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Effect of Grounded Calcium Carbonate on Mechanical Properties of Carbon Black Filled EPDM rubber

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ABSTRACT

Grounded calcium carbonate (GCC) is widely used in the industry as cheapening filler with no or little reinforcing effect. In this research, GCC which is a non-reinforcing filler was added as secondary filler to a typical Ethylene Propylene Diene rubber (EPDM) compound formulation. This research was done to study the extent of the non-reinforcing effect of GCC filler on the mechanical properties of carbon black (CB) filled EPDM compound to be used in automotive application. GCC varied from 0 to 100 phr were incorporated into carbon black filled EPDM vulcanizates. The results show that the hardness increased with the increased in GCC loading. The maximum hardness obtained was 85 IRHD. Modulus at 100% and 300% strain and tensile strength decreased upon the addition of GCC. This was due to non-reinforcing nature of GCC. This may be expected as GCC has poor rubber-filler interaction. The elongation at break increased with the increase in GCC loading which attributed to the diluent effect imparted by GCC. However, the value started to decrease as 60 phr GCC was added. The reason could be due volume occupied by the fillers exceed the volume made available in the rubber phase. From these findings, the utilization of GCC in EPDM rubber can be applied up to 100 phr GCC and even more loading can be tested according to product requirement specified by certain application while maintaining the range of required mechanical properties.

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INTRODUCTION

Fillers have been widely used as cheapening agent to reduce rubber content while maintaining or improving the mechanical properties of the compound (El-Wakil & El-Megeed, 2011; Ichazo, Albano, Hernández, González, & Carta, 2008; Kraus, 1971; Muniandy, Ismail, & Othman, 2012). Generally, fillers are group into reinforcing and non-reinforcing fillers. Carbon black (CB) is a conventional reinforcing filler used to enhance the mechanical properties including strength and stiffness of rubber(Hamza, 1998; Rattanasom, Prasertsri, & Ruangritnumchai, 2009).The medium particle size and higher structure types, such as FEF N550 GPF N650, favour reinforcement and increase both hardness and stiffness (Ferrante, 1990).

Non-reinforcing fillers cause reduction in strength properties and usually applied as diluents or extenders to generally reduce cost (Osabohien & Egboh, 2007).Limestone, chalk, etc.are a very widely occurring natural calcium carbonate (Titow, 1984) from which the fillersare obtained by fine milling (grounded calcium carbonate, GCC) or synthesised by precipitation (precipitated calcium carbonate, PCC)(Kulshreshtha & Vasile, 2002). PCC fillers is more expensive than GCC (Kulshreshtha & Vasile, 2002). GCC is widely used as cheapening filler in the industry since it is quite inexpensive and it is usually obtained by grinding natural white calcite (CaCO)(Jeong, Yang, Chae, & Kim, 2009).

Ethylene–Propylene–Diene-rubber (EPDM) is currently the largest non-tire elastomer, and the market is still growing(Dijkhuis, Noordermeer, & Dierkes, 2009). The main uses of EPDM are in automotive applications as profiles, hoses, and seals; as it imparts good resistance to aging, weathering and chemicals(Nabil, Ismail, & Azura, 2013). It is one of the expensive materials consumed in highest volume in the production of these rubber-based products. High content of raw EPDM contributed to high production cost; thereby forcing rubber products manufacturers to modify existing formulations to meet customer requirements for 'within specification'

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products at competitive price. The most common way of reducing the cost of production is to reduce the overall amount of EPDM consumption by introducing cheapening fillers (diluents) into the compound.

In this research, six formulations of EPDM rubber were compounded. The various GCC loading were added as secondary filler in addition to the 100 phr of CB. Up to 100 phr GCC was added to study the effect of GCC loading on mechanical properties of EPDM vulcanizate. The mechanical properties studied were tensile properties and hardness.

Experimentation:

Materials:

Ethylene Propylene Diene rubber (EPDM) with 4.5 wt% of ethylidenenorbornene (ENB) was supplied by Keltan DSM elastomer (Netherlands), Carbon black (CB) FEF N550 is a fast extruding furnace black with average particle size 0.05 µm is manufactured by Cabot Corporation (United States) and ground calcium carbonate (GCC) with average particle size 5 µm is supplied by ExcelkosSdn. Bhd. (Malaysia). Accelerator and other curatives were obtained from RhenogranRheinChemie Corporation (United States).

Compounding of EPDM Vulcanizates:

Formulations of the compounding ingredients expressed as parts per hundred parts of rubber (phr) for six different GCC loadings are shown in Table 1. Fixed amount (in phr) of each curative were added in the 6 formulations. Allcompounds were prepared by mixing on a conventional laboratory-sized two roll mill machine (Eenor, Open Mixing Mill) according to ASTM D 3182.

Table 1: Formulations of main components of EPDM rubber compound with various GCC loadings.

Ingredient	Quantity, phr
EPDM	100
Paraffin Oil	40
CB	100
GCC	0, 20, 40, 60, 80, 100
Zinc oxide	10.0
Stearic acid	1.5
Antioxidant	1.0
CBS	0.3
MBTS	0.3
ZDBC	0.2
TMTD	0.2
Sulphur	0.7

Mechanical Testing:

First, the compounds were cured and vulcanized using HI-TOP Hydraulic Hot Press machine operated at a pressure of 1000 psi and 160 °C press temperature to prepare sample prior to tensile, tear and hardness test. The hardness of the various EPDM vulcanizates was measured by an automated dead load hardness tester following ISO 48 with one reading only. Dumb-bell shaped test pieces for tensile test were cut using dumbbell type II cutter. The tensile test was conducted using Instron Tensile Tester with gauge length of 20 mm and crosshead speed of 500 mm/min. Five test pieces for each compound were tested and the median value was recorded and reported here. Test pieces for tear test were cut using type C (angle cut) cutter. The test were conducted with the same speed, 500mm/min.

RESULT AND DISCUSSION

Tensile strength & elongation at break:

Tensile strength values of rubber compounds quantify the stressof material will endure before failing (Chandrasekaran, 2007). The tensile strength and elongation at break of various GCC loading of EPDM vulcanizates are shown in Figure 1. The error bars shown are the standard error of the means containing three test pieces each.

Based on Figure1, the first compound with no GCC filler (blank sample) showed a tensile strength of 14.0MPa. Up to 40phr GCC added, the tensile strength has reduced to 13.2MPa with around 6% strength reduction. The reduction become more visible with further loading of GCC as 100phr GCC resulted in 10.1MPa of tensile strength with 27% reduction. The reduction of tensile strength for all compound were expected since 100phr CB was used. As referred to a previous research by Hamza (1998), optimum value for tensile strength obtained was around 17.0MPa at 40phr CB though different CB was used(Hamza, 1998). Therefore, theoretically as 100 phr CB used, the expected result should be below 17.0MPa. Azemi (2013) also reported that beyond the optimum loading, the tensile strength decreased due to the volume occupied by the filler exceeds the volume made available in the rubber phase (Samsuri, 2013). The reduction was also attributed to the non-reinforcing nature of GCC which having larger particle which weaken the interaction bonding between the fillers and rubber matrix (Ismail, Zulkepli, Wei, & Nasir, 2013; Poh, Ismail, & Tan, 2002).

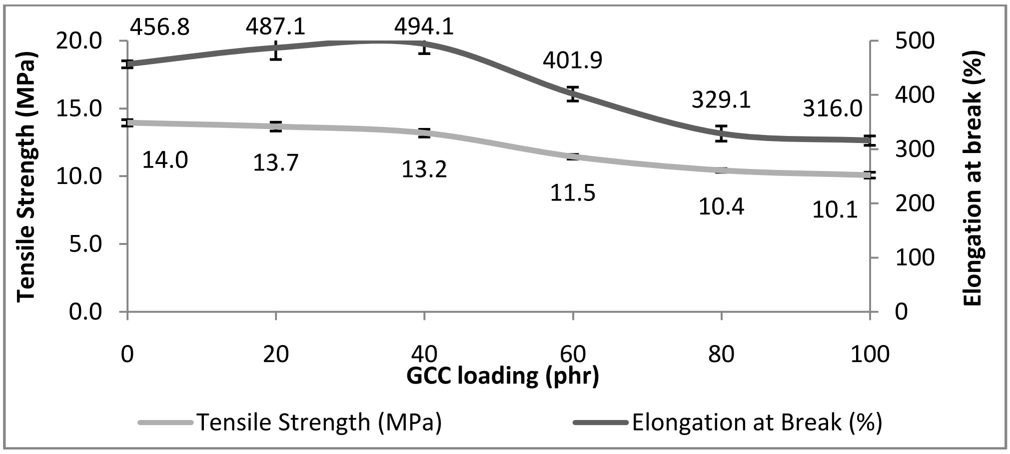

Fig. 1: Tensile properties of EPDM vulcanizate with various GCC loading

From the result obtained, elongation at break (Figure 1) of the vulcanizates show different trend.The elongation at break of blank sample was 456.8%. Upon the 20phr and 40phr GCC loading, the percentage of elongation was increased to 487.1% and 494.1% respectively.The increment of elongation at break up to 40 phr GCC was around 8% compared from blank sample. This could be due to the increasing of dilution effect of the GCC which made the vulcanizate to extend much more before rupture occurs (Poh *et al*., 2002). The dilution effect increased with increasing in GCC loading up to 40 phr. When 60 phr and further loading of GCC were compounded, the elongation at break tendsto reduce to 316.0% elongation. This could be related to the volume occupied by the fillers exceed the volume made available in the rubber phase(Samsuri, 2013) thus led to the reductioninthe percentage of elongation. That meant the dilution effect of the GCC could not provide anymore extra extension since thevolume of the total fillers were overloaded. Nevertheless, the elongation at break of all compounds were still above 300% elongation.

The changes in tensile strength and elongation at break can be divided into two phases. For 0 to 40 phr GCC loading, the reduction in tensile strength was in a range 13.0-14.0 MPa and the increment in elongation at break was in the range 450-500%. For 60 to 100 phr GCC loading, the reduction in tensile strength was in the range 10.0-11.5 MPa and elongation at break was reduced to the range of 300-400 %.

Hardness & Tensile Modulus:

Hardness is a measure of cured rubber's resistance to deform when a force is applied on the rubber surface at specific time (Dick, 2001). Figure 2 below shows hardness and modulus at 100% strain of various loading of CB filled EPDM vulcanizates.Blank sample showed a hardness value of 76 IRHD due to the presence of high loading of CB filler (100phr)(Samsuri, 2013). As expected, blank sample give the lowest hardness value compared to other compound since it has the lowest total filler loading. Addition of GCC fillers increased the hardness further. The expected trend attributed to the fact that as more filler loadings are incorporated into the rubber matrix, the compounds become more rigid, due to the reduction of rubber chain elasticity (Egwaikhide, 2007; O. Aguele & I. Madufor, 2012).

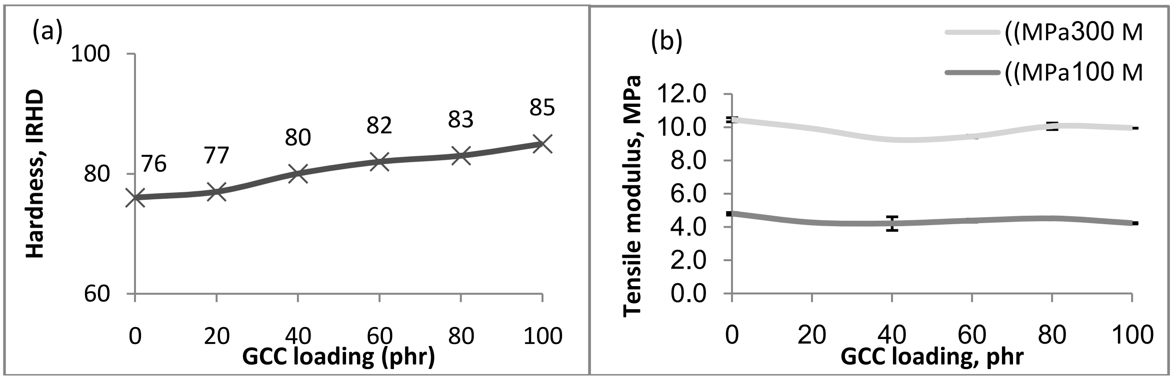

Fig. 2: (a) hardness, IRHD and (b)modulus at 100% strain, MPa of various GCC loading of CB filled EPDM vulcanizates.

Tensile modulus is affected by several factors such as surface reactivity which determines the polymer–filler interaction, aggregates, size and shape of particles, structure and filler particle dispersion in rubber(Poh *et al*., 2002). Based on figure 2(b), tensile modulus at 100% strain (M100)and 300% strain (M300) showed a different trend. Blank sample which contained CB filler only, showed the highest M100 value, 4.8 MPa. As reported by Poh *et al*. (2002), the interaction of CB and rubber is stronger than its attraction between aggregates; meanwhile aggregate interaction is stronger in calcium carbonate(Poh *et al*., 2002). Therefore, the M100 value for all GCC loaded compounds were lower compared from blank sample. Based on previous study on CB fillers, tensile modulus increased along with hardness as filler loading increased (Poh *et al*., 2002; Rattanasom *et al*., 2009).But, It is shown that M100 value was independent of GCC loading, indicating that GCC is a non-reinforcing filler (Poh *et al*., 2002).

Conclusion:

From these findings, the use of GCC in EPDM rubber can be utilized up to 40 phr GCCwithout affecting much of its mechanical properties. Further loading of GCC can be added as long as it passed the minimum requirement of product specification for automotive application.

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