NOVATEUR PUBLICATIONS
INTERNATIONAL JOURNAL OF INNOVATIONS IN ENGINEERING RESEARCH AND TECHNOLOGY
[IJIERT] ISSN: 2394-3696 Website: ijiert.org
VOLUME 9, ISSUE 6, June -2022

ASSESSMENT OF THE MECHANICAL PROPERTIES OF NR/SBR BLEND REINFORCED WITH EGG SHELL AND CARBON BLACK

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ABSTRACT
The assessment of the mechanical properties of NR/SBR blend reinforced with egg shell (ES) and carbon black (CB) were investigated. The egg shell was ground with automated machine into powder form, sieved and used in compounding the NR/SBR blend by varying the loadings at 0-50 pphr. The mechanical properties investigated such as tensile strength, tensile modulus, elongation at break, hardness, abrasion resistance, compression set and fatigue resistance, showed increase in tensile strength (ES: 13.4215-25.1126MPa) and (CB: 13.4215-31.0524 MPa), tensile modulus (ES = 4.2439-11.8009 MPa) and (CB; 4.2439-16.0031 MPa), abrasion resistance (ES: 20.1140-36.6532 mm^3/rev.) and (CB: 20.1140-40.0068 mm^3/rev.) for filler loadings 0-50 pphr then the decreased at filler loading 50 pphr. The hardness showed an increase for both filler loadings (ES: 28.1603-65.6007 Shore A) and (CB: 28.1603-72.5310 Shore A) while noticeable decrease in properties such as elongation at break (ES: 721.7220-535.2889%) and (CB: 721.7220-621.1225%), compression set (ES: 56.2310-30.0705%) and (CB = 56.2310-40.2119%), fatigue resistance (ES: 11.2880-5.0300 Kc x 10^3) and (CB: 11.2880-7.1882 Kc x 10^3) as the filler loadings increased. The NR/SBR blend composites showed an improved abrasion resistance at filler loadings 0-40 pphr before decreasing at 50 pphr (ES = 30.5211 mm^3/rev and CB = 37.8006 mm^3/rev.). The CB-filled NR/SBR blend composites exhibited excellent mechanical properties and enhanced performance when compared with ES-filled NR/SBR blend composites.

Keywords: Blend, Composites, Carbon black, Egg shell, Hardness

INTRODUCTION
Blending of chemically different polymers is an important tool in industrial production for tailoring products with optimized material properties. Performance of polymer blends depends on the properties of polymeric components, as well as how they are arranged in space. One of the most basic questions in blends is whether or not the two polymers are miscible or exist as a single phase, most blends of high molecular weight polymers exist as two-phase materials. The morphology of the phases is of great importance in this manner. A variety of morphologies exist such as dispersed spheres of one polymer in another, lamellar structures, and continuous phases (Kukaleva et al, 2000). A new approach to the science

and technology of polymer blends has emerged recently. These polymeric materials must perform under strenuous mechanical, chemical, thermal and electrical conditions imposed by the requirements of a specific application (Altan et al, 2011; Caliskan et al, 2011; Ronkay, 2011).

The blending of two or more polymers by physical or chemical means may improve a variety of physical and chemical properties of the constituent polymers (Jovanovic et al., 2013; Chanin et al, 2014). The polymer blending was a universal technology commonly used in the development of a newer product with superior properties. The blending of rubbers plays an important role in enhancing the physical properties of the final vulcanized product. The rubber blends are frequently used in the rubber industry to obtain the best combination of compound properties, processability and cost. . Natural rubber (NR) is a renewable and sustainable material, which has various good properties, such as high tensile strength, high elongation, and outstanding resilience, lending itself to be used in various applications (Chanin et al, 2014). NR can exhibit higher tensile and tear strength than synthetic rubbers due to its ability to undergo strain crystallization (Boonmahitthisud and Chuayjuljit, 2012; Vu and Choi, 2015; Chuayjuljit et al, 2015).

Styrene-butadiene rubber (SBR), nonpolar synthetic rubber, describes families of synthetic rubbers derived from styrene and butadiene. It has good abrasion resistance and thermal aging properties (Goyanes et al., 2008). Styrene-butadiene rubber has been used in NR blends since it has better abrasion resistance, crack initiation resistance, high filler-loading capacity and lower heat-build up behavior (Findik et al, 2004; Tangudom et al, 2014). SBR is generally used in wear applications. SBR is usually blended with NR to improve its tensile and tear properties (Findik et al, 2004; Tangudom et al, 2004). Thus, SBR has been used in a wide range of products such as side walls of tires, belts, hoses, foot wears, foamed products, etc (Findik et al, 2004; Tangudom et al, 2004). It was previously shown that tensile strength, elastic modulus, elongation at break, hardness and wear resistance of NR/SBR composites increase with increasing NR content and prevulcanization time (Varkey et al, 2000; Findik et al, 2004).

Fillers represent one of the most important additives used in rubber compounding. Fillers are added to rubber formulation in order to optimize properties needed for service application. Due to strong environmental regulations worldwide and increased interest in the proper utilization of renewable natural resources, efforts have been made to find alternative reinforcements that are environmentally friendly while providing the same performance as their synthetic counterparts (Egwaikhide et al, 2007). With their low cost, easy availability, ease of chemical and mechanical modification, and high specific mechanical properties, natural fibres represent a good, renewable and biodegradable alternative to the most common synthetic reinforcement (Lovely et al, 2006). Locally available materials such as limestones, eggshells, corn cobs, groundnut shells, rubber seed shells, rice husk, cocoa pod husk and Cherry (Chrysophyllum albidum) are amongst the underutilized renewable resources in our society today which can be used directly or converted by simple processes to valuable materials in polymer or related applications (Osabohien et al, 2004).

OBJECTIVE

To study the mechanical properties of NR/SBR blend reinforced with egg shell and carbon black

MATERIALS AND METHODS

Materials

The natural rubber (NSR-10) and styrene butadiene rubber (SBR) used for the research work were obtained from Rubber Research Institute of Nigeria (RRIN), Iyanomoh Benin-City. Egg shell was obtained from Njoku Poultry Farm, Nsukka, Enugu State, Nigeria. Carbon black and rubber compounding chemicals such as processing oil, TMTD, TMQ, zinc oxide, sulphur and stearic acid were obtaining from Rovet Chemicals.

Characteristics of Fillers

The egg shell was ground with automated grinding machine then sieved with a mesh of size 75µm mesh, which was the particle size of fillers (egg shell and carbon black) used for the experiment.

Preparation of blend composites

The rubber was masticated and mixed with an additives using the two roll mill and adopting the standard method specified in the ASTM-D 3184-80 for all the blend composite samples. The filler loadings were varied at (0-50 pphr). The Table 1 shows the formulations for the NR/SBR blend composites. The rubber mixes were prepared on a laboratory size two roll mill. It was maintained at 70^0C to avoid cross-linking during mixing after which the rubber composite was stretched out. Mixing follows (ASTMD 3184–80, 1983).

Table 1: Formulations for Reinforced NR/SBR Blend Composites.

Ingredients	Part per Hundred of Rubber (pphr)
NR	70
SBR	30
Zinc oxide	3.5
Stearic acid	2.5
TMQ	1.5
TMTD	1.5
Fillers (Egg Shell and Carbon Black)	0-50
Sulphur	4.5
Processing oil (wax)	5.0

Mechanical Properties of the Vulcanizates

Tensile Test

The test specimens were cut from the moulded dump-bell rubber sheets along the grain direction. The thickness and width of each test piece at the middle was maintained at 2.5 and 6mm respectively. Each test piece was clamped into the grips of the tensometer. The stress applied, the load and elongation at break was recorded. The test samples were tested in the machine giving straight tensile pull, without any bending or twisting. The machine measures both the tensile stress and the tensile strain. The tensile stress is the strength of pull in the area between the notch marks; it is based on original cross sectional area. The tensile strain is a measure of how the test sample has been stretched by the pull.

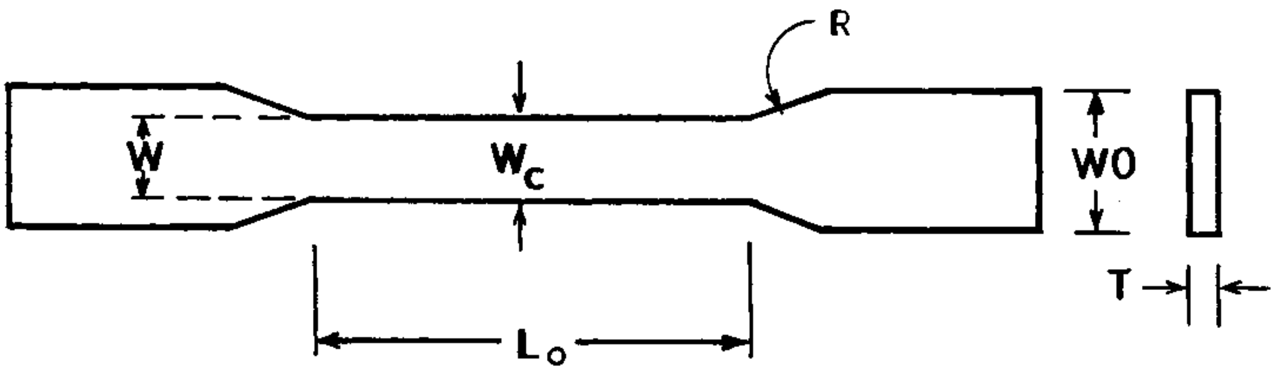

Figure 1: Sample Dumbbell for Tensile Testing

Hardness Test

Hardness test carried out by Wallace Dead Load Hardness Tester according to ASTM D-1415 using disk samples (3 mm thick and 45 mm diameter) and for 40 second penetration time. The average value of readings was adopted by the International Rubber Hardness Degree (IRHD) units.

Compression Set Test

The compression set is the difference between the original thickness of the sample and the thickness after the test expressed as a percentage of the original thickness. Compression set evaluate the extent by which the specimen fails to return to its original thickness when subjected to standard compression load for a given period of time at a given temperature. Stress of 2.8MP was used and allowed for 24 hours at 70ºC for 30mins.

$$\text{Compression Set } (\%) = \frac{t_o - t_r}{t_o} \times 10 \qquad 1$$

Where: t_o = Initial Thickness and t_r = Recovered thickness of Sample.

Abrasion Resistance Test

Wallace Akron abrasion tester was used. The angle between the test sample and the wheel was adjusted to an angle of 15º. The abrasion was carried out for 100 revolutions and the material loss for each run was noted. The specimen was re-reweighed between each test run. The mean of the four revolutions of the abrasive wheel was calculated.

$$\text{Abrasion Resistance} = \frac{\text{Weight Loss of the Standard}}{\text{Weight Loss of the Sample}} \times 100 \qquad 2$$

Flex Fatigue Test

The measurement was carried out in accordance to the procedure described in ASTMD-430 using the Flex machine, which function by inducing surface cracking of the rubber vulcanizates sample.

RESULTS AND DISCUSSIONS

Mechanical Properties

Table 2: Mechanical Properties of Egg Shell and Carbon Black filled NR/SBR Blend Composites

Properties	Filler Loadings (pphr)					
	0	10	20	30	40	50
Tensile Strength (MPa)	13.4215	(19.5503) [26.3211]	(23.3524) [27.9524]	(24.8520) [30.3450]	(25.1126) [31.0524]	(23.0650) [28.6840]
Tensile Modulus (MPa)	4.2439	(8.8120) [10.9035]	(10.4251) [12.1005]	(11.6207) [15.0661]	(11.8009) [16.0031]	(10.0325) [15.1663]
Elongation at Break (%)	721.7220	(521.5602) [702.2070]	(609.9027) [695.7030]	(601.1109) [693.4057]	(589.1102) [678.1387]	(535.2889) [621.1225]
Hardness (Shore A)	28.1603	(52.0344) [54.0824]	(56.1130) [59.3092]	(60.8521) [62.1558]	(62.0057) [70.8006]	(65.6007) [72.5310]
Abrasion Resistance (mm^3/rev.)	20.1140	(25.7007) [27.0224]	(30.1178) [32.4112]	(35.1108) [38.1235]	(36.6532) [40.0068]	(30.5211) [37.8006]
Compression Set (%)	56.2310	(46.9003) [56.3125]	(43.0009) [53.2160]	(41.2352) [52.6012]	(36.2545) [43.9421]	(30.0705) [40.2119]
Fatigue Resistance (Kc x 10^3)	11.2880	(9.0006) [11.0300]	(8.0880) [10.8867]	(7.9025) [10.1153]	(6.9315) [8.6235]	(5.0300) [7.1882]

Key: pphr = part per hundred of rubber ( ) = Egg Shell (ES) [ ] = Carbon Black (CB)

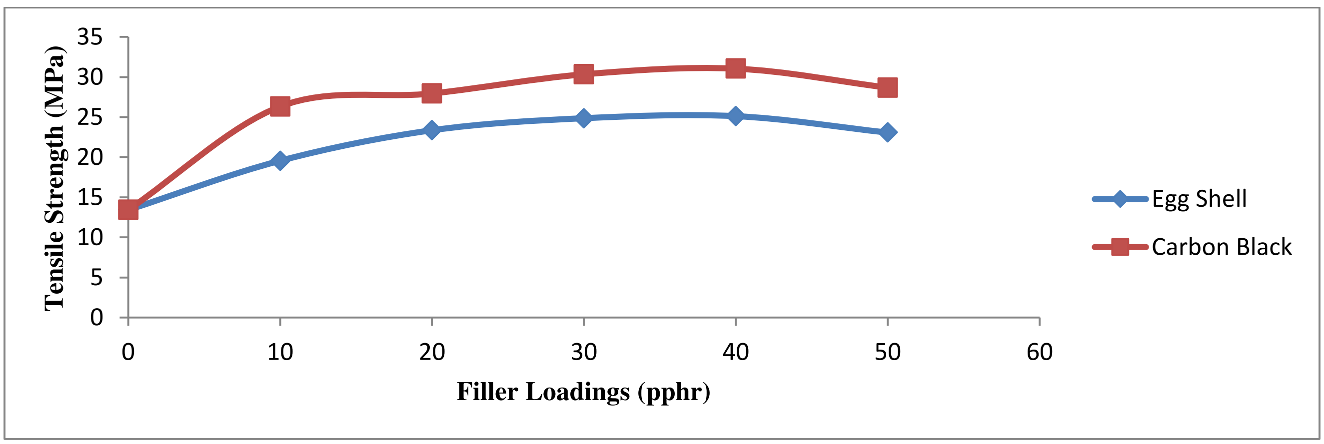

Figure 2: Egg Shell and Carbon Black Loadings on Tensile Strength of NR/SBR Blend Composites

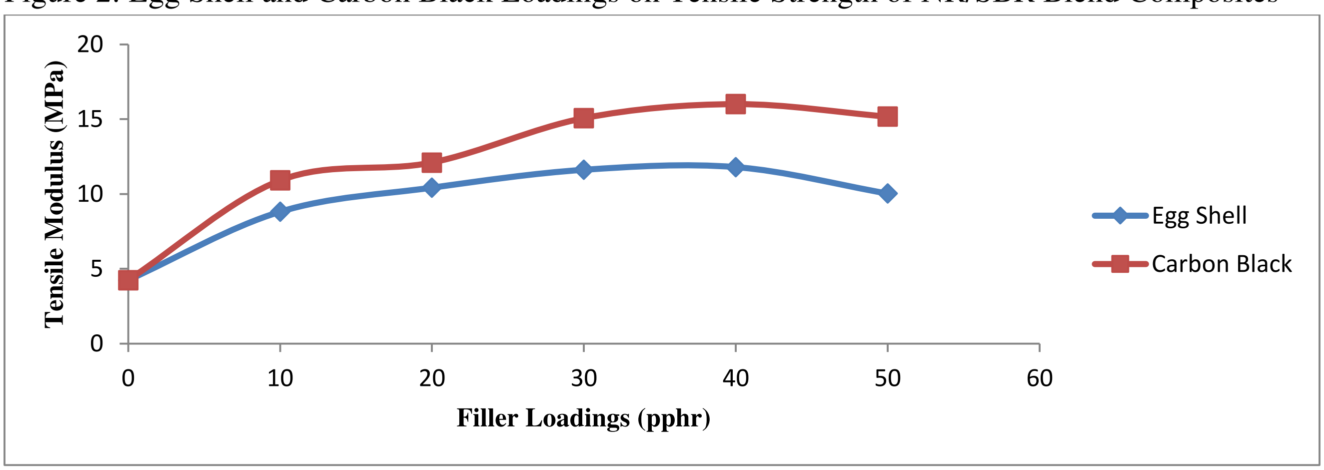

Figure 3: Egg Shell and Carbon Black Loadings on Tensile Modulus of NR/SBR Blend Composites

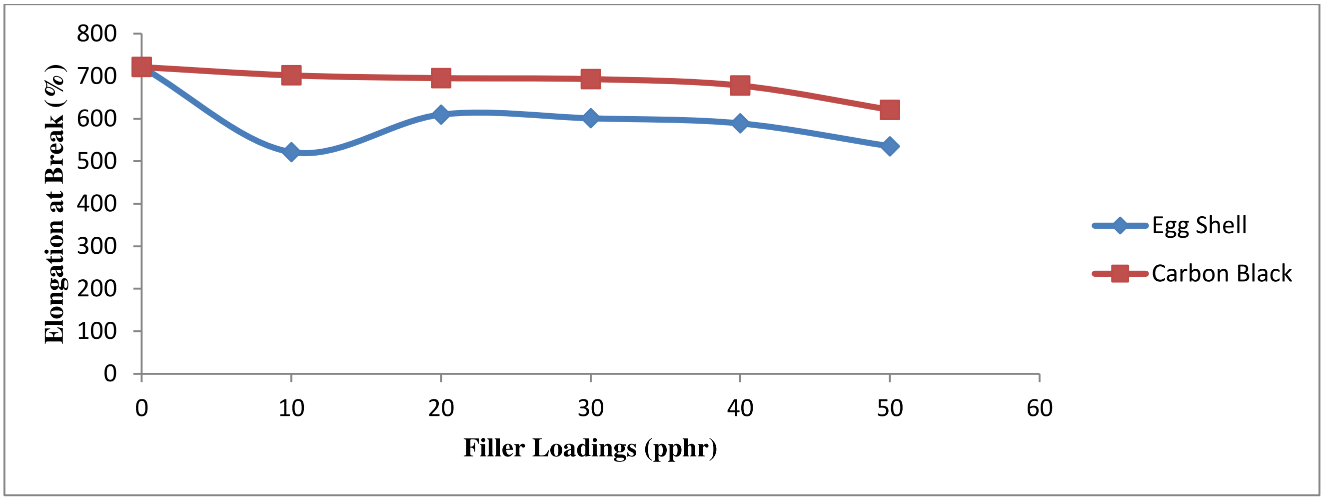

Figure 4: Egg Shell and Carbon Black Loadings on Elongation at Break of NR/SBR Blend Composites

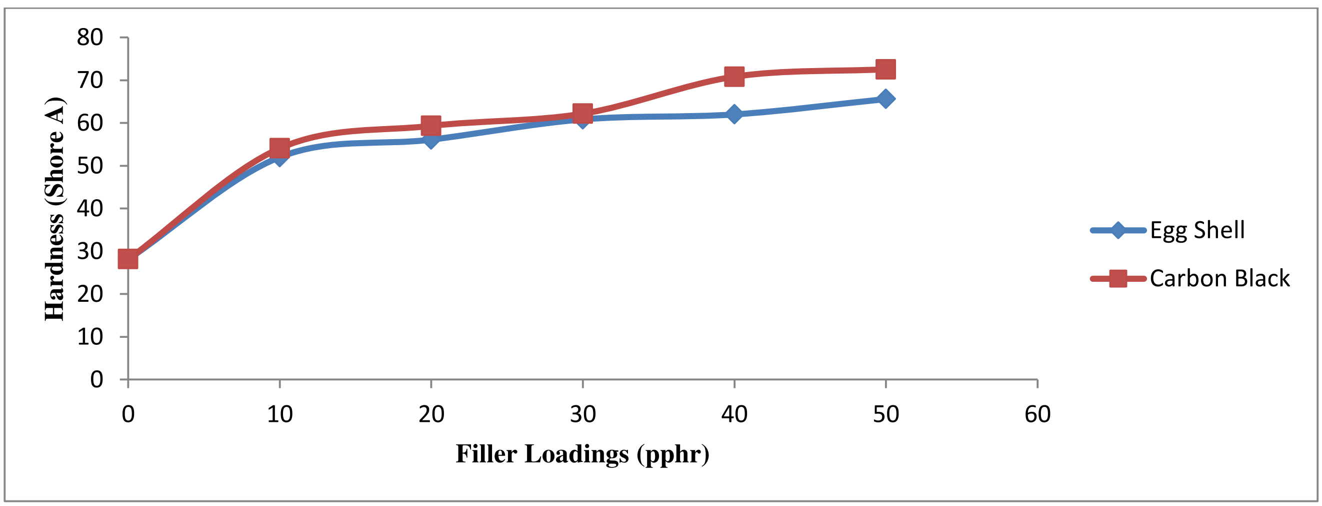

Figure 5: Egg Shell and Carbon Black Loadings on Hardness of NR/SBR Blend Composites

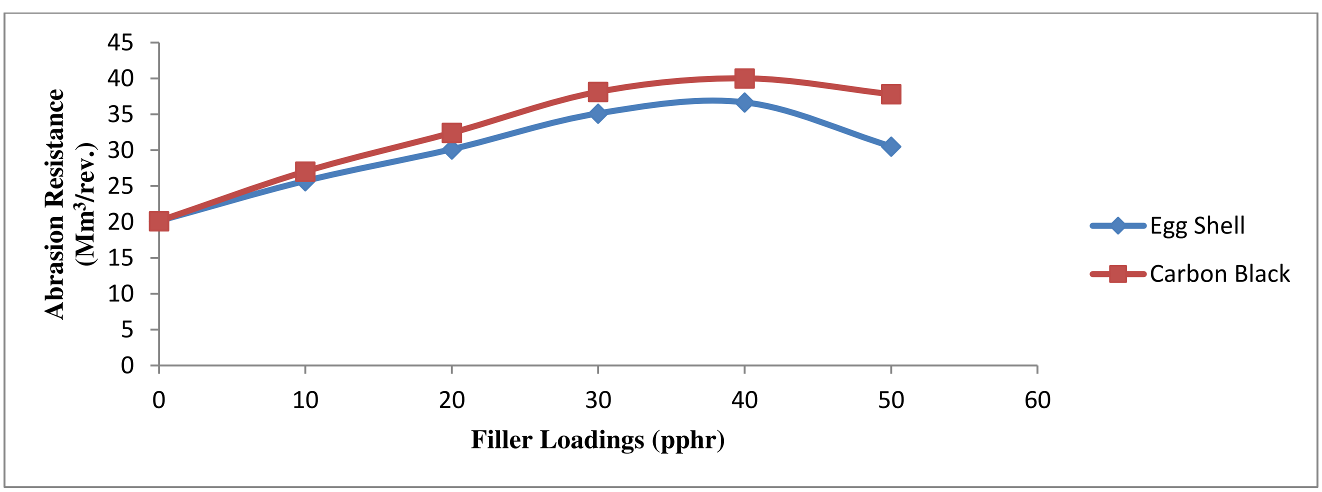

Figure 6: Egg Shell and Carbon Black Loadings on Abrasion Resistance of NR/SBR Blend Composites

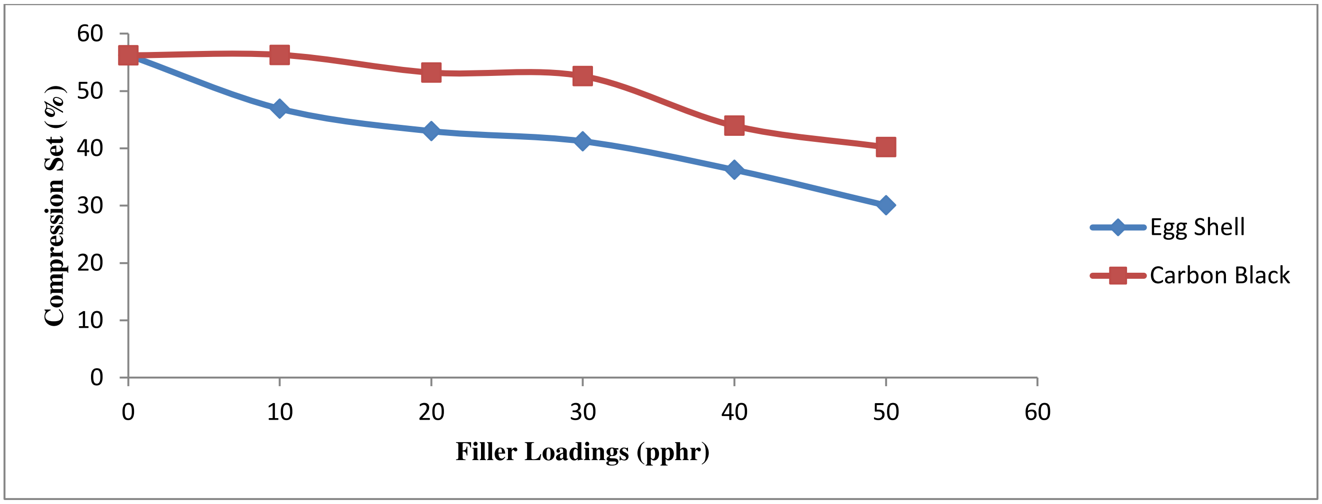

Figure 7: Egg Shell and Carbon Black Loadings on Compression Set of NR/SBR Blend Composites

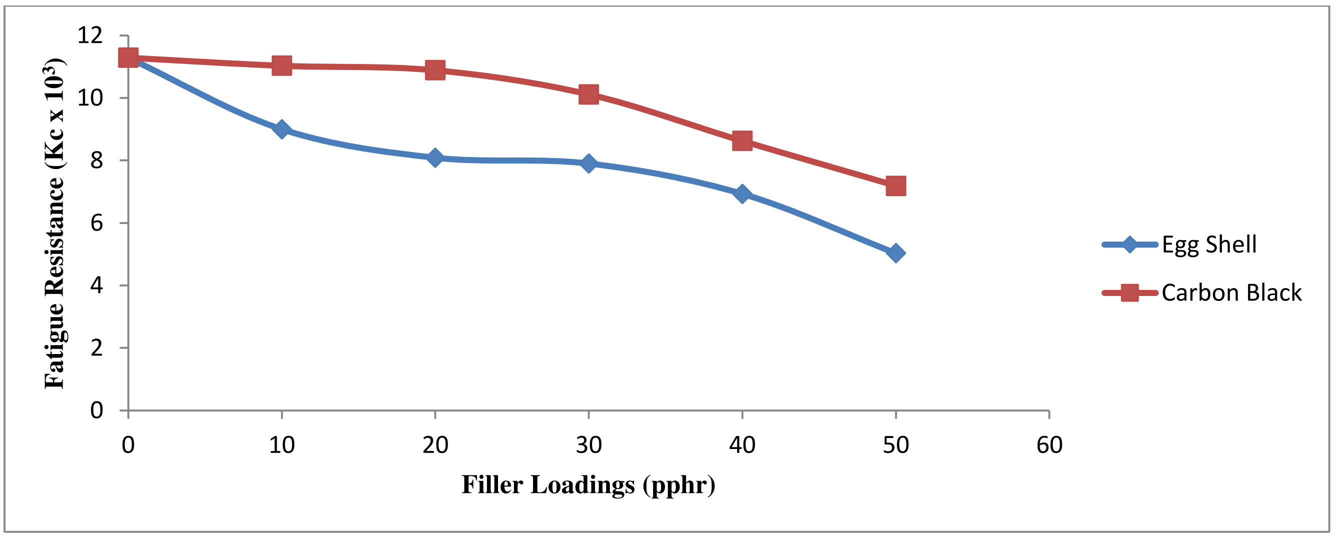

Figure 8: Egg Shell and Carbon Black Loadings on Flex Fatigue of NR/SBR Blend Composites

Mechanical Properties

The experimental data related to the mechanical properties of 70/30 pphr NR/SBR blend and its composites filled with (0-50 pphr) of the respective particle fillers are shown in Table 2 and Figure 2-8. Factors affecting the tensile strength are the ratio of filler materials in the compound, particle size, interaction of filler materials with the compound, cross-links and production techniques (Akcakale, 2008; Saban et al, 2019).

It can be observed that in all the composites that the tensile strength improved as ES and CB filler loading increased as shown in Tables 2 and Figure 2. This is as result of the reinforcing capability of ES and CB cause by the binding force generated between the interface of fillers and rubber matrix. This may also be attributed to the nature of surface properties, particle size and surface (Egwaikhide et al, 2007). The tensile strength of the unfilled NR/SBR blend had the lowest tensile strength of 13.4215 MPa, the tensile strength increased to the highest values (25.1126 MPa) and (31.0524 MPa) at 40 pphr for ES and CB loadings respectively. The increased tensile strength was achieved because of the ES and CB fillers were uniformly distributed in the rubber matrix and the stress was highly transferred across the interface of the fillers and NR/SBR matrix. At 50 pphr, ES-filled NR/SBR and CB-filled NR/SBR blends composites, the tensile strength decreased to 23.0650 MPa and 31.0524 MPa indicating at that point, the blend composites cannot take up more of the fillers. Also the decrease in tensile strength in EG-filled NR/SBR blend composite at 50 pphr can be attributed to the self-agglomeration of ES filler at high loading which then lowered the rubber interaction (Anyaporn et al, 2017). The CB-filled NR/SBR blend composites had the better tensile strength than the EG-filled NR/SBR blend composites in all the filler loadings indicating that CB is more reinforcing because of the present of high carbon content. The effectiveness of filler may be measured by its carbon content. Fillers with higher carbon content, provide greater reinforcement than those with lower carbon content because carbon itself is very good reinforcing filler (Amoke et al, 2017).

Table 2 and Figure 3 showed the result of tensile modulus of the ES and CB-filled NR/SBR blend composites. It is observed that the tensile modulus increased as the filler loading increased. This suggests that the incorporation of these fillers can effectively reduce the mobility of rubber chains and the stiffness of the resulting blend composites are increased (Anyaporn et al, 2017). The tensile modulus increased to the highest value of 11.8009 MPa and 116.0031 MPa for ES-filled and CB-filled NR/SBR blend composites respectively at 40 pphr while tensile modulus decreased at filler loading 50 pphr for ES-filled NR/SBR blend composite (10.0325 MPa) and for CB-filled NR/SBR blend composite (15.1663 MPa), showing that at the filler loading (50 pphr) there is possibility that the composite cannot take up more fillers or poor compounding of the fillers into the matrix. CB-filled NR/SBR blend composites having the higher tensile modulus values than the ES-filled NR/SBR blend composites, because the ES filler show a greater tendency towards filler agglomeration.

The result of elongation at break presented on Table 2 and Figure 4 shows as the filler loading of CB increased, the elongation at break decreased but for ES, the elongation at break decreased at 10 pphr (521.5602%) but increased to 609.9027% at 20 pphr. At filler loadings 30-50 pphr the elongation at break decreased. The observation is expected since the stiff particles would act as restriction sites for the rubber chain movement in application of strain and so tend to reduce the elongation at break of the blend composites. However, the elongation at break of the unfilled NR/SBR blend is largely retained due to the unloading of the fillers within the rubber matrix. The CB-filled NR/SBR blend composites had higher values of elongation at break than that of ES-filled blend composites at all the filler loadings.

Table 2 and Figure 5 show the result of hardness on the NR/SBR blend composites. The hardness of the NR/SBR blend increased as the filler loadings of ES and CB increased. The increase in hardness with increasing filler loading of ES and CB particles is expected due to the restriction of segmental motion of the rubber chain, therefore creating more rigid vulcanizates and the resilience decreases. The hardness from 28.1603 Shore A of the unfilled NR/SBR blend to 52.0344-65.6007 Shore A for ES and 54.0824-72.5310 Shore A for CB by inclusion of filler loadings 10-50 pphr of ES and CB respectively. it was observed that the increased hardness by the addition of CB was much higher than that induced by the addition of ES. This indicates that the CB was evenly distributed within the rubber matrix, creating strong bond between the filler and rubber matrix while ES being an agro-waste might as result to self-agglomeration within rubber matrix resulting in low hardness values than CB-filled NR/SBR blend composites.

Thus, the abrasion resistance of a solid body is defined as its ability to withstand the progressive removal of the material from its surface as a result of the mechanical action of rubbing, scraping or erosive nature (Amoke et al, 2021). The result of abrasion resistance presented in Table 2 and Figure 6 showed that as the filler loading increased for both fillers, the abrasion resistance increased from 0-40 pphr, which show a better abrasion resistance. This observation may be attributed to the degree of dispersion of ES and CB particles within the matrix. This indicates that filler loading is not a function of the measured parameter (Egwaikhide et al, 2007). The abrasion resistance decreased at maximum loading of the fillers (50 pphr). The CB-filled NR/SBR blend composites had higher abrasion resistance than that of ES-filled NR/SBR blend composites.

The compression set decreased as the filler loading increased in the NR/SBR blend as shown in Table 2 and Figure 7. This is expected because as filler loading concentration increased in the rubber matrix, the voids in the matrix is reduced, hence there is decrease in percentage compression (Abode, 2010). The ES-filled NR/SBR blend composites had lower values of compression set than CB-filled NR/SBR blend composites, indicating that the ES-filled NR/SBR blend composites had the better compression set than the CB-filled blend composites. The decrease in compression set decreases the toughness of the blend composites.

The data in Table 2 and Figure 8 showed an increase in filler loading of ES and CB, the flex fatigue of the NR/SBR blend composites decreased. A decrease in flex fatigue may be explained in terms of adherence of the filler to the polymer phase leading to the stiffening of the polymer chain and hence resistance to stretch when strain is applied (Tenebe et al, 2013). As filler loading increases, it could be expected that more filler particles and aggregates will not be dispersed and wetted efficiently by the rubber matrix (Sukru et al, 2008).

CONCLUSION

The assessment of the mechanical properties of egg shell (ES) and carbon black (CB) filled NR/SBR blend composites has been investigated. These fillers have demonstrated their potential reinforcing abilities in NR/SBR blend. The CB-filled NR/SBR blend composites showed better mechanical properties compare to ES-filled NR/SBR blend composites. The CB-filled NR/SBR blend composites had improved tensile strength and tensile modulus compared to ES-filled NR/SBR blend composites tensile strength and tensile modulus at filler loading 10-40phr while the hardness for CB-filled NR/SBR blend composites and that of ES-filled NR/SBR blend composites increased at filler loadings 10-50 pphr. The CB-filled NR/SBR blend composites exhibited high values of abrasion resistance than ES-filled NR/SBR blend composites in all the filler loadings. The abrasion resistance increased as the filler loadings increased except at unfilled which had the least value of abrasion resistance. The compression set and flex fatigue decrease as the filler loadings increased for CB-filled NR/SBR blend composites and ES-filled NR/BSR blend composites. The ES-filled NR/SBR blend composites had the lower values of compression set and flex fatigue than the CB-filled NR/SBR blend composites, showing that ES-filled NR/SBR blend composites had better compression set than CB-filled NR/SBR blend composites.

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