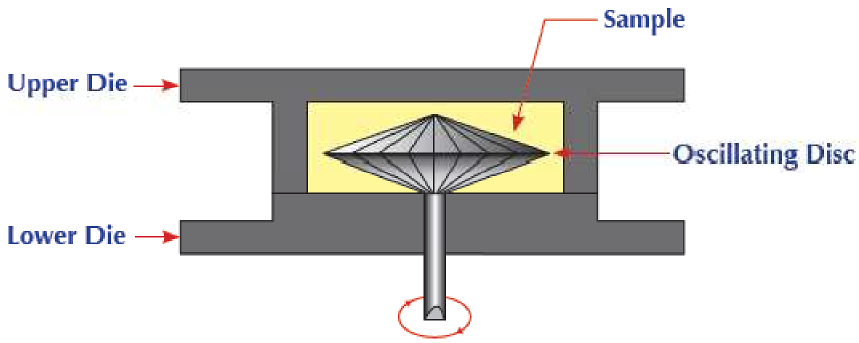

Fig. 1 Principle of the Mooney viscometer [9]

The stiffness of the specimen compound increases when cross links are formed during cure. The direct proportionately posited between the shear modulus and the cross linking density is based on the statistical theory of rubber elasticity .At a constant temperature of the test, a cross linking isotherm is the function of time of that property which serves to measure the course of the cross linking reaction. In the context of Rheometer, the cross linking isotherm is thus the function of time, of the oscillating shear force F, or of the Rheometer indication proportional to it, occurring at a given temperature as a result of vulcanization. A plot of this torque (force) value against time gives a typical graph called 'Cure Curve or Rheograph'. A trained eye can monitor the initial trough i.e. processing behaviour of the compound, the slope of rise during cure phase i.e. curing characteristics and final shape of the curve. Internationally designated parameters like MI, MH, TS2, TS5, TC50, TC90 etc. are markers which can be used to describe a graph. The Cure Curve obtained with Rheometer is a finger print of compound's vulcanization & processing character

Testing Procedure

A piece of uncured compound rubber is place on the heated rotor, and the heated top die cavity is immediately brought down on to the lower die thus filling the cavity. In Fig. 1, the curve shows an immediate initial rise in torque upon closure of the heated cavity. At the top of this first 'hump', the compound has not had much chance to absorb heat from its surroundings, and since viscosity is temperature dependent it will be somewhat higher in these first few seconds. As the compound absorbs heat from the instrument, it softens. Its temperature then stabilizes, and its viscosity has a constant value prior to the onset of cure. This assumes that it is not masked by a very short scorch time. This is the first important feature on the curve [4]. It is the minimum viscosity of the rubber at the chosen temperature and degree of oscillation; and it has the symbol M. After a certain time, the viscosity (torque) begins to increase, indicating that the curing process (vulcanization or cross-linking) has begun. The time from the closure of the cavity to this moment is the next important property, the scorch time. It has the symbol t,l, which means, the number of minutes to a 1 dN.m rise above M, (used with a 1' arc). If a 3' arc is chosen, then a scorch time with the symbol t,2 is used, which is a 2 dN.m rise above M. The torque continues to increase, until there is no more significant rise. At this point the compound is vulcanized, and this maximum torque value is designated by the symbol M. The last major piece of information to be extracted is the time it takes to complete the cure, known as the cure time. The symbol for this property is t'x. This is defined with some according Figs 2 and 3 precision as 'the time taken for the curve to reach a height expressed as the value of ML .plus a 'percentage' of the difference between M, and ML'. If we think of this 'percentage' as a decimal, then 90% is 0.9 (this number is commonly chosen), and mathematically this height would be expressed as O.S (M-M) +M. This cut off at 90% is often known as the technical cure time of the compound, and has the symbol t'90. Some compounds exhibit what is sometimes known as marching modulus, see Fig. 3, where the rate or speed of vulcanization 'marches' on more and more slowly as the cure proceeds. This is often seen in compounds containing CR. [5-6].

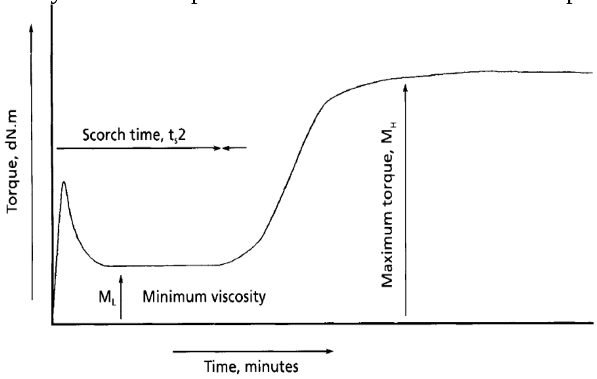

Fig. 2 Typical oscillating disc cure meter curve [4]

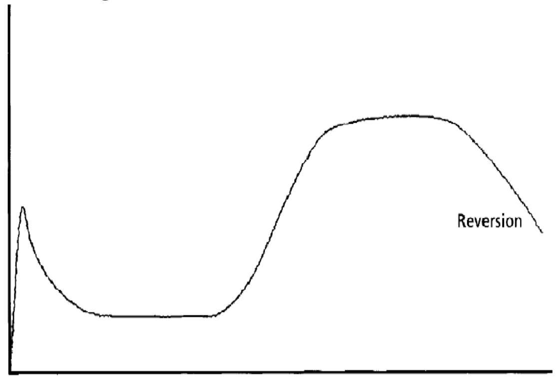

Fig. 3 ODR reversion curve [4]

RESULT AND DISCUSSION

Influence the surface area and structure of particles carbon black on the cure characteristics of natural rubber with grade Standard Malaysian Rubber (SMR20) with different average nitrogen surface area (34 for N660, 76 for N330, 77for N326, and 91for N375) m2/g are shown in Figs 4, 5 and 6. It is obvious from Fig.4 that the scorch time and optimum cure time decreases with increase in Surface area and structure of particles carbon black, but the reversion time of cure curve increase with Increase in surface area and structure. Fig.5 shows that the increase gradually Max. and Min torque with surface area and structure. It is already reported that maximum torque depends on the crosslink density and chain entanglements. This result is attributed to the fact that carbon black affects the crosslink density by reacting with the chemical ingredients of formulation thus leading to a higher torque .Fig.6 shows that increase the Mooney viscosity ML(1+4) at 100C° with increase the surface area and structure ,but decrease the cure rate which express the (100/t90-ts2) with increase the Surface area and structure this results clearly indicates that improvement of cure characteristics When the surface of the carbon particles is oxidized in the manufacturing process, surface pitting occurs which disturbs the relationship between particle size and Nitrogen Surface Area. The interior surface of the pits or micro pores can significantly increase the Nitrogen Surface Area leaving the particle size relatively unchanged. Such oxidation is indicative of a very long reaction time for a given particle size and is always accompanied by slow cure rates and low modulus values: early manufacture of N660, N330, and N326and N375.

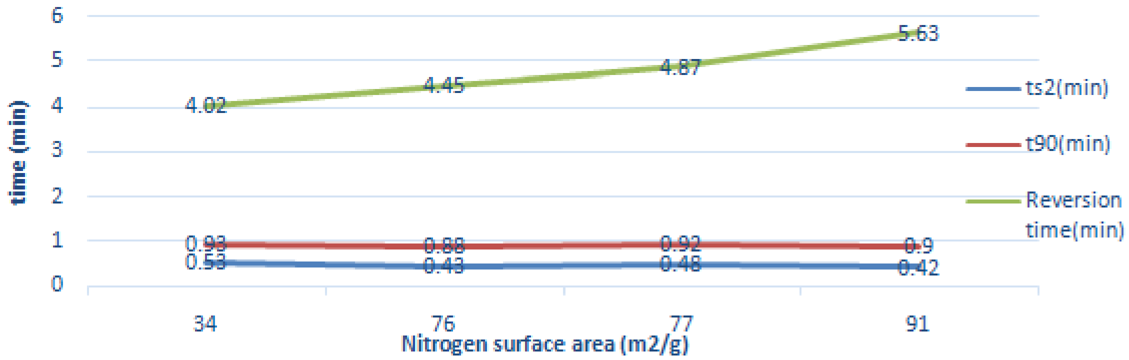

Fig.4 Relation between ts_2, Optimum cure time (t90) and Reversion time (min) with surface area of Particles carbon black (m^2/g)

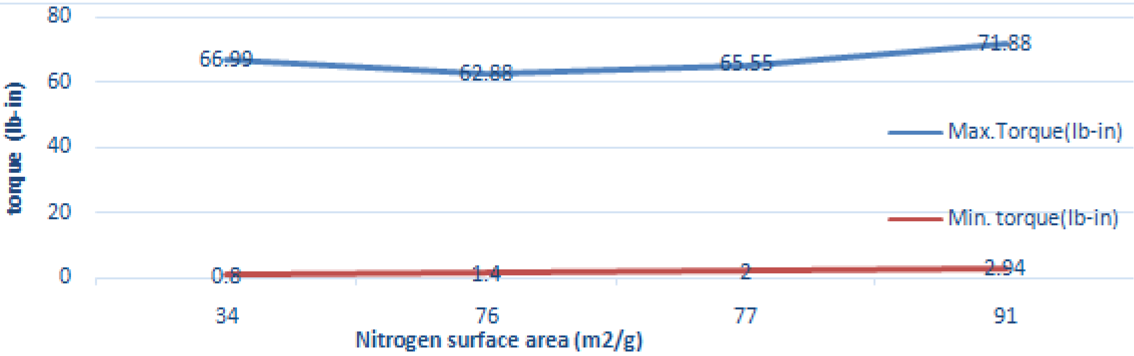

Fig.5 Relation between Maximum and Minimum torque (Ib-in) with surface area of Particles carbon black (m^2/g)

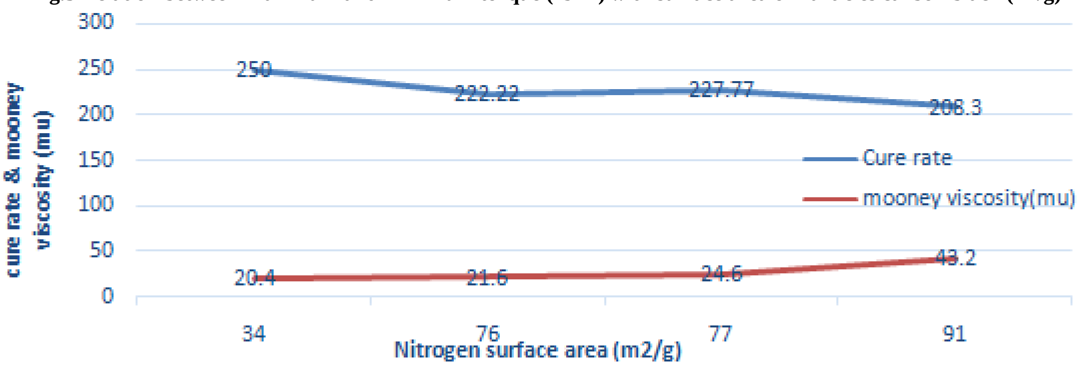

Fig.6 Relation between cure rate and Mooney viscosity (mu) with surface area of Particles carbon black (m^2/g)

CONCLUSION

Surface area and structure are probably the most important morphological characterization of reinforcing elastomers by carbon black because these properties effective in reduction of scorch time and increase in Max and Min. torque of OSD rehometer and increase the reversion period time of cure characteristics and enhance the mechanical properties.

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