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## THE INFLUENCE SURFACE AREA AND STRUCTURE OF PARTICLES CARBON BLACK ON CURE CHARACTERISTICS AND MECHANICAL PROPERTIES OF NATURAL RUBBER

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### ABSTRACT

Surface area is probably the most important morphological characterization for reinforcing the matrix of composite polymer, therefore we used different grades of carbon Black fillers containing different surface area and structure for improvement the cure characteristics and Mechanical properties of composite natural rubber. The grades of carbon black should be add at the same loading levels with different average nitrogen Surface areas  $m^2/g$  and structures for grades ( $34m^2/g$  for N660,  $76 m^2/g$  N330,  $77 m^2/g$  for N326,  $91 m^2/g$  for N375). We note increase the max. torque(Ib-in) for Oscillating disk of Rehometer and Mooney viscosity MI(1+4) at  $100C^\circ$  Also increase in reversion time Because the cure curve it is reversion type with increase the surface area of particles carbon black .

And slowly decrease for the scorch time (min) and increase the cure time with the surface area of particles carbon black, Either about the mechanical properties ,we note increase in Mod300% (Mpa), Compression (C.S %), Tensile strength (Mpa), Hardness shore A and Elongation at brake (%) with increase in surface area. This results are goodness Indications for role surface area of particles carbon black to enhanced cure characteristics and mechanical properties of composite natural rubber.

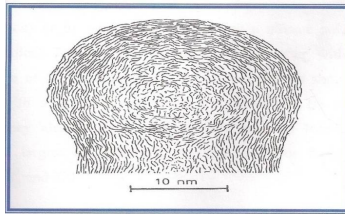
**Keywords:** Carbon Black, Cure Characteristics, Mechanical Properties, Surface Area, SMR<sub>20</sub>.

### 1. INTRODUCTION

**1.1 Carbon black--**This is a material of major significance to the rubber industry, so it is no surprise that most rubber products we see in the market place are black in color. We have moved a long way from collecting carbon from smokey oil flames, which produced a material called

lampblack. The next historical step was to burn natural gas against iron channels, then scrape off the carbon to produce a highly reinforcing material called channel black. Both the use of this black in the rubber industry and its source of supply is currently limited and its cost is some what high.

There are two common methods of producing carbon black today. Heating natural gas in a silica brick furnace to form hydrogen and carbon, produces a moderately reinforcing material called thermal black. Alternatively, if we incompletely burn heavy petroleum fractions, then furnace blacks are produced. These are the most important blacks in terms of quantity used and available types [1]. Carbon black is essentially elemental carbon and is composed of aggregated particles. The particles are partly graphitic in structure and of colloidal dimensions which by parallel alignment and overlapping give the particles their semi- graphitic nature as shown in fig. 1 [2].



**Fig. 1: Concentric layer orientation of carbon black particles [2]**

The particles size ranges (10-400 nm) in diameter, the smaller ones being the less graphitic ,the commonly used carbon black Composed rubber is made by the furnace which provides carbon black with particles size (10-90 nm) such as SAF, HAF, and SRF...etc [3] . The most important characteristics of carbon black are structure and aggregates sizes, particles sizes, surface activity, porosity [4]. The great majority of carbon black post-treatment studies have been conducted to increase strength/quality of reinforcement. So the chemical modifications that have been tested are strongly linked to the different theories envisaged for reinforcement. In the 1960s, carbon black–elastomer interaction was considered as the result of a *chemical bonding* between acidic surface functions and natural rubber alkaline moieties. So many studies have been conducted to increase carbon black activity by surface oxidation oxygen at high temperatures, H<sub>2</sub>O<sub>2</sub>, ozone, nitric acid. The type of oxidation used determines the number and the type of functions obtained; it is interesting to underline that such chemical modifications are used at industrial scale for specialty carbon blacks (inks, pigments). In the early 1980s, Danneberg proposed the mechanism of molecular slippage and post treatments turned to chemical grafting of polymeric chains onto carbon black surface.

### 1.1.1 Surface area and structure of carbon black

Surface area is probably the most important morphological characterization of reinforcing fillers because it corresponds to the extension of the interface, i.e., the interaction between elastomer and filler surface. As evidenced by transmission electron microscopy (TEM), surface area is directly linked to the size of primary particles so that American Society of testing Materials (ASTM) has chosen this parameter for carbon black nomenclature. Iodine number is good indication for measure surface area (particle size), the higher the iodine number the smaller the particle size [5]. Carbon black consists of extremely small particles (from around 10 to 300 nm) in a grape like aggregate. This gives two primary properties allowing a whole range of grades designated by both a particular particle size (surface area) and a specific level of structure. The rubber compounder thus has a whole range of properties available to him. More precisely, ASTM D1765 nomenclature includes four digits, the first one relates to vulcanization speed (N as normal or S as slow), then tree numbers, which first correspond to the primary particle diameter.

ASTM D 24 assigns the second and third digits to new products as the various manufacturers develop them. When a competitor matches a new product it normally carries the same "N" number as that provided the originator. In general, lower structure blacks have been assigned lower numbers; higher structure blacks, higher numbers. However, there are exceptions and structure values are not always proportional to the assigned second and third digits.

For example, one such code name is an oil furnace black type called N110, which is a grade with a very small particle size (therefore highly reinforcing) and fairly high structure. An example of a thermal black type is N990. It has a large particle size, and low structure resulting in a much lower level of reinforcement but higher resilience. Structure relates to irregularity in shape of filler aggregates (Fig-2), determined by the extent and manner of clustering of primary particles. A filler aggregate with high structure has a large void volume within the space it pervades, in which rubber molecules may become "occluded." The combination of occluded rubber and filler then becomes the reinforcing entity, so that the effective volume fraction of filler is increased (Aggregates are not broken down into primary particles upon mixing with rubber.). Generally, reinforcement is enhanced by high structure and strong bonding between filler and Pervaded By the aggregate. The larger the ratio of pervaded to actual volume is, the higher the "structure". Dibutyle phthalate (DBP) is good indication for measure the structure or size of carbon black aggregate. The higher the (DBP) number, the higher the structure.



**Fig. 2: Schematic of a carbon black aggregate consisting of fused primary particles. The dotted line (a surface in 3D) depicts the volume [6]**

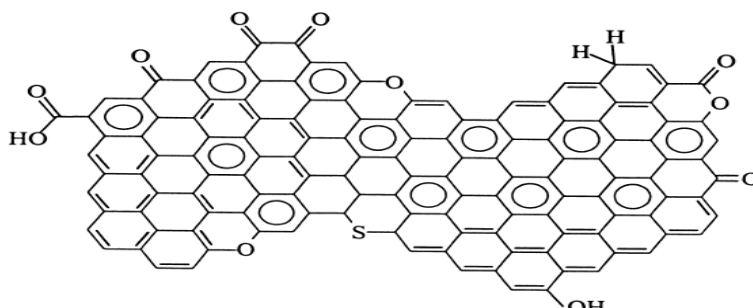
For two carbon blacks with the same particle size, an increase in structure, as in changing from an N326 to an N341, can mean smoother and more dimensionally stable extrusions and calendared compounds. A decrease in carbon black particle size (increase in overall surface area to volume ratio) increases the Tensile strength of the cured vulcanizate [5, 6]. Particle size diameter can only be done by TEM characterization and is difficult and costly, so surface area of fillers is usually obtained by different Adsorption methods. (From Janzen and Kraus and Janzen [1])

## **1.1.2 Surface chemistry of carbon black**

### **1.1.2.1 Oxygenated Functions**

Oxygenated functions on carbon black surface were observed in the early 1950s and completely characterized by H. P. Boehm in the 1960s. At this time, interaction between carbon black and natural rubber was considered the consequence of chemical reactions between the carbon black surface's acidic groups and basic moieties present in the natural rubber structure. The carbon black surface function characterization consists of suspending a given amount of carbon black in solutions of known normality of basis of different strength:  $\text{NaHCO}_3$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{NaOH}$  in water, and  $\text{EtONa}$  in ethanol then carbon black is filtered and the number of reacted acidic groups obtained by titrating the remaining basis in filtrate (Fig. 3). In the 1960s, carbon blacks were mainly prepared by channel processes, and their acidic functions were present at about  $10^{-3}$  eq/g, which allows relatively easy determinations; but now, with furnace blacks, the surface acidic functions are generally of About  $10^{-5}$  eq/g, and specific techniques or drastic reaction conditions must be used.

Obviously, such delicate determination must be conducted on previously extracted black, in order to eliminate basic mineral impurities that would hinder any characterization of the rare acidic groups present on carbon black. This observation allows one to believe that acidic groups on carbon blacks are mainly produced by surface oxidation in the production process, probably during drying following pelletization. Therefore, acidic groups could be considered an alteration of carbon black surface. This point of view is supported by the fact that oxidized blacks which have an acidic surface group content of about  $10^{-2}$  eq/g exhibit a very low reinforcement ability, even if their slow vulcanization is corrected



**Fig. -3: Chemical functions on carbon black surface [7]**

### 1.1.3 Double Carbon Bonds, Hydrogen Content

All chemical studies done in the past on carbon black have focused on chemical impurities or on functions produced by partial surface oxidation of carbon black, and not on its own surface reactivity. Now carbon blacks can not be considered as chemically inert surfaces. Their reaction with iodine or oxygen, their structure evidenced by (Scanning tunnelling microscopy) STM demonstrates the presence of a great number of reactive double bonds on their surface. Following Medalia and Kraus, such double bonds could react with sulfur, olefins, and radicals to provide chemical bonding between carbon black surface and polymer, but their direct quantitative determination has never been obtained. Several authors have observed a strong correlation between hydrogen content of carbon blacks and their reinforcement ability. The content and reactivity of hydrogen present on graphitic edges have been determined by isotopic exchange and correlated to carbon black reinforcement ability.

This characterization is particularly difficult and usually hydrogen mass content is used; these values also are surprisingly well correlated to reinforcement ability of carbon blacks. [5]

### 1.1.4 Dispersibility of carbon black

Even if the relationship between filler dispersion and abrasion resistance is well established, relatively few studies have been done on the characterization of filler dispersibility. This is mainly due to the fact that carbon black dispersibility was commonly judged satisfactory, partly because it is indeed high, but more probably because all mixing apparatuses were designed for dispersing carbon blacks. The use of silica has given a new light to this domain, because, contrary to carbon black, dispersibility is one of the key properties for achieving silica reinforced mixes. Obviously, filler dispersibility is mainly influenced by interactions between agglomerates and/or aggregates, in other words, the force/energy needed in order to separate two objects. For carbon black, these interactions are mainly due to van der Waals forces, which are very low compared to the hydrogen bonding existing between silica objects. In addition, pelletization process has a great influence on dispersibility any action leading to a higher compaction of the filler increases interaction between filler objects and so decreases its dispersibility. In addition to filler's surface energy, which is of major importance, dispersion can also be influenced by filler's morphological properties. Dispersion

is highly influenced by filler surface area: the higher surface area, the lower the dispersion. This result is probably due to the fact that high surface area usually has smaller aggregates, which will develop more interactions with their neighbors in the dry state. Filler structure also has a neat influence on dispersion: the higher the structure, the higher the dispersion. This result is well established and likened to the fact that more “open” aggregate structures develop a lower number of contact with their neighbors in the dry state. [5]

## Previous Works

- ❖ In 1993 the Gale Group in a Thomson corporation Company were studied “the influence of carbon black morphology and surface activity on vulcanizate properties”. We found the carbon black properties such as particle size (surface area), structure, surface reactivity all have a major influence on specific reinforcement [8].
- ❖ In 2002 the M. T. Ramesan was studied “The Effects of Filler Content on Cure and Mechanical Properties of Dichlorocarbene Modified Styrene Butadiene Rubber/Carbon Black Composites” he was found reduction in optimum cure time and increase in rheometric torque with an increase in concentration of fillers. Mechanical properties such as tensile strength, tear strength, compression set increase with increase in loading of filler [9].
- ❖ In 2010 the Franck Sosson, Lénaïk Belec\*, Jean-François Chailan, Pascal Carriere, Alain Crespy, were Studied “Highlight of a compensation effect between filler morphology and loading on dynamic properties of filled rubbers” we found This investigation highlighted the equivalence between carbon black (CB) loading and structure influences on dynamic mechanical properties in the linear behavior of several filled synthetic rubber compounds. Different morphologies (specific surface area and structure) of CB incorporated at different loadings were formulated to modulate the filler-rubber matrix interphase content, usually named “tightly bound rubber” [10].
- ❖ Aim of Search : study the effective of surface area of particles carbon black for improvement the cure characteristics and Mechanical properties of composite natural rubber.

## 2. EXPERIMENTAL SECTION

**2.1 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<sup>2</sup>/g, ID No(82), DBP(72), pure density (446-470)kg/m<sup>3</sup>, particle size (26-30nm), high abrasive furnace. carbon black N330 with pure density (370-393)kg/m<sup>3</sup>, surface area 76 m<sup>2</sup>/g, ID No(82),DBP(102), particle size (26-30nm) high abrasive furnace. Carbon black N375 with surface area 91 m<sup>2</sup>/g, ID No(90), DBP(114), pure density (345-350) kg/m<sup>3</sup>, particle size (26-30nm) high abrasive furnace. Carbon black N660 with surface area 34 m<sup>2</sup>/g, ID No(36), DBP(90) pure density (432-458)kg/m<sup>3</sup>, particle size (49-60nm) general purpose furnace. Zno with surface area 6 m<sup>2</sup>/g, purity of zinc oxide 99% Min, SP.gravity 5.5. Stearic acide with SP.gravity 0.85, Ash at 550C° 0.1% Max., volatile matter at 65C° 0.5% Max. Insoluble sulfer with SP.gravity 1.57, sulfer content 80±2%, Ash at 550C° 0.2% Max, volatile matter at 80C° 0.5% Max.

**2.2 Rubber mixing:** SMR<sub>20</sub>-masterbatch were prepared in a two roll mill at ~ 50°C according ASTM D15 a Homogeneous mixture and minimise the influence of mixing conditions. The vulcanisation systems, including the various grades 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 SMR<sub>20</sub> compounds with various grades 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**

| Item | Materials Compound                | Compound of C.B N660 with NSA(34m <sup>2</sup> /g) phr | Compound of C.B N330 with NSA(76m <sup>2</sup> /g) phr | Compound of C.B N326 with NSA(77m <sup>2</sup> /g) phr | Compound of C.B N375 with NSA(91m <sup>2</sup> /g) phr |
|------|-----------------------------------|--|--|--|--|
| 1    | Standard Malaysian Rubber (SMR20) | 100  | 100  | 100  | 100  |
| 2    | Carbon Black filler               | 45   | 45   | 45   | 45   |
| 3    | ZnO                               | 3  | 3  | 3  | 3  |
| 4    | Stearic acid                      | 1  | 1  | 1  | 1  |
| 5    | Process oil                       | 5  | 5  | 5  | 5  |
| 6    | MBT                               | 0.5  | 0.5  | 0.5  | 0.5  |
| 7    | TMTD                              | 1  | 1  | 1  | 1  |
| 8    | 6PPD                              | 1  | 1  | 1  | 1  |
| 9    | Sulfer                            | 2  | 2  | 2  | 2  |
| 10   | Total                             | 158.5  | 158.5  | 158.5  | 158.5  |

Cure conditions, temp. =140 C° & time =40 min for sheet 2mm and time =20 min for sample of Hard .,C.S%

**2.3 Curie 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 D2705 a type of moving die rheometer. The optimal vulcanisation time (t<sub>90</sub>) and scorch time (t<sub>02</sub>) of the compounds were determined. The compounds were cured in laboratories of Materials Engineering –Dep. Of polymers and composite Materials Eng. pressing cure at 140°C and 100 bar, according to the t<sub>90</sub> of the specific compounds (see in table -1 ).

**2.4 Mechanical properties.** Tensile tests (tensile strength Mpa, Elong.at breack %, Mod.300% Mpa) were carried out on dumb-bell shaped specimens according to ISO 6892 by using Universal testing Machine-Chain. Compression set (CS%) tests were performed at 23°C for 72 hours according to ASTM D395. Hardness of the samples was measured with Hardness-meter Shore A tester TH200 (Beijing time high technology Ltd) according to ASTM D2240-02. SP.gravity by using (GP-1205) according ASTM D 1817.

### 3. RESULTS AND DISCUSSION

#### 3.1 Cure Characteristics

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, 77 for N326, 91 for N375) m<sup>2</sup>/g are shown in Figures.

1, 2 and 3. It is obvious from figure- 1 that the scorch time and optimum cure time are slowly decrease 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. Figure-2 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 effected on the crosslink density by reacting with the chemical ingredients of formulation thus leading to a higher torque .Figure -3 shows that increase the mooney viscosity ML(1+4)@100C° with increase the surface area and structure, but decrease the cure rate which express the  $(100/t_{90}-t_{s2})$  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 micropores 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, N326 and N375

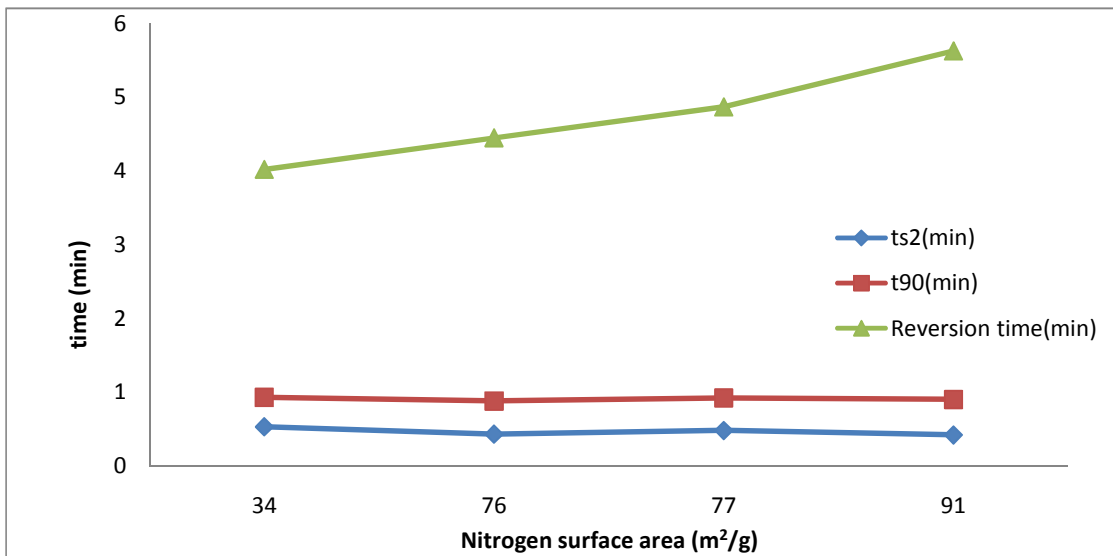


Fig.1: the relation between  $t_{s2}$ , Optimum cure time ( $t_{90}$ ) and Reversion time (min) with surface area of Particles carbon black (m²/g)

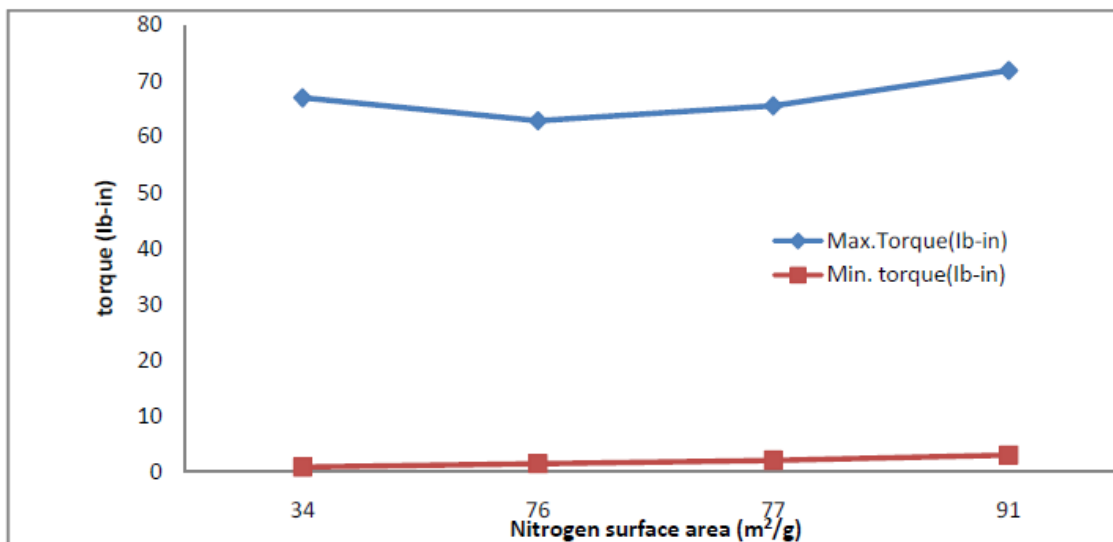
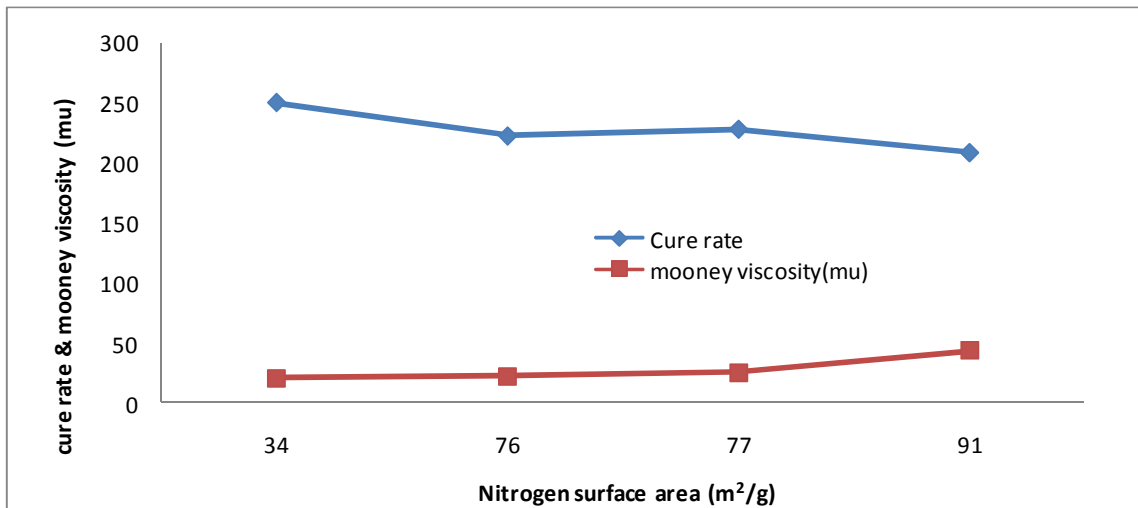


Fig.2: the Relation between Max. and Min torque (lb-in) with surface area of Particles carbon black (m²/g)



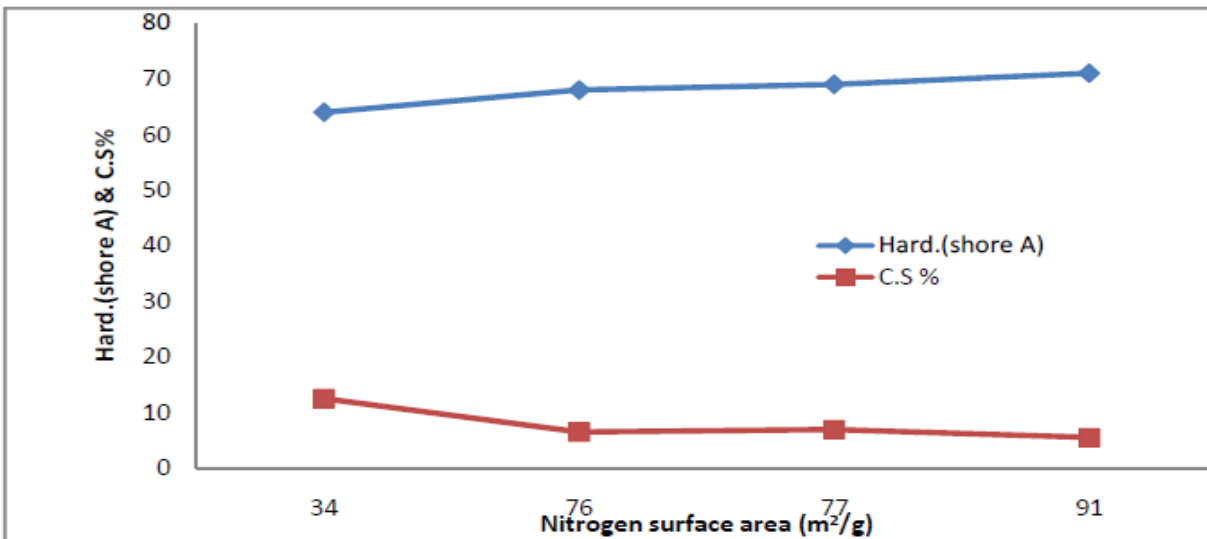


**Fig.3: the Relation between cure rate and mooney viscosity (mu) with surface area of Particles carbon black (m<sup>2</sup>/g)**

### 3.2 Mechanical Properties

Surface area is probably the most important morphological characterization of reinforcing fillers because it corresponds to the extension of the interface, i.e., the interaction between elastomer and filler surface [5], therefore the mechanical properties are improvement with addition different Average nitrogen surface area and structure, that's clearly from figures 4,5,6.

In spite of N326, N330, N375 are the narrow range of particles size (26-30) nm, but differentiation in Iodine number is good indication for measure surface area (particle size), the higher the iodine number the smaller the particle size and differentiation in Dibutyle phthalate (DBP) is good indication for measure the structure or size of carbon black aggregate. The higher the (DBP) number, the higher the structure [5]. All results in cure characteristics and mechanicals properties that included in table -3.



**Fig. 4: The Relation between Hard.(shore A) Compression Set % with surface area of Particles carbon black (m<sup>2</sup>/g)**

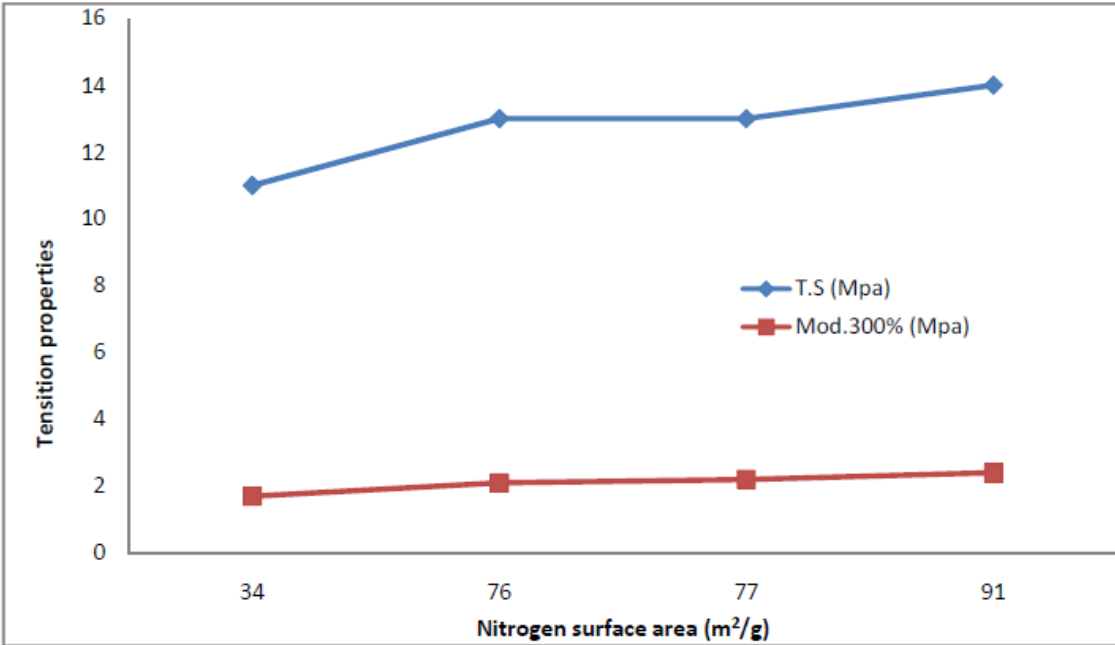


Fig .5: the Relation between tension properties (T.s & Mod.300% (Mpa)) with surface area of Particles carbon black (m<sup>2</sup>/g)

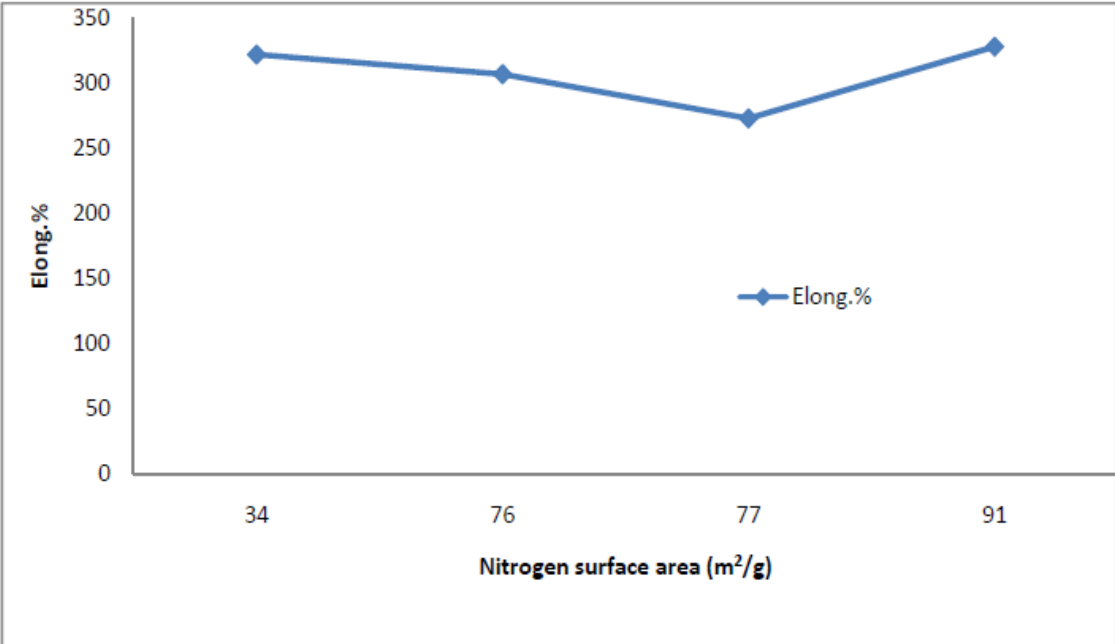


Fig .6: the Relation between tension properties (Elong.%) with surface area of Particles carbon black (m<sup>2</sup>/g)

**Table -3: The results of cure characteristics and mechanical properties**

| Item                 | Mechanical Properties                   | Carbon black particles with different Nitrogen surface area(NSA) |                               |                              |                               |
|----------------------|---|--|-------------------------------|------------------------------|-------------------------------|
|                      |   | 34 m <sup>2</sup> /g for N660                                    | 76 m <sup>2</sup> /g for N330 | 77m <sup>2</sup> /g for N326 | 91 m <sup>2</sup> /g for N375 |
| 1                    | SP.gravity (-)                          | 1.107  | 1.108                         | 1.114                        | 1.106                         |
| 2                    | Hardness (shore A)                      | 64   | 68                            | 69                           | 71                            |
| 3                    | Compress Set (C.S%)                     | 12.5   | 6.5                           | 6.9                          | 5.5                           |
| 4                    | Tensile Strength (Mpa)                  | 11   | 13                            | 13                           | 14                            |
| 5                    | Elongation at break (%)                 | 322.4  | 307.3                         | 273.3                        | 328.4                         |
| 6                    | Modulus of elasticity (Mpa)             | 1.7  | 2.1                           | 2.2                          | 2.4                           |
| Cure Characteristics |   |  |                               |                              |                               |
| 1                    | MooneyViscosity (mu)<br>(ML(1+4)@100C°) | 20.4   | 21.6                          | 24.6                         | 43.2                          |
| 2                    | Max.Torque(Ib-in)                       | 66.99  | 62.88                         | 65.55                        | 71.88                         |
| 3                    | Min. Torque (Ib-in)                     | 0.8  | 1.4                           | 2                            | 2.94                          |
| 4                    | Scorch time (ts2(min))                  | 0.53   | 0.43                          | 0.48                         | 0.42                          |
| 5                    | Optimum Cure<br>time(t90(min))          | 0.93   | 0.88                          | 0.92                         | 0.9                           |
| 6                    | Reversion time(min)                     | 4.02   | 4.45                          | 4.87                         | 5.63                          |
| 7                    | Cure rate                               | 250  | 222.28                        | 227.77                       | 208.3                         |

#### 4. CONCLUSION

Surface area and structure are probably the most important morphological characterization of reinforcing elastomers by carbon black because this 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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