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Fabrication, characterization and mechanical properties of hematite (α-Fe_2O_3) filled natural rubber/low-density polyethylene composites

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ABSTRACT

In this study, natural rubber (NR) and low-density polyethylene (LDPE) were melt blended in a 2-roll mill to fabricate NR/LDPE composites with liquid natural rubber (LNR) and hematite (α-Fe_2O_3) as compatibilizer and filler respectively. The effects of liquid natural rubber (LNR) on the mechanical properties and morphology of NR/LDPE composites were investigated. NR/LDPE composites were prepared in three different compositions of 70/30, 50/50 and 30/70, with filler content at varying concentrations (3, 5 and 7 g) while LNR was kept at 10 g for the modified composites. Results obtained showed increment in the tensile strength, tensile modulus, compressive and flexural properties but with decrease in the elongation at break the α-Fe_2O_3 and LDPE contents increase. The modification of the composites with LNR further enhanced the mechanical properties. FTIR spectrum of LNR revealed a peak at 3395 cm^{-1} suggesting the hydroxyl (OH) end group attached to the LNR chain due to the photochemical degradation of NR to yield LNR. FTIR spectra of the selected composites showed the presence of iron oxide bond (Fe-O) at 571 cm^{-1}, 467 cm^{-1} and 463 cm^{-1} due to the incorporation of α-Fe_2O_3 filler particles in the composites. SEM images showed improved matrix/filler interfacial adhesion induced by the LNR compatibilizer.

KEYWORDS:
hematite, liquid natural rubber, low-density polyethylene, mechanical properties, natural rubber

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INTRODUCTION

Materials made by the combination of two or more different types of fillers are known as hybrid composites. The purpose of filler is to provide the rubber mixes with the suitable processing and functional properties of the composites.[1] Natural and synthetic rubbers have been modified via the incorporation of fillers and other additives to improve their service properties.

By proper selection of fillers the material cost can be significantly reduced.[2] Fröhlich et al.[3] had reported that the effect of filler addition to an elastomeric compound is dependent on the type of surface and its energy status functional groups present and their construction agglomeration ability the capacity to form a structure and its spatial network. To obtain optimal balance of technical properties and to lower the cost of the product hematite was added to the composite. Hematite is readily available cost-effective and environmentally friendly characterized by a small band gap[4] with unique electrical[5] and catalytic properties.[4] It is the most thermochemical stable form of iron oxide under ambient conditions.[6] These characteristic properties make hematite appropriate for a varied range of applications such as catalysts for water splitting[4,7] sensors[8,9] and for chemical reactions drug delivery systems[10] pigments and batteries.[11] No doubt when particles or fillers are added to the polymer matrix stiffness is largely enhanced but strength elongation and impact properties are usually compromised. However, it has been found that hematite can also act as a reinforcing filler in polymer matrix if the particle size is small especially at low filler content.[12–15]

Low-density polyethylene (LDPE) is a thermoplastic polymer produced from ethylene monomer by a free radical polymerization process. It has a density range of 0.910–0.940 g cm^{-3} and high degree long chains which give molten LDPE the unique and desirable flow properties. LDPE is suitable for packaging roofing agricultural electrical cable and other applications.[16] Natural rubber (NR) is a renewable material that is used in great amounts by the defence, medical, transportation, and other allied industries because of its outstanding properties such as elasticity resilience and abrasion resistance. The remarkable properties of NR resulted from strain-induced crystallization with the increased modulus on the stretching process.[17] However NR is easily attacked by oxidizing reagents is highly sensitive to sunlight exposure and has low resistance to petroleum.[18,19] These shortcomings are a major barrier to using NR in many industrial applications. Hence, the need to modify NR to improve its service and properties.[20,21]

Thermoplastic natural rubber (TPNR) describes a polymeric blend that is composed of natural rubber (NR) and thermoplastic, such as polypropylene, polystyrene, polyethylene, etc.[22] TPNR are a polymorphous system made of hard and soft regions and in recent times have gathered lots of interest owing to a vast array of their intermediary characteristics, production ease and less expensive cost.[22,23] Moreover, owing to the form of the TPNR, it has drawbacks in certain physical and mechanical properties due to the challenge encountered in blending the constituent polymers. Due to the poor interfacial adhesion between NR and LDPE, LNR amongst other compounds has been employed as a compatibilizer, hence enhancing the mechanical properties of the TPNR blends. These compatibilizing agents serve to enhance the miscibility of the constituent polymers by lowering the interfacial tension between the different segments, thereby promoting the interlinking of the segments.[24,25] In addition, the mechanical properties of TPNR like all other polymeric materials are improved by the infusion of fillers.[26,27] The capability of fillers to have physical and/or chemical interconnection with TPNR compounds in proper conditions is also a significant aspect of reinforcement. While the physical and chemical makeup of the infused filler substance will determine its potency as a reinforcement material, the measure to which this occurs is dependent on several factors such as the filler-polymer interconnection, filler amount present and possibly filler-filler interconnection.[28,29] Furthermore, uniform distribution and proper

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dispersion of the filler substances are important for the mechanical strengthening of every polymer including TPNR.[30] Particles like magnetite (Fe_3O_4), α-Fe_2O_3, aluminium oxide (Al_2O_3), titanium oxide (TiO_2) silicon dioxide (SiO_2) and lead oxide (PbO) when added into a polymer matrix may result to a marked enhancement of various mechanical properties of the resultant composites.[28,31–34]

Nurhajati et al.[35] noted that appreciation in NR and bismuth trioxide (Bi_2O_3) filler resulted in depreciation of the tensile strength and elongation at break of linear low-density polyethylene/natural rubber (LLDPE/NR) composites. Ramlee et al.[36] in their investigation reported that infusion of TiO_2 nanofillers caused an improvement in the tensile strength and hardness of polyvinylchloride/epoxidized natural/titanium oxide (PVC/ENR/TiO_2) nanocomposites. Jiang et al.[37] postulated that composites made from thermoplastic elastomers filled with Fe_3O_4 nanoparticles displayed a marked increase in tensile strength and elastic recovery upon infusion of Fe_3O_4 nanoparticles.

According to Mirjalili et al.[38] the incorporation of an increasing amount of nanosized α-Al_2O_3 filler particles (1 to 4 wt.) in the PP matrix brought about enhancements in the tensile strength flexural strength and flexural modulus of the resultant composites. On the other hand further increase in the α-Al_2O_3 loading yielded a decrease in the aforementioned investigated mechanical properties of the composites owing to agglomeration of α-Al_2O_3 particles. Altan et al.[39] noted that the incorporation of metal oxides such as TiO_2 and ZnO nanoparticles as fillers in increasing amounts (1%, 3% and 5%) into the PP matrix led to an increase in yield strength tensile strength and elastic modulus of the resultant composites. However, elongation of the composites decreased with increase in the aforementioned metal oxides. Piffer et al.[40] observed that an increase in iron ore tailings (IOT) comprised of Fe_2O_3 in composites of PP and maleic anhydride grafted PP increased the elastic modulus. On the other hand the ultimate tensile, strength elongation, at, break and impact strength of the composites declined with increasing IOT loading. Lin et al.[41] stated that regardless of the low amount of nano ZnO filler particles (1 wt.%) the tensile strength tensile modulus and elongation at break of the prepared PP/nano ZnO composites increased markedly.

Teoh et al.[42] have reported the improvement of tensile strength of NR/LDPE with the addition of silica into the blends. The effect of particulate fillers on natural rubber/high-density polyethylene blends for roofing application has been studied by Wickramaarachchi et al.[43] They reported that six particulate fillers viz. calcium carbonate barium sulphate kaolin talc snobrite clay and dolomite were used to prepare natural rubber/high-density polyethylene blends to ascertain the most suitable filler for roofing application. It was observed that the addition of talc dolomite and kaolin to the NR/HDPE blend showed reduced impact strength which is the most important property of a roofing application. However the other three fillers showed improved impact strength at specific loadings. Sampath et al.[44] investigated the effect of an organotitanate coupling agent on the properties of calcium carbonate filled low-density polyethylene and natural rubber composites. They reported a progressive increase in the mechanical properties studied with optimum properties at a coupling agent content of 0.7 pphp (parts per hundred of polymer). Ahmad et al.[45] in their study reported that the partial replacement of Aramid short fibres by clay at a certain proportion did improve the mechanical properties and better surface morphology of the composites particularly with the 10 liquid natural rubber (LNR).

Not much has been done in the use of α-Fe_2O_3 particles as filler for matrix based on NR/LDPE. Hence this work presents the preparation of NR/LDPE composites filled with particles of locally sourced hematite (α-Fe_2O_3). Also we attempt to characterized and investigate some mechanical properties of the resultant NR based composites filled with hematite (α-Fe_2O_3) filler particles. To the best of our knowledge the use of hematite from Katsina State in Nigeria as a filler for NR/LDPE in the presence of LNR as a compatibilizer has not been reported.

MATERIALS and METHODS

Materials

NR latex was obtained from the Rubber Research Institute of Nigeria (RRIN), Nigeria. Ribbed smoked sheet natural rubber with plasticity retention of 64 was supplied by Stevemoore Chemical Co. (Zaria, Nigeria). Zinc oxide (ZnO), stearic acid, sulphur, mecarptobenzothiazole (MBT), 1, 2-dihydro-2, 2, 4-trimethylquinoline (TMQ), toluene and hydrogen peroxide (H_2O_2), all of the analytical grades were also purchased from Stevemoore Chemical Co., Zaria. LDPE with a density of 0.92 g cm^{-3}, melt flow index (MFI) of 2.4 g per 10 min and crystalline melting temperature of 115 °C was purchased from Ceeplast Industries Nig. Limited, Aba, Nigeria. Hematite lumps were obtained from natural deposits in Kastina State, Nigeria and were crushed to 10 μm size particles.

Preparation of liquid natural rubber (LNR) compatibilizer

The preparation of LNR followed a method adopted by Ngo et al.[25] Briefly, 4 g of NR latex was dissolved in 40 ml of toluene for about 15 min. Then, 10 ml of H_2O_2 was introduced into the solution of NR latex and toluene and stirred for 15 min. The resultant mixture was placed into a dish and kept in a UV irradiation box for 1h. LNR produced was removed from the UV irradiation box and the supernatant layer of toluene was decanted. The prepared LNR was rinsed with distilled water and dried in an oven at 70 °C for 24 h.

Preparation of NR/LDPE/hematite composites

NR/LDPE/Hematite composites were made according to the method adopted by Mokhtar et al.[46] but with slight modifications. Briefly, LDPE was first melted in a 2-roll mill at 140 °C for 3 min. NR was added and left to melt for 3 min. Other ingredients save sulphur were introduced into the admixture and left to mix for 3 min. Hematite (α-Fe_2O_3) particles were incorporated into the admixture and left to mix for 3 min. Finally sulphur was introduced and the mixture was left to mix for another 3 min. Thereafter the resultant admixture was placed in a 3 mm thick mould and compressed in a compression moulding machine at 140 °C and 4 MPa for 7 min. The compressed sheets were stored for further use.

Preparation of NR/LDPE/LNR/hematite composites

The fabrication of NR/LNR/LDPE/Hematite composites was accomplished according to the methods used by Suaysom and Keawwattana[47] and Ngo et al.[25] LDPE was first charged in a 2-roll mill at 140 °C for 3 min. Then NR was introduced and allowed to melt for 3 min before LNR was added and left to intermix with the admixture of LDPE and NR for 3 min. The addition of other ingredients followed a similar pattern as stated in the preparation of NR/LDPE/Hematite composites.

In all the compounding formulations, material composition comprises NR/LDPE (70/30, 50/50, 30/70), Hematite (3, 5 7 g), LNR (10 g), ZnO (5 g), sulphur (2 g), stearic acid (2 g), MBT (1.5 g) and TMQ (1 g) as the case maybe.

Chemical composition analysis of hematite (α-Fe_2O_3)

Chemical analysis of the as-obtained hematite (α-Fe_2O_3) filler sample was achieved with the aid of EDX-720 X-ray Fluorescence (XRF) spectrometer.

Mechanical properties test

Tensile compression and flexural properties were determined using an Instron Universal Testing Machine (Instron ID No. 3369K1781) having a capacity of 50 kN (11 250 lb). Investigation of the tensile properties was done in line with ASTM D638. Compressive strength

and compressive modulus of the samples were investigated in line with ASTM D3410/D3410 M. Flexural properties of the composite samples were investigated in line with ASTM D7264/ASTM D7264 M-07. Three samples representing three measurements were used for each test and an average of the results was taken as the resultant value.

Characterization of, NR/LDPE/LNR/hematite, composites

The physicochemical properties of NR, LNR and NR/LDPE/LNR/Hematite composites were analyzed using SEM and FTIR. Fourier Transform Infrared Spectroscopic analysis (FTIR) of NR LNR and selected composites were studied with the aid of Shimadzu FTIR-8400S machine in the range of 400–400 cm^{-1} with a resolution of 4 cm^{-1}. The dispersion of the filler particles in the matrix was studied with the aid of the Carl Zeiss Scanning Electron Microscopy (SEM) machine Model EVO LS10. Samples were fractured in liquid nitrogen and coated with thin carbon film to avoid charge built up due to their low conductivity.

RESULTS and DISCUSSION

XRF analysis of hematite (α-Fe_2O_3) filler

Table 1 presents the result of the chemical analysis of iron ore from Katsina state using XRF in weight per cent. The ore contains 54.118% Fe_2O_3 and 34.289% SiO_2 as major constituents with 7.027% TiO_2, 1.768% CaO, 1.337% ZrO_2, 0.382% MnO, 0.331% V_2O_5, 0.263% ZnO, 0.147% NiO and 0.129% Cr_2O_3 as minor constituents and K_2O, NbO and PbO as traces. It is important to note that the quality of raw iron ore and its sustainability for commercial utilization is largely determined by its chemical composition.[48] Iron ores as expected should have high Fe contents and low impurity element contents to justify investment during exploitation. From the XRF results the iron content is high compared to most iron ores from other parts of Nigeria. Similar results have been reported by Agava et al.[49] and Salawu et al.[50] confirming the predominantly presence of hematite from most the iron ores sourced from Nigeria.

Mechanical properties of the NR/LDPE filled composites

The mechanical properties of composite materials are greatly dependent on some factors such as the filler content filler orientation filler length properties of the filler reinforcement and matrix and processing method and condition. Hence, the effect of LNR, filler and LDPE contents on the mechanical properties of the prepared NR/LDPE composites has been evaluated below.

Table 1. XRF of hematite filler sample

Analyte	Content,%
Fe_2O_3	54.118
CaO	1.768
TiO_2	7.027
Zr O_2	1.337
Cr_2O_3	0.129
ZnO	0.263
NiO	0.147
V_2O_5	0.331
K_2O	0.082
SiO_2	34.289
MnO	0.382
NbO	0.074
PbO	0.053

Note: No of measurement (n = 3)

Tensile strength and tensile modulus

Effect of Filler Content: It was observed that a rise in the filler content resulted in the increment of the tensile strength (TS) and tensile modulus (TM) of the composites (Figures 1 and 2). The composites achieved peak values of TS and TM at 7 g filler content. Then increase in the filler content was believed to generate adequate stress transfer and amplification in the reinforcement experienced therefore causing an increase in TS and TM of the composites. These results are in agreement with previous research works by Alam and Choi,[51] Chavan et al.[52] and Channapagoudra et al.[53] Alam and Choi [51] investigated the reinforcing and magneto mechanical properties of natural-rubber (NR) composites using iron oxide/multilayer graphene (MLG) as hybrid filler. The results showed that magneto-sensitive mechanical properties through the incorporation of iron oxide of about 2.91 vol.% iron oxide were greatly improved, the tensile strength of the composite with the iron oxide/MLG hybrid filler was 160% higher than that of unfilled rubber. Chavan et al.[52] studied the fabrication and evaluation of mechanical properties of Fe_2O_3 particulate filled hybrid composites at three different filler content of 6, 8 and 10%. They reported that the filled composites exhibited high tensile strength compared to the unfilled composites. It was revealed that the composite with 6% Hematite ore (filler) of 40% iron content showed the highest tensile strength (249.17 N mm^{-2}) compared with the rest. The performance could be linked to improving particle dispersion and strong polymer/filler interface adhesion ensuring effective stress transfer. Channapagoudra et al.[53] studied the effect of hematite filler on the mechanical properties of glass/epoxy composites at varying hematite concentrations of 5, 10 and 15%. In their study they reported the increase in tensile strength of hematite filled glass/epoxy composites is greater than the unfilled composites with optimum strength at 10% filler content and showed a decline beyond this value, thus attributing the decline to weak

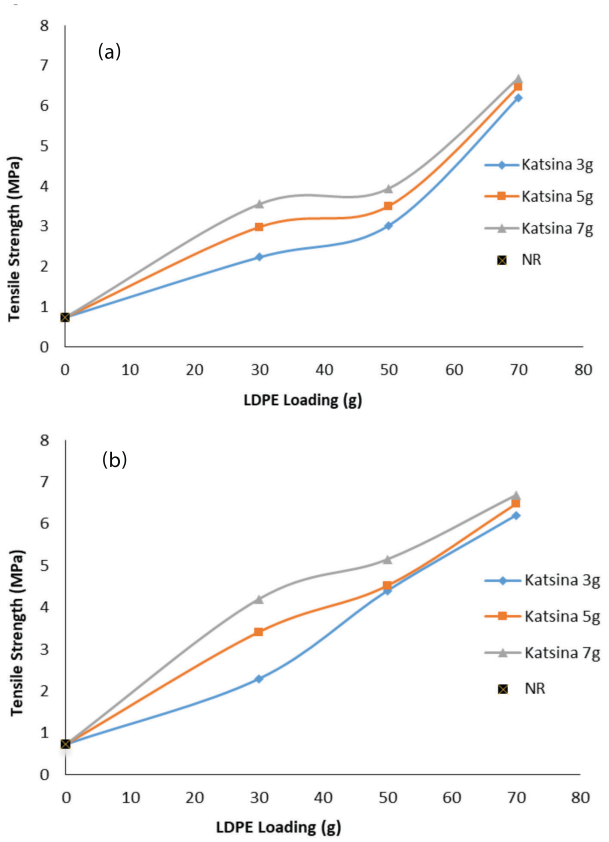

Figure 1: (a) Tensile strength of NR/LDPE composites and (b) TNR/LDPE/LNR composites; (n = 3)

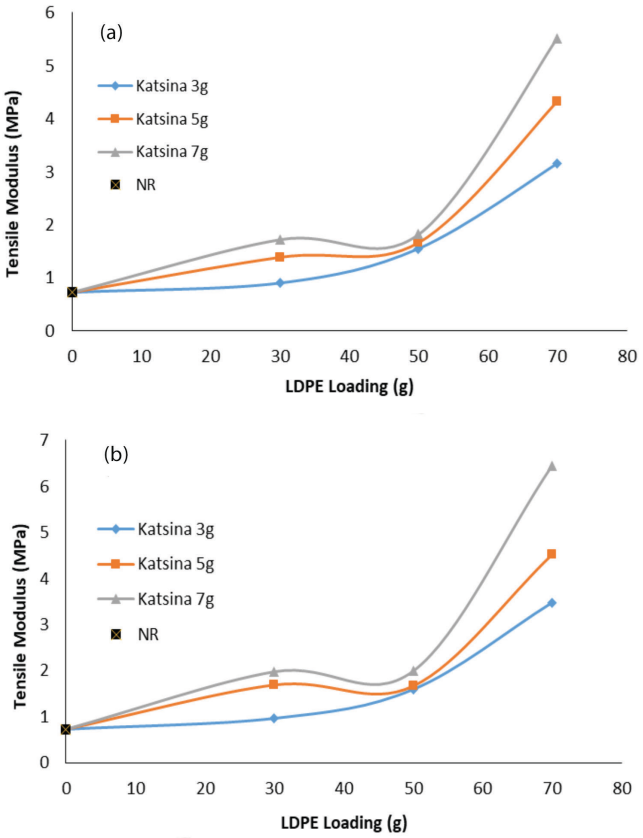

Figure 2: (a) Tensile modulus of NR/LDPE composites and (b) NR/LDPE/LNR composites; (n = 3)

bonding strength between the components interface at high filler content. Also, Ismail et al.[54] in their investigation reported that the tensile modulus of recycled NR/PP composites increased as the palm oil fuel ash (POFA) content increased up to 10 phr. They attributed the addition of POFA to have caused a decline in chain mobility with a corresponding increase in the stiffness of the composites.

Considering the LDPE content in the NR/LDPE Composites there is an increment in the TS and TM of the prepared composites as the LDPE content increases (Figures 1 and 2). As expected the increment in the amount of LDPE is believed to confer on the composite improved strength and stiffness.[55] Similar observations have been reported in the investigations carried out by Ahmed[56] and Ngo et al.[25] Ahmed[56] worked on the preparation of blends of acrylonitrile-butadiene rubber/high-density polyethylene (NBR/HDPE) with chloroprene, rubber (CR) as compatibilizer in the presence of marble waste (MW) as filler. He investigated the effects of the blend ratio and CR on cure characteristics and mechanical and swelling properties of MW-filled NBR/HDPE composites and reported that an increase in the HDPE content resulted in an increase in the tensile, strength tear modulus hardness and cross-link density of the composites. Ngo et al.[25] in their study of the antibacterial nanocomposites based on Fe_3O_4–Ag hybrid nanoparticles and natural rubber-polyethylene blends reported that the addition of the HDPE component to the NR matrix produced a higher tensile strength value than neat NR and further increased when the NR/HDPE composites was treated with compatibilizer.

Effect of LNR: From Figures 1(a) and 2(a) it can be seen that the composites without LNR displayed low values of TS and TM when compared with composites with LNR (Figures 1(b) and 2(b)). The results show that the incorporation of LNR promoted vulcanization with improved filler dispersion and good interfacial bonding between NR and LDPE thus attaining remarkable improvements in the TS and TM of the NR/LDPE composites. The strong adhesion was assumed to promote improved stress transferability due to increasing matrix/filler interphase interlocking in the composites. For instance at 7g filler content the values of TS and TM increased from 6.48 MPa and 5.52 MPa to 6.69 MPa and 6.45 MPa respectively. It is expected the active sites present on LNR like –OH, –OOH and –OR would improve the matrix-matrix and fibre-matrix interactions.[57] Bijarimi et al.[58] and Jamil et al.[59] reported similar trends in their work regarding the use of LNR as a compatibilizer to improve the properties of rubber composites. Bijarimi et al.[58] investigated the toughening effect of liquid natural rubber on the morphology and thermo-mechanical properties of the poly (lactic acid)(PLA) ternary blend. They reported that PLA–LNR–LLDPE (linear low-density PE) exhibited enhanced strength compared to virgin PLA and PLA–LLDPE blend and believed that LNR performed acted as a better toughening reinforcing agent. Ahmad et al.[60] investigated the thermal and mechanical properties of natural rubber filled with varying compositions of high-density polyethylene using LNR as a compatibilizer. They reported that the tensile properties of the modified NR improved considerably when liquid natural rubber (LNR) was added to the composite.

Elongation at break

Effect of Filler Content: Figure 3 reveals that increment in the filler content brought a downward trend in the elongation at break of the composites. Then, the decrease experienced could be attributed to the consequential rise in the degree of reinforcement occasioned by the rising filler content. This resulted in the uncoiling of the molecular chains of the makeup of the composite. The report is in line with the study done by Piffer et al.[61] who investigated the evaluation of mechanical and thermal properties of polypropylene (PP)/iron ore tailing (IOT) composites and reported a decrease in the elongation at break on the addition of IOT. Again the addition and gradual increase in the LDPE content also showed a progressive reduction in the elongation at break of the composites (Figure 3). The result is expected because LDPE as a thermoplastic polymer possesses greater rigidity and lower measure of elasticity[62] than natural rubber. Also Ahmed[56] worked on the preparation of chloroprene rubber (CR) compatibilized acrylonitrile butadiene rubber/high-density polyethylene (NBR/HDPE) filled with marble waste reported that the elongation at break decreased with the increase in the LDPE content.

Effect of LNR: Composites without LNR (Figure 3(a)) recorded lower values for elongation at break than those with LNR as illustrated in Figure 3(b). As earlier advanced the intermolecular and interfacial interactions between the various component of the composites are made possible by the presence of an LNR compatibilizer.[25] Moreover LNR to some measure serves as a processing ingredient thus increasing the rubbery portion in the LNR-containing composites.[63]

Compressive properties

The effect of LNR, filler and LDPE contents on the compressive strength and compressive modulus for modified and unmodified NR/LDPE composites are presented in Figures (4 and 5).

Effect of Filler Content: From Figures 4a and 5a it is observed that the compressive strength (CS) and compressive modulus (CM) of the composites showed a direct relationship with the addition of filler. The behaviour suggests that the increase in filler content increases the properties under study. As previously acknowledged this observed situation could be linked to good filler-matrix interaction and enhanced stress transfer. These reports are in agreement with the reports put forward by Gencel et al.[64] and Dankwah.[65] Gencel et al.[64] worked on the mechanical properties of polymer concretes containing different amounts of hematite or colemanite and evaluated the mechanical performance properties including the compressive strength of the composites. They reported that the incorporation of hematite offers great improvement of the mechanical properties studied when compared with the composites filled with silica and sand alone. Also Figures 4(a) and 5(a) reveal that there is a corresponding increase in CS and CM of the composites as LDPE

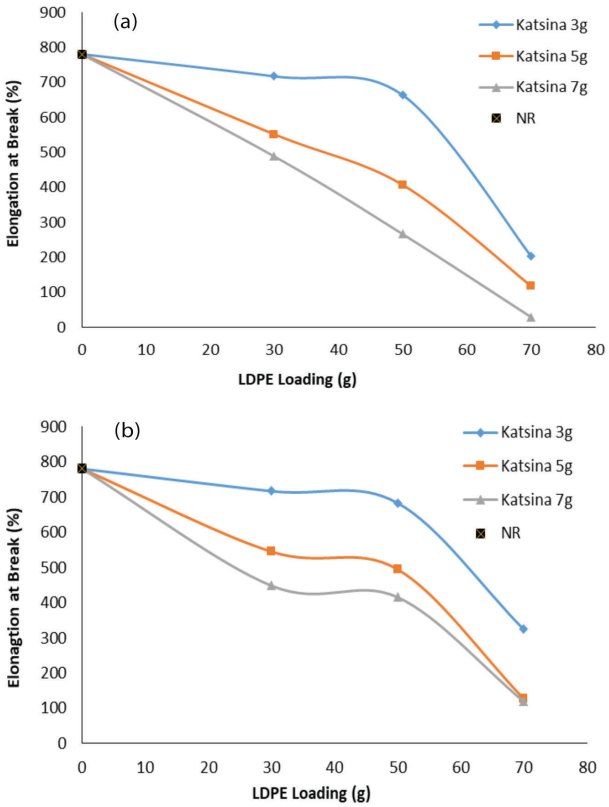

Figure 3: (a) Elongation at break of NR/LDPE composites and (b) NR/LDPE/LNRE composites; (n = 3)

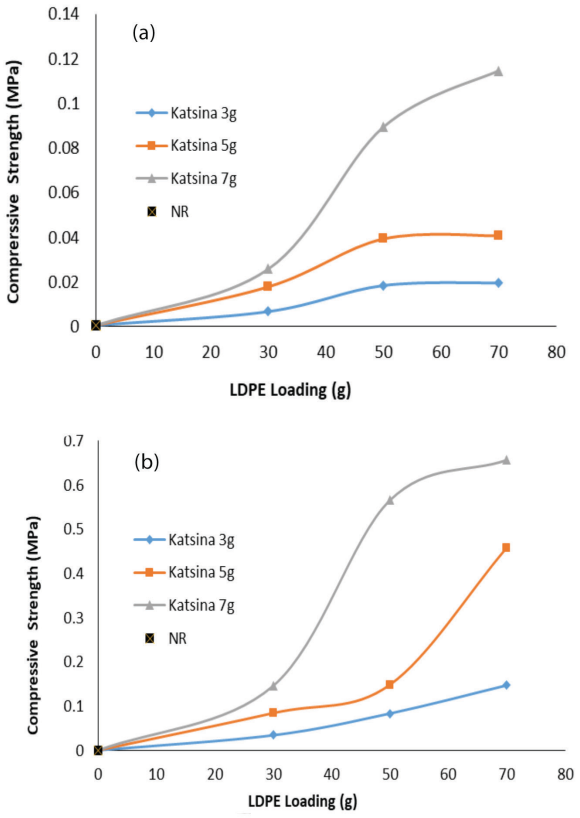

Figure 4: (a) Compressive strength of NR/LDPE composites and (b) NR/LDPE/LNR composites; (n = 3)

content in the composite is increased. This observation was also attributable to the steady increase in the semi-crystalline LDPE nature and the synchronous decline in the amorphous nature of the elastomeric NR.[66]

Effect of LNR: The effect of LNR on the CS and CM of the NR/LDPE composites are shown in Figures (4(b) and 5(b)). It is evident from the figures that the addition of LNR to the composites further enhanced the compressive strength and compressive modulus. The results showed that at 5 g filler content the values of CS and CM increased from 0.041 MPa and 0.115 MPa to 0.046 MPa and 0.135 MPa, respectively. As earlier reported the increase in values of the properties under study was a result of improved intermolecular cohesion and interfacial bonding of NR and LDPE portions owing to the presence of LNR.[25] It has also been reported that the addition of liquid natural rubber as a compatibilizer could be presumed to decrease the interfacial tension vis-à-vis forming physical or chemical bonds with PE molecules.[67]

Flexural properties

The flexural strength (FS) and flexural modulus (FM) of NR composites filled with hematite filler and low-density polyethylene have been investigated (Figures 6 and 7). Also, the effect of the addition of liquid natural rubber to composites on flexural properties was evaluated.

Effect of filler content: The flexural strength (FS) and flexural modulus (FM) behaviour of the NR/LDPE composites on the addition of hematite filler are illustrated graphically in Figures 6(a) and 7(b) respectively. It is evident from the figures that the incorporation of the filler gave rise to an increase in the values of the FS and FM. It is reasonable to state that the hematite filler acted as a reinforcing filler. The rise in the FS and FM also indicated that there was a satisfactory mix of the matrix and filler particles as the filler content increased.[64] This investigation is similar to the investigation reported by Chauhan et al.,[68] Channapagoudra et al.[53] and Chavan et al.[52] Channapagoudra et al.[53] studied the effect of hematite filler material on the mechanical properties of glass/epoxy composites prepared via the hand lay-up technique. The tensile flexural impact and hardness behaviour of the

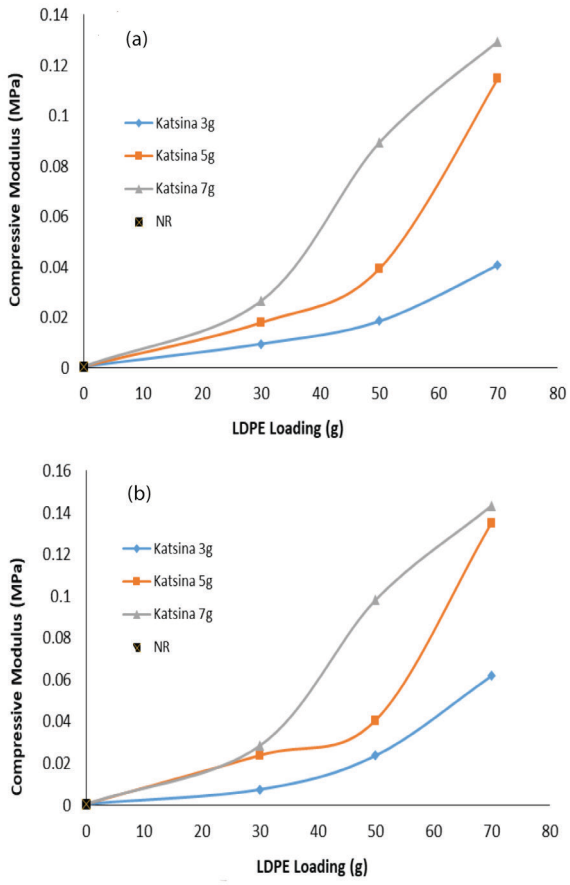

Figure 5: (a) Compressive modulus of NR/LDPE composites and (b) NR/LDPE/LNR composites; (n = 3)

resultant composites were investigated. The results obtained showed that the hematite filled composites had enhanced over the unfilled glass/epoxy composites. Chauhan et al.[68] investigated the mechanical properties of glass/orthophthalic polyester resin composites filled with hematite filler at three different contents (3, 6 and 10 wt.%). They concluded that the incorporation and increase of hematite filler in the composites increased the flexural strength. Chavan et al.[52] on their part conducted a study on the effect of hematite ore and volume fraction on the mechanical properties of Fe_2O_3 particulate filled hybrid composites and reported that the addition of hematite ore had a strong and positive effect on the properties.

Also from Figures 6(a) and 7(a) the rise in FS and FM as the LDPE content increases was noticed. It is observed that the incorporation of LDPE in NR resulted in an increase in the flexural strength of the composites. The increase in FS could be ascribed to the existence of LDPE as a rigid component in NR. However such a hard fraction acts as a filler thus increasing the FS of the component. Simply put the flexural strength of rubber vulcanizates usually rises due to the addition of polyethylene to natural rubbers.[69] Again the increase in the LDPE content of the mixture resulted in the elasticity reduction. It is reasonable to say that at higher LDPE content the natural rubber phase remains as distributed particles. The LDPE phase therefore contributed to the increased FM of NR/LDPE composites. Similar results have been reported by Ahmed[56] and Wickramaarachchi et al.[55]

Effect of LNR: It is evident that the FS and FM of the composites with LNR exhibited higher improvement than the composites without LNR (Figures 6(b) and 7(b)). For instance at 7 g filler content the values of FS and FM increased from 9.82 MPa and 153.67 MPa to 14.66 MPa and 169.26 MPa respectively. The outcome is due to the bonding role played by LNR which led to improved filler dispersion and interfacial adhesion of NR and LDPE domains. Other researchers have reported a similar trend involving NR-based blend/composites compatibilized with LNR.[24,25,63,70,71]

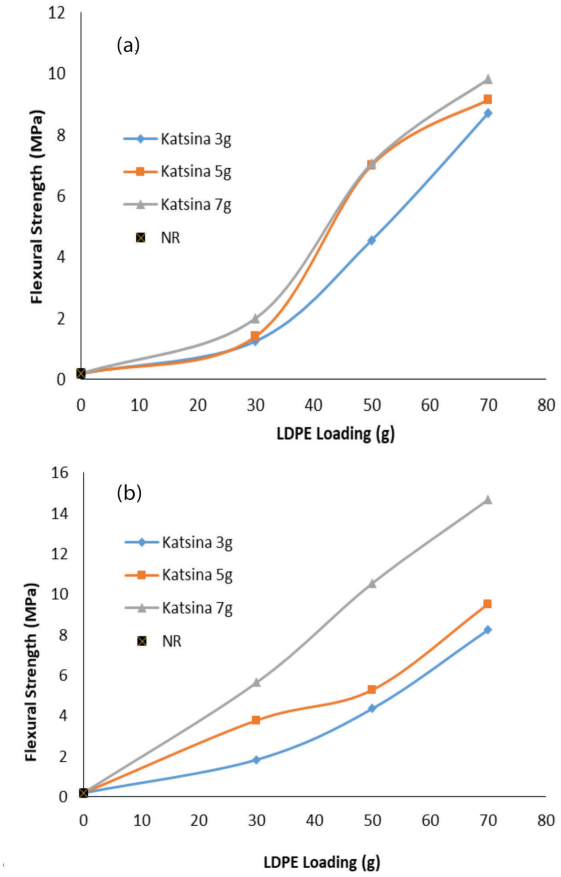

Figure 6: (a) Flexural strength of NR/LDPE composites and (b) NR/LDPE/LNR composites; (n = 3)

Physicochemical characterization of NR, LNR and NR/LDPE composites

FTIR analysis

The FTIR spectra of NR and LNR are illustrated in Figures 8(a) and 8(b) respectively. As expected Figure 8(a) displayed some characteristic peaks of NR such as carbon-carbon double bond (C=C) stretch at 1634 cm^{-1}; CH_2 and CH_3 deformation at 1456 cm^{-1}; and 837 cm^{-1} for C-H out of plane bending vibration.[72] In Figure 9(b), specific new peaks were observed indicating possible degradation of NR leading to the formation of LNR. Such peaks include 1400 cm^{-1} denoting methylene (CH_2) and methyl (CH_3) deformation 1632 cm^{-1} denoting carbon-carbon double bond (C=C) stretch. Moreover a broad peak at 3395 cm^{-1} suggests hydroxyls (OH) stretching and vibration. The presence of the OH group is attributed to the photochemical degradation of NR.[25] These peaks validated the conversion of NR to LNR.

The 30NR/70LDPE and 30NR/70LDPE/LNR composites for 7 g Hematite content were used as representative samples for this study. The FTIR spectra of these samples are presented in Figures 8(c) and 8(d). From the figures, it is evident that selected composites contain the characteristic peaks of NR and LDPE. Some of the NR characteristic peaks in the composites as shown in Figures 8(c) and 8(d) include 1663 cm^{-1} and 1655 cm^{-1} for C=C stretch; 837 cm^{-1} and 839 cm^{-1} for C-H out of plane bending vibration [72] respectively and 719 cm^{-1} for C-H bends.[25] Some characteristic peaks of LDPE as seen in Figures 9(c and d) are 2918 cm^{-1} and 2851 cm^{-1} for CH stretch in backbone of the polymer chain; 1466 cm^{-1} form ethylene (CH_2) bend; 1375 cm^{-1} and 1377 cm^{-1} for methyl (CH_3) bend; and 719 cm^{-1} for CH_2 rock.[73,74]

The FTIR spectrum of 30NR/70LDPE composite containing hematite filler showed characteristic peaks of hematite at 571 cm^{-1}

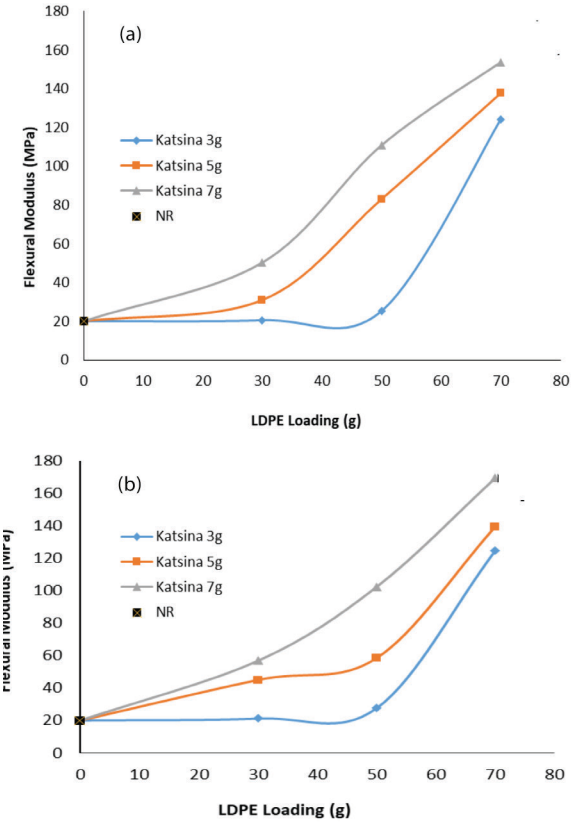

Figure 7: (a) Flexural modulus of NR/LDPE composites and (b) NR/LDPE/LNR composites; (n = 3)

and 467 cm^{-1} which suggest an iron oxide bond (Fe-O) stretching and vibration respectively. The spectrum of 30NR/70LDPE/LNR composite filled with hematite gave a characteristic peak of hematite at 463 cm^{-1} indicating Fe-O vibration.[75] The Fe-O peaks are not present in the spectra of NR and LNR as presented in Figures 8(a) and 8(b). The absence of this type of peak further confirms the presence of hematite in the composite samples prepared. Moreover the band between 2727.44 cm^{-1} and 1897 cm^{-1} in Figure 8(c) reduced in intensity in Figure 8(d) (2725 cm^{-1} and 1879 cm^{-1}). The scenario reflects the presence of a compatibilizer.[76] Again it is clear that the FTIR spectra of the representative composites did not differ much from each other. The behaviour indicates a physical kind of interconnection of the hematite particles and the matrices.[77] Similar behaviour had been reported in the works of Mussatti et al.[78] and Belgin and Aycik.[79]

SEM Morphological analysis

The micrographs and phase behaviour of the LNR modified NR/LDPE system were obtained from the SEM images of fracture surfaces. The representative SEM images of NR LNR and NR/LDPE composites are presented in Figure 9 As stated before 30NR/70LDPE and 30NR/70LDPE/LNR composites for 7 g Hematite content. It is evident that in Figures 9(a) and 9(b) latex particles of NR before photochemical degradation are not as conspicuous as latex particles of LNR after the same degradation process. It is also evident that the LNR latex particles appear as clear spherical shaped particles. A similar report has been reported by Ibrahim et al.[80] who studied the preparation characterization and properties of liquid natural rubber with low non-rubber content via photodegradation. Figures 9(c) and 9(d) represent NR/LDPE and NR/LDPE/LNR filled with hematite particles at 7 g content respectively. As evident in the figure α-Fe_2O_3 filler particles are presented as distinguishable white spots dispersed in the matrix. Belgin and Aycik[79] had reported a similar appearance the presence of particles of mineral fillers in their study on the effect of particle size of mineral fillers on polymer-matrix composite shielding materials against ionizing electromagnetic radiation. Furthermore Figure 9(c) displays a disjointed system owing to interfacial tension existing between NR and LDPE segments brought about by the absence of a compatibilizer. On the other hand Figure 9(d) shows a homogeneous interface between NR and LDPE constituents and to a reasonable degree the filler particles. The observed scenario could be attributed to the presence of LNR; thus performing its role as a compatibilizer in ensuring improved matrix/filler interaction with a reduction in the interfacial tension between NR and LDPE segments. The improvement in the interfacial adhesion between matrix and fillers using compatibilizers such as LNR has been reported by Ngo et al.[25]; Guo et al.[81]; Mussatti et al.[78] and Belgin and Aycik.[79]

CONCLUSION

The result shows that liquid natural rubber (LNR) has been demonstrated to be a good compatibilizer for natural rubber blends. LDPE and LNR jointly enhanced the property performance of NR/LDPE composites. Tensile strength, tensile modulus compressive and flexural properties were found to improve significantly with the addition of hematite (α-Fe_2O_3) fillers however with a reduction in the elongation at break. The composites containing LNR were homogeneous and with optimum mechanical properties found around the 7 g filler content. Morphology of the composites revealed apparent compatibility of NR and LDPE in the presence of LNR compatibilizer with further confirmation shown by the FTIR spectra.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

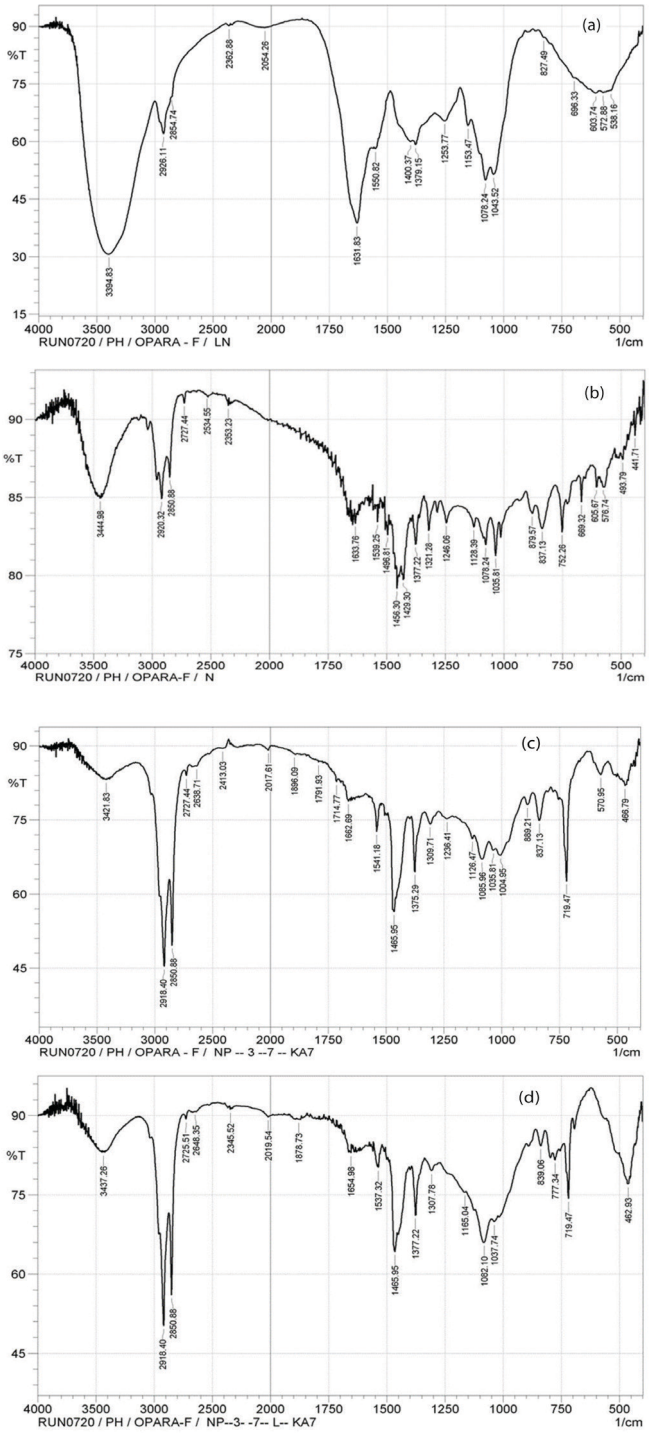

Figure 8: FTIR spectrum of (a) NR; (b) FTIR; (c) 30NR/70LDPE and (d) 30NR/70LDPE composites; (n = 1)

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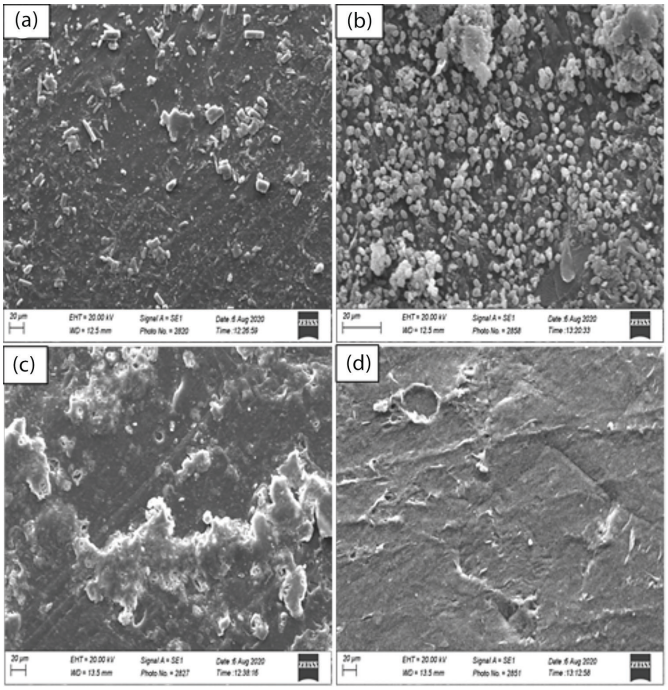

Figure 9: SEM micrographs of (a) NR (b) LNR (c) 30NR/70LDPE/α-Fe2O3 and (d) 30NR/70LDPE/LNR/α-Fe2O3 composites; (n = 1)

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