Fjrs.futa.edu.ng

FUTA Journal of Research in Sciences

ISSN: 2315 – 8239 (Print); E-ISSN: 2489 - 0413

FUTA Journal of Research in Sciences, Vol. 12 (2) 2016: 270 - 279

# EVALUATION OF THE CURE CHARACTERISTICS OF COCKLE SEA SHELL (*Anadara granosa*) POWDER AS FILLER IN NATURAL RUBBER COMPOSITES

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**ABSTRACT**

This research work investigated and compared the cure characteristics of cockle sea shell powder and carbon black as fillers in natural rubber compounds. 65µm particle size of cockle sea shell powder, and N330 carbon black were used at filler loading of 10 – 70pphr. Standard Nigeria Rubber (SNR10) was used as the matrix. The cure characteristics of the composites were determined using Oscillating Disc Rheometer (ODR2000). The minimum and maximum torque of cockle sea shell powder increased with filler loading. The increment was not obvious at 10 – 40pphr. Scorch and cures time decrease as the filler loading increases, cure rate of the cockle sea shell composites increases with increasing filler loading. Percentage reversion decreases as the filler loading increases: with highest value of 16% at 10pphr, least value of 9% at 70pphr for cockle sea shell powder filled natural rubber and carbon black filled natural rubber has the highest value of 8% at 10pphr, least value of 4% at 70pphr. All these parameters followed similar trend with that of carbon black composites. When compared with carbon black composites, Cockle Sea shell powder composites has higher minimum and maximum torque, cured faster which in turn makes the percentage reversion faster and higher than the composites of carbon black. Considering cost, availability and applications cockle sea shell filler may serve as a good substitute for carbon black.

**Keywords:** Cockle sea shell, Compounding, Cure, Mixing and Rheograph.

## INTRODUCTION

Fillers are particulate or fibrous materials that are used to reinforce rubber composites for enhanced physical properties (Billmeyer, 1981). In the rubber industry, fillers that are commonly in use are carbon black, china clay and calcium carbonate. Carbon black is derived from petro-chemical sources but the unstable price of crude oil has led to the search for filler that are derived from other sources (Ski, 1970). The purpose of this work was to evaluate the cure characteristics of cockle sea shell powder filled natural rubber composites, in comparison to carbon black filled natural rubber composites. Cockle Sea shells are inorganic in nature they are exoskeletons of molluses such as snails, clams, oysters and many others. Cockle, common name applied to the heart-shaped, jumping or leaping marine mollusks, belonging to the order Eulamellibranchia. The brittle shells are of uniform size, are obliquely spherical, and possess distinct radiating ridges, or ribs (Columbia Encyclopedia, 2016). Cockle can also be said to be one of the most common types of sea shells. It is easily found on almost every beach of the world. It is enjoyed all around the world as a seafood dish. There are two basic types of cockles which can be further classified into around two hundred more classes. They are smooth, rigid and identical in shape (Hassam, 2012). Scanning electron microscope analysis revealed the structure of cockle shell powder to be rod-like aragonite crystals whereas commercial $CaCO_3$ had cube-like calcite crystals (Hoque et al., 2013). Cockle sea shells have three distinct layers and are composed mostly of calcium carbonate with only a small quantity of protein- (≤ 2%) (Scientific American, 2012), apart from carbon black, silica, and calcium carbonate ($CaCO_3$) are conventional fillers used in the rubber industry. The advantages of natural fillers over the traditional fillers for natural rubber composites are low cost, low density, renewability, and environmental friendliness (Ismail *et al*., 2005). Several researchers have carried out work in the use of agro waste as fillers in rubber. Osaremwinda and llori (2002) investigated the use of rice husk in rubber product, Mustapha (2013) investigated the physico-mechanical of natural rubber/ Bone also, and Eric (2004) worked on the mechanical properties of natural rubber/palm kernel shell. A number of studies have being carried out on the cure characteristics of natural rubber compounds (Egwaikhide *et al*., 2007a; Ishiaku *et al.*, 1999a). This research work is aimed at developing filler for natural rubber, which can be an alternative to the commonly used commercial carbon black filler with a consequent reduction in cost. The cockle sea shells are sea wastes all over the world which the powder have been applied here as filler for natural rubber in comparison with standard carbon black filler, with attention being paid to cure characteristics.

## MATERIALS AND METHOD

### Materials

Zinc oxide was supplied by Hangsum plastic additives Co. Ltd. Jingjiang. China. Stearic acid was supplied by Arkimya Sanayi, Turkey, N330 carbon black was supplied by Benya chemical, China. Mmercatobenso-thialzi disulphide, (MBTS) was supplied by Samuh chemical, Dombivli, India and sulphur was supplied by Sulphurnet Rietveldseweg 8 NL-4105LG Culemborg, Netherland, Paraffin oil, Toluene. Kerosine, Petrol and diesel were obtained at Nigerian National Petroleum Corporation. Distilled water was obtained at Federal Institute of Industrial Research, Oshodi, Lagos., Standard Nigeria Rubber (SNR-10), was obtained from the Institute of Rubber Research Benin, Edo state and Cockle sea shell were also obtained from Badagry, Lagos State.

### Preparation of cockle Sea Shell powder

The shells were properly washed with detergent and oven dried at 80$^o$C for 1hr to remove moisture. The shells were crushed to small sizes and then milled, using electric milling machine. The required particle size was obtained using 63µm laboratory test Sieve, BS410-1:2000 standard. The powder collected was kept in a tight container prior to use.

### Compounding

The recipe used in the formulation of the natural rubber composites are given in Tables 1 and 2. Mixing was carried out on two roll mill accordance with ASTM- D3184-80.

**Table 1: Formulation for Natural Rubber Composites with Cockle Sea Shell powder as Filler**

| Content | (pphr) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Natural Rubber | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Zinc Oxide | **5** | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Stearic Acid | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| Cockle Sea Shell powder | 0 | 10 | 20 | 30 | 40 | 50 | 60 | 70 |
| Paraffin Oil | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Sulfur | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| MBTS | 1.8 | 1.8. | 1.8 | 1.8. | 1.8 | 1.8. | 1.8 | 1.8. |

**Table 2: Formulation for Natural Rubber Composites with Carbon Black as Filler**

| Content | (pphr) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Natural Rubber | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Zinc Oxide | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Stearic Acid | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| Carbon Black | 0 | 10 | 20 | 30 | 40 | 50 | 60 | 70 |
| Paraffin Oil | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Sulfur | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| MBTS | 1.8 | 1.8. | 1.8 | 1.8. | 1.8 | 1.8. | 1.8 | 1.8. |

**Cure Characteristics Determination**

The curing characterization was carried out with an Oscillating Disc Rheometer (ODR 2000), at 150$^{o}$C in accordance to the ASTM D 2084-01 standard. 4g of each sample was placed between the two dies (i.e upper and lower dies) of the Oscillating Die Rheometer. The minimum and maximum torques, scorch time $t_2$, cure time $t_{90}$ and cure rate were determined from the rheographs generated by the rheometer.

**Cure Rate Determination**

The cure rate was determined using the expression;

$$\text{Cure rate} = \left[\frac{100}{(t90 - ts2)}\right] \ldots\ldots\ldots\ldots\ldots (1)$$

Where $t_{90}$=cure time at 90%

$ts_2$= time to reach 2 unit increase in torque above minimum (at $M_L$ +2 unit above it)

**Percentage Reversion**

The percentage reversion was calculated using this expression

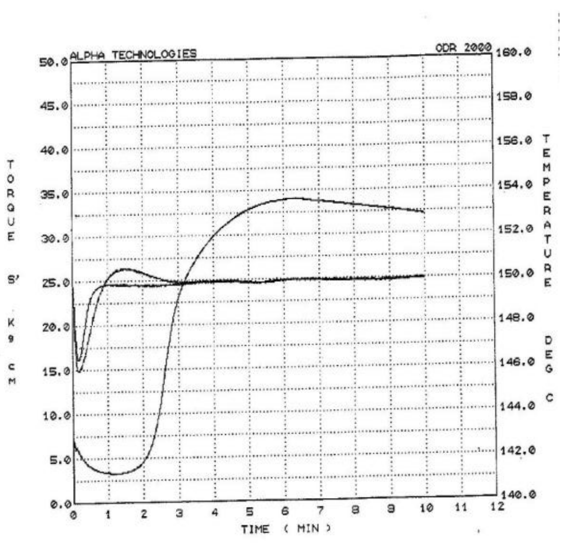
$$\%R = \left\{\frac{(Tmax - Tt)}{(Tmax)}\right\} 100 \ldots\ldots (2)$$

Where $T_{max}$ = maximum torque, $T_t$ = torque at t minutes

## RESULTS

### Cure Characteristics

The minimum and maximum torque, scorch and cure time values were reported directly from the rheograph chart for all the composites as shown in Fig. 1 and Fig. 2. The values for cure rate and percentage reversion were calculated, using the equation 1 and equation 2 respectively. Torque is a measure of forces to the tension or rotation about an axis equal to the product of the force vector and the radius vector from the axis of rotation to the point of application of the force. Figure 3 shows the minimum torque of the composites which increase with increase in filler loadings. It can be seen that, cockle sea shell powder / natural rubber composites has higher minimum torque than carbon black filled natural rubber composites at various filler loadings. Figure 4 shows the minimum torque of the composites which also increase with increase in filler loadings. From 10 to 30pphr, carbon black filled natural rubber composites have higher maximum torque than those of cockle sea shell powder. From 40 to 70pphr cockle sea shell powder filled natural rubber composites have higher maximum torque than the composites of carbon black. Fig. 5 and Fig. 6, show the scorch time and cure time of the composites respectively. The scorch time and cure time decreases as the filler loading increases for both fillers, while the cockle sea shell powder has the lower value for all the loading. Fig. 7 shows that the percentage curing of the vulcanizate per min increases as the filler loading increases. The cure rate of the composites of carbon black increases with increasing filler loading at a steady rate from 10pphr to 70pphr. Composites of sea shell also followed the same trend with significant increment from 10pphr to 70pphr filler loading. It was observed that cockle sea shell composites have distinctly higher cure rate at various filler loading. The cure rate behavior of the cockle sea shell powder might likely be connected to the nature of the filler being filler that is proteinous in nature and has calcium as its major constituent. Figure 8 shows that reversion decreases with increases in filler loading for the fillers i.e. carbon black and cockle sea shell powder. Reversion with distinct higher values of between 16% - 18% for 10 to 30 phr, decreases steadily from 40 - 70pphr for composites with sea shell this may be due to the composition of the filler. Carbon black has a steady reversion of about 8%-7% from 10pphr to 50pphr. There was a drastic reduction at 60 and 70pphr.

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| | | | |
|---|---|---|---|
| ^ Temp: 150.0 °C | ML: | 3.34 kg-cm | |
| v Temp: 150.0°C | MH: | 34.11kg-cm | |
| $t_{S1}$: 1:56 m:s | $t_{S2}$: | 2:07 m:s | |
| $t_{10}$: 2:14m:s | $t_{50}$ | 2:46 m:s | |
| | $t_{90}$: | 4:18 m:s | |

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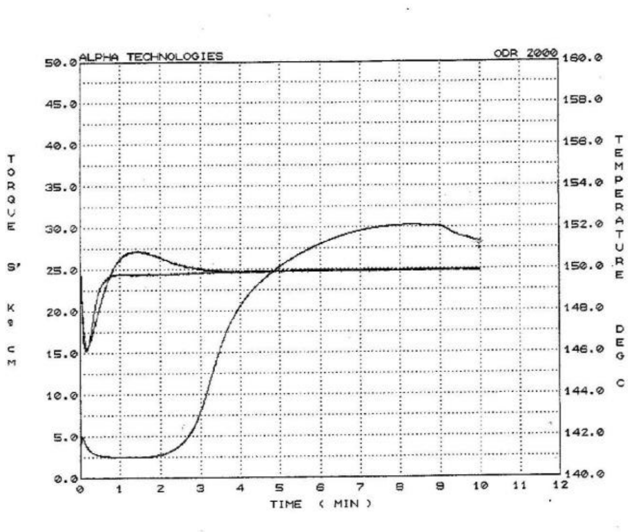
**Figure 1: The Rheograph Chart of Natural Rubber for 40pphr Cockle Sea Shell Powder Filler**

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^ Temp: 150.0 °C ML: 2.51 kg-cm

v Temp: 150.0°C MH: 30.27kg-cm

$t_{S1}$:2:22 m:s $t_{S2:}$ 2:37 m:s

$t_{10}$:2:44m:s $t_{50}$ 2:34 m:s $t_{90}$: 5:46 m:s

----------------------------------------------------------------

**Figure 2: The Rheograph Chart of Natural Rubber for 40pphr Carbon Black Filler**

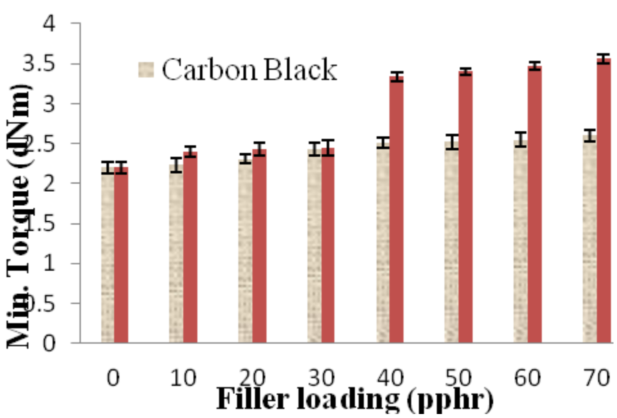


**Figure 3: Minimum torque of cockle sea shell powder and carbon black filled natural rubber composites at various filler loading.**

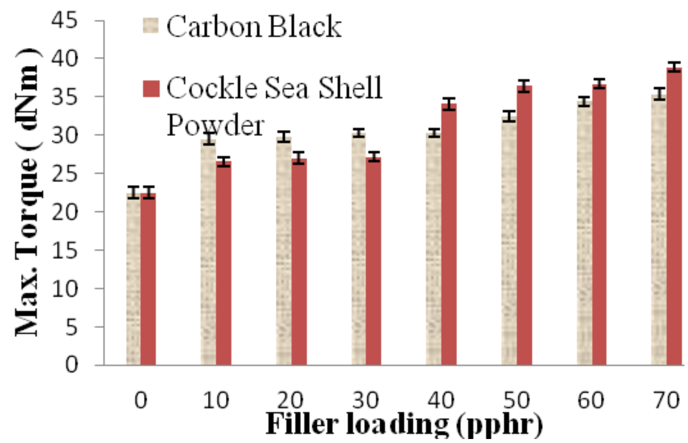


**Figure 4: Maximum torque of cockle sea shell powder and carbon black filled natural rubber composites at various filler loading.**

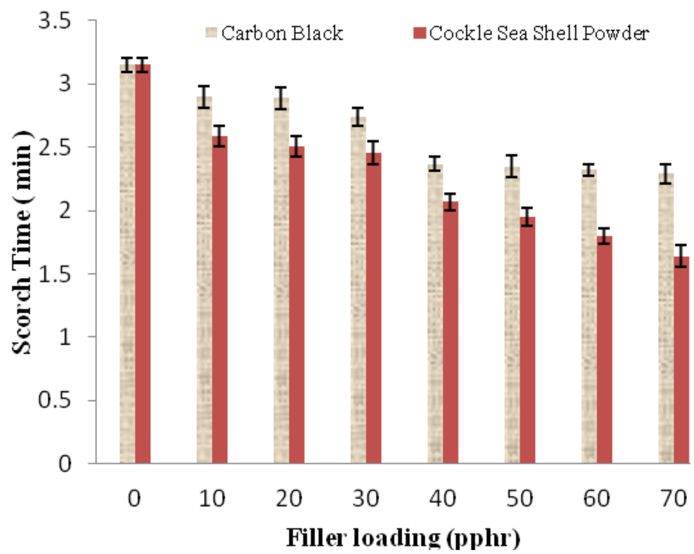


**Figure 5: Scorch time of cockle sea shell powder and carbon black filled natural rubber composites at various filler loading.**

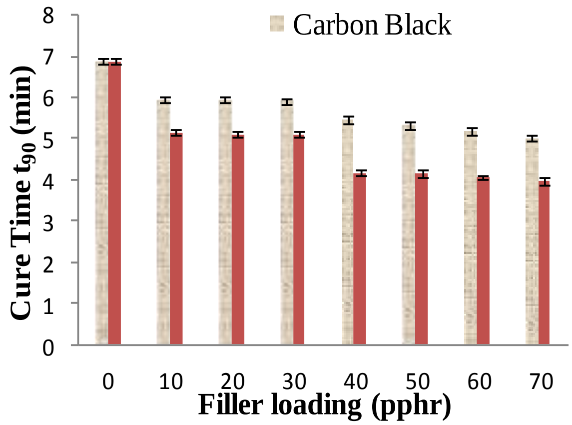


**Figure 6: Cure time of cockle sea shell powder and carbon lack filled natural rubber composites at various filler loading.**

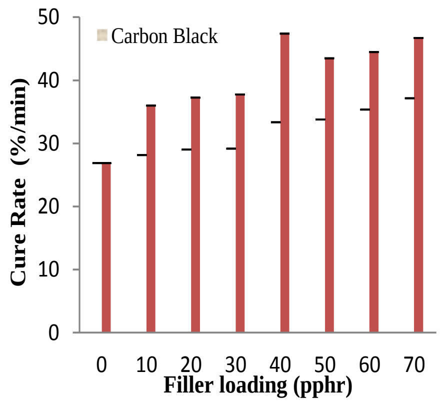


**Figure 7: Cure rates of cockle sea shell powder and carbon black filled natural rubber composites at various filler loading.**

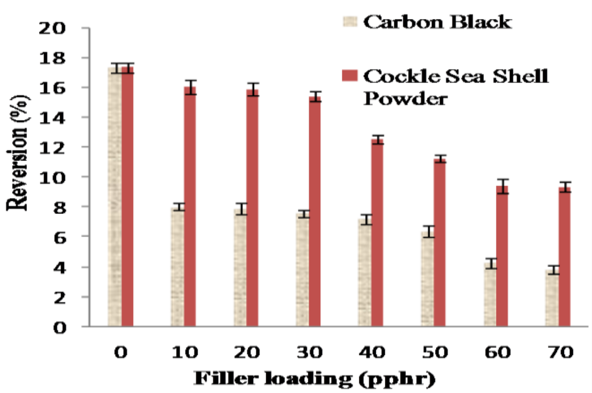


**Figure 8: Percentage reversions of cockle sea shell powder and carbon black filled natural rubber composites at various filler loading.**

**DISCUSSION**

Fig. 3 and Fig. 4, the increasing value of minimum and maximum torque with increase in filler loading is due to increase in viscosity. This is expected to increase with increase in filler loading. The increase in viscosity could also be due to agglomeration of the filler particles in the composites materials. Also, matrix content decreases in ratio with increase in filler content, so an increase in the concentration of the filler particles will reduce flow. Torque maximum increases continuously with increase in the concentration of fillers. This is because the filler particles have relatively high modulus compared to the matrix, so this will lead to an increase in maximum torque. A similar trend was observed by Ishiaku *et al* (2007b) in their investigation into the cure characteristics and vulcanization properties of a natural rubber composites extended with convoluted rubber powder. The differences in cure characteristics may be attributed to the fact that each of the fillers possesses different filler properties (viz, surface area, surface activity, particle size etc.) (Ismail *et al.*, 1998) The higher values of minimum and maximum torque of cockle sea shell composites than the composites of carbon black at various filler loading, showsthat, more axial force was required at various filler loading for cockle sea shell composites than the composites of carbon black. This has further established the excellent interaction among the molecules of carbon black and natural rubber, which allows free flow of energy, except for maximum torque from 10 to 30pphr, which prove otherwise. This might be due to the wettability nature of the materials at that point. Fig. 5 and Fig. 6, show the scorch time and cure time of the composites respectively. Generally, the additions of fillers reduce not only scorch time, but also the optimum curing time (Sae-our *et al*., 2002). The decreasing scorch and cure time with filler loading is a result of the higher milling time of the stock during mixing. Shorter $ts_2$ and $t_{90}$ are caused by premature curing due to shearing force generated during compounding process. As the filler loading increases, the time of incorporation of additives and filler also increased. Excessive heat accumulated from longer compounding time is responsible for the shorter scorch and cure times (Daud *et al.*, 2016). As also reported by, Ismail et al (1999) the scorch time and cure time reduced with increasing filler loading. Mustapha (2013) also show that the cure time of bone ash filled natural rubber vulcanizates decrease with increase in filler content. Also, according to Senapati et al (1989) the decrease in scorch and cure time with filler loading is a result of the milling time of the stock during mixing. As filler concentration increases, the time of incorporation also increases. It was observed that at various filler loading the scorch time and cure time of carbon black /NR composites was higher than those of cockle sea shell / NR. This shows that, at various filler loading more time was required for milling cockle sea shell / NR composites, than composites of carbon black / NR. This suggest that, there is faster and better interaction between natural rubber and carbon black molecules than between cockle sea shell powder and natural rubber molecules. The nature of the fillers may likely be responsible for this, as carbon black has better surface activity, and also known for its excellent interaction with natural rubber molecules. Fig. 4 shows the cure time $t_{90}$ for the materials, it can be seen that the cure time decreases with increase in the filler loading. For all the various filler loading carbon black – natural rubber composites has the highest value of cure time, cockle sea shell powder has a close values for the entire filler loadings. Though cockle sea shell powder is inorganic filler but it still gives a reasonable result may be because it has calcium as one of its major constituent. Fig. 7 shows that the percentage curing of the vulcanizate per min increases as the filler loading increases. The cure enhancement of the vulcanizate can be associated with the filler related parameters such as surface area, surface reactivity, particle size and moisture content. Other factors affect the cure time of rubber composites, such as temperature, curing system and thickness are the most important. It has been reported that cure rate is directly related to the humidity and water content of the composites mix (Butter and Freakly 1991). However, in the present study the most probable factors that account for the observed cure enhancement are chemical composition of the filler, surface area, moisture content and the marked increment in the torque observed for the composites mix, shows that the presence of filler in the rubber matrix has reduced the mobility of the macro molecular chains of the rubber. A similar trend was observed by Egwaikhide *et al* (2007a) in their investigation into the effect of coconut fibre filler on the cure characteristic of physio - mechanical and swelling properties of natural rubber composites. It was generally observed that at various filler loading, cockle sea shell powder composites has higher cure rate than the composites of carbon black. This suggested that carbon black composites can withstand higher temperature than thecomposites filled with cockle sea shell powder. Fig. 8 shows that reversion decreases with increases in filler loading for the fillers i.e. carbon black and cockle sea shell powder. The presence of the filler has great effect on reversion because as the ratio of filler to natural rubber increases indicating the formation of more stable crosslinks, there was a decrease in the percentage reversion. It could be seen that vulcanizate without filler has the highest percentage reversion. Reversion is defined as the loss of crosslink network structures which occurs during non - oxidative, (or anaerobic) thermal ageing (Kenneth Bates, 2013). Reversion processes are severely detrimental to the crosslink degradation that takes place; it reduces stiffness, tear strength and tensile strength. An increase in carbon loading showed a decrease in reversion (Sloan 1997). Similar trend was reported Ismail and Ishiaku (2000) reported in their research on palm oil fatty acid additive (POFA) filled natural rubber compounds. Other mechanical properties showing this inverse dependence were modulus, ultimate strain and hysteresis. The effect of carbon black on reversion was investigated by Sloan (1997) and found that reversion was not a linear function of carbon black loading. The research of Bristow (1991) presented evidence that the grade of natural rubber affects the reversion behavior of the vulcanizate. The spin up wear of aircraft tires has been investigated by Padovan *et al* (1993), upon landing the friction generated by the tire and the runway can raise the temperature of a thin boundary layer of the tire to begin reversion. Since percentage reversion is a function of temperature, time and nature of the filler, it's clearly shown in Figure 6, that carbon black / NR composites have better heat absorbent ability than cockle sea shell powder / NR composites at various filler loading. The higher calcination temperature promotes higher calcination rate as this will increase the particles kinetic energy and thus, accelerates decomposition of $CaCO_3$ to CaO. The SEM analysis conclude that the higher calcination temperature give the structure of the sample more porous. Hence, more $CO_2$ will be released to give the more conversion to CaO (Ahmad and Bin 2013).

## CONCLUSION

It was established that the cure time $t_{90}$ of the composites differs, sea shell has the shortest scorch time and cure time ($t_{90)}$ at various filler loading. Carbon black has the least minimum torque at various filler loading. This implies that cockle sea shell composites get cured faster than carbon black composites at the same temperature. The higher minimum and maximum torque of cockle sea shell composites than those of carbon black indicates that cockle sea shell composites get stiffened faster than carbon black composites during the curing process. This also makes the cure rate to be faster and earlier than composites of carbon black. The fast cure rate of the cockle sea shell composites, in turn makes the percentage reversion faster and higher than the composites of carbon black at various filler loading. The reversion behaviour and the cure characteristics performance of cockle sea shell powder in this study established that cockle sea shell powder were good enough and could serve as alternatives to carbon black

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