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Research Article

INVESTIGATING THE STRUCTURAL PERFORMANCE OF HIGH-DENSITY POLYETHYLENE COMPOSITES REINFORCED WITH OPEN-AIR AND FURNACE RICE HUSK ASH

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Abstract

High-density polyethylene (HDPE) stands as a versatile thermoplastic polymer widely used across diverse industries, ranging from household appliances to automotive components and packaging. Despite its advantageous properties, such as durability and chemical resistance, HDPE faces challenges related to poor weather resistance. To overcome these limitations and enhance HDPE's performance while maintaining cost-effectiveness, various filler materials have been explored. Natural fibers like sisal, kenaf, and bagasse, among others, have been investigated for their potential as fillers to blend with HDPE, as reported in existing literature. Rice husks (RH), a byproduct of rice milling, represent a particularly abundant filler material. Incorporating RH into HDPE matrices offers an opportunity to improve its properties and widen its application spectrum. In this paper, we explore the feasibility and effectiveness of utilizing RH as a filler for enhancing HDPE's performance. Drawing upon previous studies and reviews, we analyze the impact of RH filler on HDPE properties, including mechanical strength, thermal stability, and weather resistance. Through a comprehensive examination of the literature, we highlight the potential benefits and challenges associated with RH-filled HDPE composites. Additionally, we discuss various processing techniques and formulations employed in the fabrication of RH-filled HDPE composites, aiming to optimize their performance and compatibility. By synthesizing findings from different research works, this study provides valuable insights into the utilization of RH as a sustainable filler material for enhancing the properties of HDPE and expanding its application range in diverse industrial sectors.

Keywords: High-density polyethylene (HDPE), Rice husk (RH), Filler materials, Composite materials, Polymer enhancement

INTRODUCTION

High-density polyethylene (*HDPE*) is a thermoplastic 1995; Zhang et al., 2004), it has found diverse polymer produced from the monomer ethylene, and one application that include household appliances, of the most important commercial polyolefin. Due to its automobiles, wiring and cables, aeronautics and excellent combination of properties (Diraddo and Laroche, packaging. However, HDPE and its wide use have drawbacks that emanates from its poor weatherability (Khan and Ahmed, 2003; Zebarjad et al., 2006). To improve HDPE properties and reduce cost, fillers such as natural fibers (Lei et al., 2007), sisal fibers (Li et al., 2008), Kenaf fibers (Salleh et al., 2014) back fibers (Yemele et al., 2010) can be blended with HDPE amongst others as in the reviewed in various literature (Sarasini and Fiore, 2018; Dashtizadeh et al., 2017; Ng et al., 2020). Rice husks (RH), are a byproduct of rice milling process and produced in large quantities. For instance, the world rice production was approximately 782 million tonnes in 2018 (FAO, 2020). Rice husks constitute 20 % of rice paddy, and its composition is as follows: cellulose (50%), lignin (25–30%), silica (15– 20%), and moisture (10–15%). Bulk density of rice husk is low and lies in the range 90–150 kg/m^3 (Singh, 2018). These proportions may however change depending on the geographical conditions, rice variety, methods of sample preparation and analysis among others (Chandrasekhar et al., 2003; Johar et al., 2012). Rice husk has low commercial value, and when burnt to extract energy, the ash is of low value and most of it ends up in landfills (Govindarao, 1980; Shivam et al., 2019). This among other reasons has attracted research interests to use ash obtained from burning rice husks as fillers in thermoplastic polymers (Zaaba and Ismail, 2019). The use of RHA as a filler in HDPE offers a twopronged advantage, that is, it reduces pollution of both waste rice husks and HDPE (Kohl et al., 2020; Van Thanh et al., 2020), and it ultimately leads to production of a novel material with its properties enhanced (Abdul Azam et al., 2020; Collazo-Bigliard et al., 2019). This was the motivation of this study.

Formulation of RHA-HDPE composites and their characterization has been undertaken in some studies in different ways. For instance, in their study on rice husk ash as a valuable reinforcement for high density polyethylene, Ayswarya et al. (2012) were able to formulate RHA-HDPE composites of different percentages. Compatibilization was first done where the compatibilizer (MA-g-HDPE) was a grafting reaction between maleic anhydride (MA) and HDPE with the help of dicumyl peroxide (DCP) initiator at 145℃. The study amongst others like, Ginting et al. (2017) and Daramola et al. (2015), did not consider higher percentages of RHA neither did they consider the properties of the composites made by RHA from uncontrolled burning of rice husks which is a cheaper way of obtaining RHA.

Another study conducted by Dominic et al. (2014) on rice husk silica as an efficient biofiller in high density polyethylene, pretreatment of rice husks with HCl was first done followed by calcination in a furnace at 650℃ for 6h to get silica. HDPE was then allowed to melt at 145℃for two minutes and then varying amounts of RHS (0.5-3 wt%) were added and mixed to form the composite. The test specimens were then cut from the cooled blends. SEM micrographs of pure HDPE showed a number of voids. On addition of RHS, the RHS particles occupied the voids uniformly providing more reinforcement. The wavy texture of HDPE-RHS surface showed signs of greater energy absorption. FTIR analysis of the samples was not conducted as well as considering RHA instead of RHS. Ginting et al. (2014) in his study on preparation and characterization of RHA as a filler material into nanoparticles of HDPE thermoplastic composites, the composites were made by mixing HDPE, nanoparticle RHA and PE-g-MA in different percentages of HDPE (100, 95, 93, 91, 89, 87 wt %). A constant amount of compatibilizer (3% wt) was used. A different study was done by Tong et al. (2014) on mechanical and morphological properties of recycled HDPE composite using rice husk filler. The RH fibers were first dried in an oven to reduce the moisture content. The compounding of the fibers and recycled HDPE with the coupling agent (MA-g-HDPE) was done. Five different fiber loadings (0, 10, 20, 30 and 40% wt) were used. For structural characterization of the samples, SEM micrograph image for 0% wt RH fiber showed no clear gaps in between the recycled HDPE particles. This proved that recycled HDPE was well mixed during the extrusion process. For 20% wt RH fibers, SEM image showed no clear gaps between RH fibers and recycled HDPE matrix indicating good interface bonding. For 40% wt RH fiber, the fibers were perfectly attached to the matrix and were evident that the fibers were strongly imbedded in the matrix. FTIR analysis was not carried out. Also, RHA was not considered in preparation of the samples.

Parvinzadeh et al. (2010) studied on Surface Characterization of Polyethylene terephthalate/Silica nanocomposites. In their study, an ATR-FTIR was done on the samples. It was found that there was a broad band at 3200-3400 cm^{-1}, intense bands at 3000-3200 cm^{-1} and bands at 1600-1800 cm^{-1}. These bands were related to intermolecular O-H bonds, CH_2 asymmetric stretching and C=C or C=O stretching vibrations. The –OH stretching vibrational peak became broader at 3200-3400 cm^{-1} band due to hydrogen bonds. This was for the hydrophilic nanosilica incorporated into PET matrix. Other sharp bands at 1400-1600 cm^{-1} and 950-1250 cm^{-1} were assigned to C=C stretching and C-H in plane bending of benzene rings. It was also noted that on addition of hydrophilic nanosilica into the PET matrix, the O-H intermolecular bonds and CH_2 stretching vibrations were intensified. A more intense band was also noted at 10001200 cm^{-1} related to Si-O-C interactions in the PET/hydrophilic nano-silica composites. SEM micrographs indicated that the PET had a smooth and uniform surface. On adding nano-silica particles to the matrix, nano-silica particle-particle and nano-silica particle-PET chain interactions led to formation of agglomerates on the surface of PET composites. This led to increased surface roughness of the composites. HDPE and RHA were not incorporated in this study.

The aim of this study was to composite RHA and HDPE (both from pellets and packaging containers). In this study however, it is sought to obtain rice husks in two ways, that is, through open air burning, which is cheap but mostly results to RHA with amorphous silica, and secondly, through controlled furnace calcination that would prove expensive but results to crystalline silica in the ash when right temperature conditions are chosen. The author sought to compare the properties of the composites made from HDPE that is recycled from old used packaging containers, and pure HDPE pellets obtained from plastic industries in Nairobi, Kenya using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM)

Table 1. Percentage concentration of HDPE and RHA.

RHA (%)	HDPE (%)
0	100
5	95
10	90
25	75
35	65
50	50

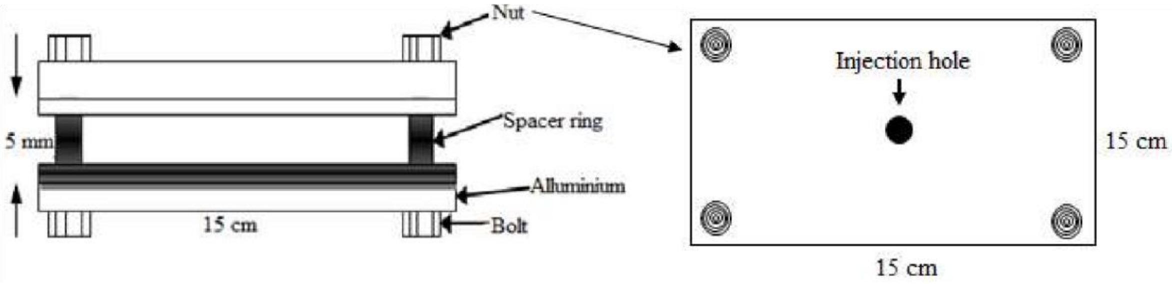

Figure 1. Dimensions of the metallic mold chamber used in this study. Molten RHA-MA-HDPE was injected through
the injection hole.

MATERIALS AND METHODS

Materials

HDPE was obtained in two ways; first as HDPE pellets from industries and also in form of HDPE packaging containers collected from Chuka municipality dumpsites in Tharaka Nithi County, Kenya. Pure HDPE has a melt flow index of 16 g/10 min (190 ℃/2.16 kg) and a melting temperature range of 130-160 ℃ (Dominic et al., 2014). Other chemicals used were maleic anhydride and hydrogen peroxide obtained from sigma aldrich. RHA was obtained open air burning (oRHA) and furnace (fRHA) calcination at 700 ℃.

Methodology

Compatibilization

The compatibilizer (MA-g-HDPE) was made by melt mixing HDPE and maleic anhydride (MA) in the ratio 90:10 with the help of hydrogen peroxide (H_2O_2) initiator (6% wt) in the molten state. It was allowed to solidify and then used in the preparation of RHAHDPE blends. This followed a modified procedure as done by Ayswarya et al. (2012) and Montanheiro et al. (2016).

Preparation of the HDPE-RHA Composite

Samples collected from the dumpsite were heavily washed and allowed to dry to avoid contamination of the samples; it was cut into small pieces then melted as follows. HDPE was allowed to melt in an oven at a temperature of 150°c for 20 min then varying amounts of RHA were added as indicated in Table 1 and a constant amount of compatibilizer (consisting of 15 % of the weight of the total blend) was blended with HDPE. The mixture was mechanically stirred to ensure a homogenous mixture. Exact masses of RHA and HDPE were measured using an analytical balance. The molten mixture was then allowed to cool to room temperature after being injected into the designed metallic mold chamber, whose design and dimensions are shown in Figure 1. The injection was made easier using a designed metallic syringe designed as shown in Figure 2. After cooling, it was then cut into samples to be used for the different characterizations.

Structural characterization

This was determined following the method of Mohamad (2007) using Fourier Transform Infrared Radiation (FTIR). The FTIR spectra for the samples were recorded on an ATR Perkin Elmer A100 spectrometer. The resolution was 4 cm^{-1} and each sample was scanned four times at a range of 4000-380 cm^{-1}. Morphology and particle size determination followed a modified procedure obtained from Ghasemi and Younesi (2011), using Zeiss Ultra plus Scanning Electron Microscopy (SEM) operated at 5 kV. The prepared samples were first sprinkled uniformly over an adhesive tape and spatter coated with a thin layer of gold, and an electron acceleration voltage of 20 kV was applied.

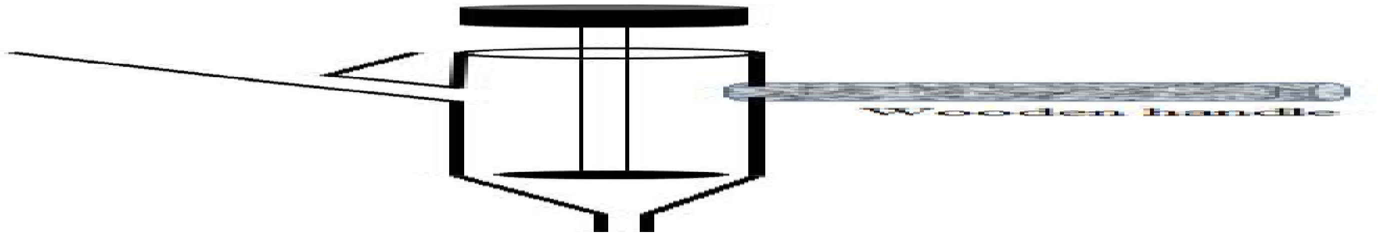

Figure 2. Plunger for injecting molten HDPE-MA-RHA mixture.

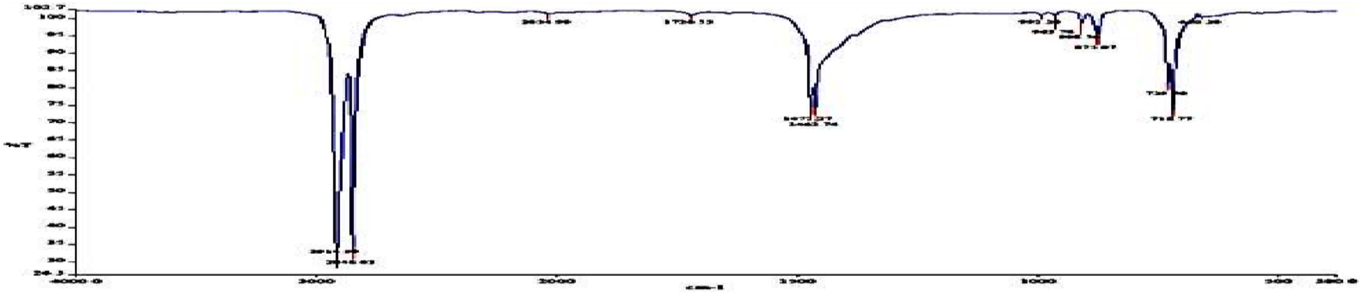

Figure 3. FTIR Spectra of pure HDPE pellets.

RESULTS AND DISCUSSION

FTIR Spectra of pure HDPE and recycled HDPE

The FTIR spectra of pure HDPE and recycled HDPE are shown in Figures 3 and 4, done for wavenumbers between 4000 cm^{-1} and 380 cm^{-1}. The peaks for pure and recycled HDPE were similar. There are two similar prominent sharp peaks for the two types of HDPE between 2914 cm^{-1} and 2849 cm^{-1} showing that they have the highest transmittance of IR at this region. The other peaks are also in close range to each other as seen in the spectra for both types of HDPE (Lin et al., 2015). During manufacture of plastic products there are many additives added, mostly dependent on the polyolefin type and end use of the product. Luckily as outlined in a study by Hahladakis et al. (2018), stabilizers, antioxidants and UV stabilizers additives are used in lower quantities or none in polyethylene products (HDPE and LDPE). It gives assurance that the HDPE obtained from recycling doesn't differ much from pure HDPE. This was similar results to the study by Hadi et al., 2014. It is now expected, similar to a study by Adhikary et al. (2008), that the properties of the composites based on recycled HDPE will be equivalent to those based on pure HDPE.

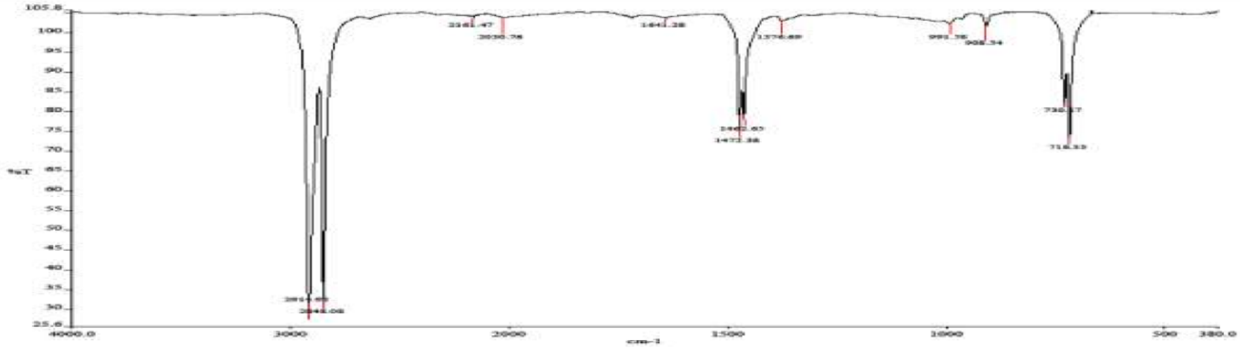

Figure 4. FTIR Spectra of recycled HDPE.

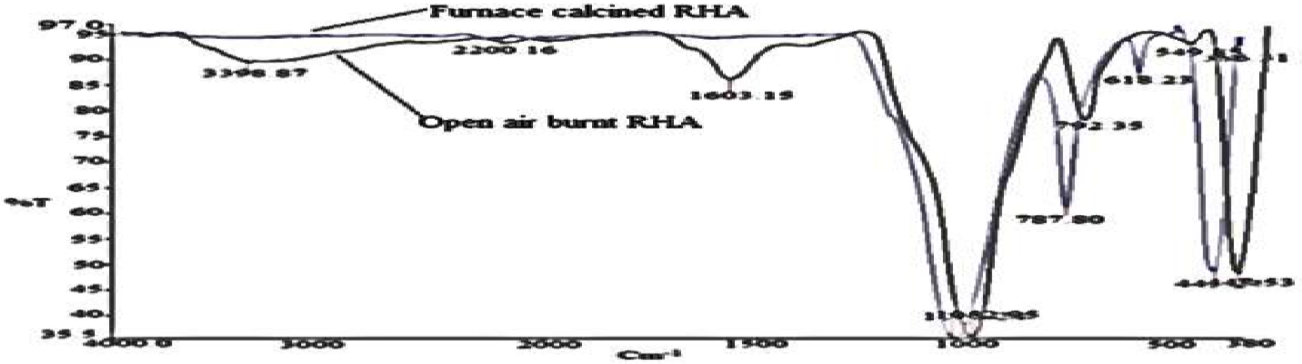

Figure 5. FTIR Spectra of oRHA and fRHA.

FTIR spectra of open air burnt and furnace calcined RHA

FTIR spectra of oRHA and fRHA are shown in Figure 5.

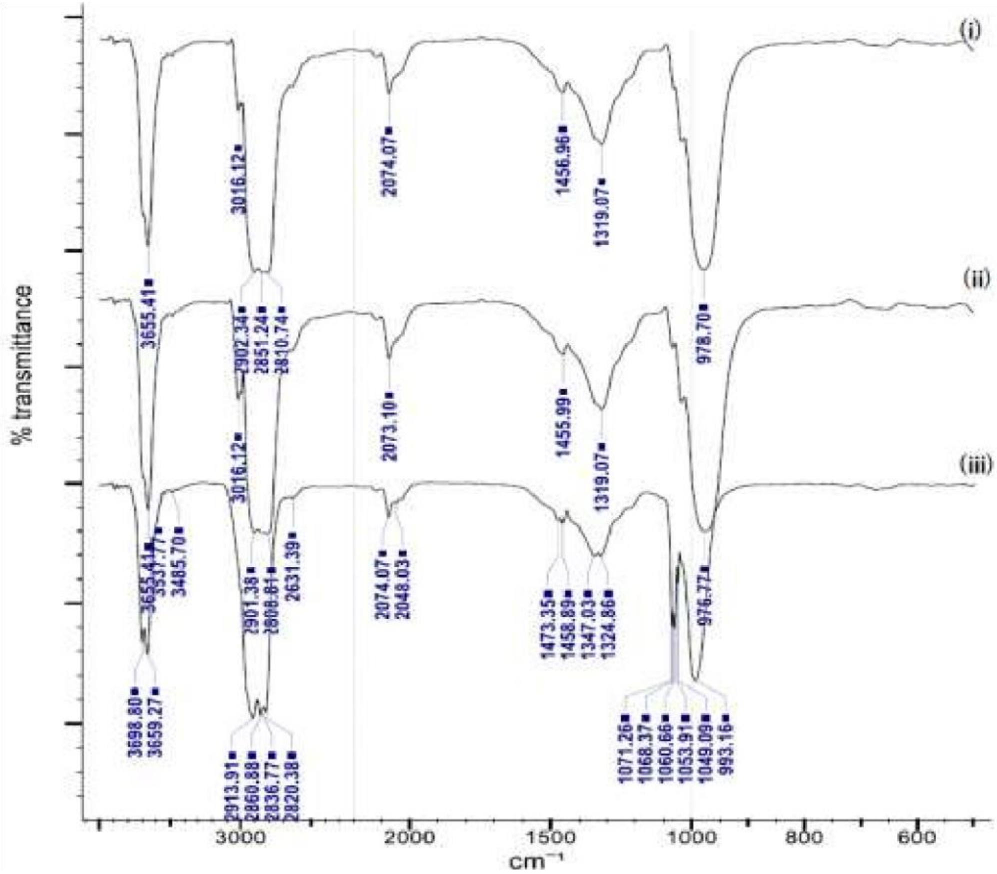

Figure 6. FTRI spectra for fRHA-MA-HDPE (pure) composites (i) 5 % fRHA (ii) 10% fRHA and (ii) 35 % fRHA.

The broad band evident in oRHA peaking at 3398.87 cm^{-1} represents the silinol OH groups and surface adsorbed water (Ren et al., 2013; Zhang et al., 2013), and it is absent in fRHA. This was shown in other studies (Deshmukh et al., 2012). Another unique and differentiating band in oRHA is the band peaking at 1603.15, representing C=C stretching of aromatic carbon (Chuai et al., 2001; Khalil et al., 2001; Prachayawarakorn and Yaembunying, 2005). The peaks between 1200 cm^{-1} and 700 cm^{-1} represents the vibration modes of the Si-OSi network (Della et al., 2002). The peaks at 1051.24 cm^{-1} for fRHA and 1052.95 cm^{-1} for oRHA show the silixone groups Si-O-Si (Pongdong et al., 2015). The band at 443.24 cm^{-1} and shifted to 447.53 cm^{-1} is associated to Si-O bending mode (Della et al., 2002). These peaks show the presence of SiO_2 with a slight peak shifting that matches earlier studies. This indicates the presence of Nano structured SiO_2 in the RHA, as expected.

FTIR spectra of the RHA-MA-HDPE composites

Figure 6 to 9 shows the spectra of RHA and compatibilized HDPE-RHA composites for different proportions of RHA and the two different RHA preparation methods. The FTIR spectra broadly shows peaks around the following larger bands, 995.08-1068.37 , ,

1324.86-1473.35 2048.03-2074.07, 2821.35-2914.88 and 3659-3698.80 cm^{-1}. There is a great similarity in the peaks was noted for samples with 5, 10 and 35% fRHA. Additional peaks exist at 875-450 cm^{-1} for the RHA-MAHDPE made composited of 50% oRHA is evident. These are the peaks present in the RHA before compositing. This could mean that most of the RHA at this percentage mix ratio has not blended well in the HDPE matrix. It could have agglomerated into pockets within the HDPE without forming a uniform blend. However, other peaks have slightly shifted for the RHA-HDPE samples compared to those of pure ash and pure HDPE indicate changes in structure. This is due to formation of new bonds between the –OH and Si-O-Si groups in the RHA and –CH group in the HDPE.

The peaks between, 3655.41-3708.44 cm^{-1} in the sample with 5, 10 and 35% RHA (both oRHA and fRHA), show the presence of O-H group in the RHA arising from silanol functional groups. There is a broad band at 3599.19 cm^{-1} for 25% fRHA-recycled HDPE, 3567.78 cm^{-} 1 -1 for 25% open-air RHA-pure HDPE, 3482.80 cm for

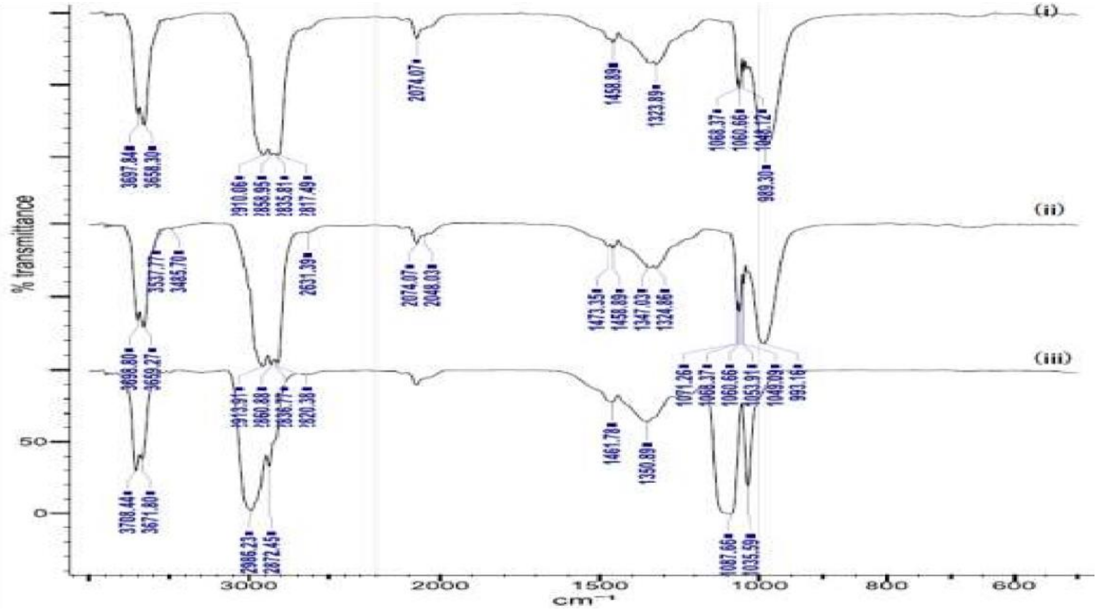

Figure 7. FTRI spectra for oRHA-MA-HDPE (pure) composites (i) 5 % oRHA (ii) 10% oRHA and (ii)
35 % oRHA.

50% fRHA-recycled HDPE, 3436.34 cm^{-1} for 50% openair RHA-recycled HDPE and 3418.08 cm^{-1} for 50% openair RHA-pure HDPE. These bands are related to intermolecular O-H bonds. The sharp peaks at 2848.03 cm^{-1} and 2914.99 cm^{-1}(100% recycled HDPE) which shift slightly to 2848.08 and 2914.92 cm^{-1} (100% pure HDPE) appear as one broad band between 2808.81-2914.88 cm1 . This is for all 5, 10 and 35% RHA except for the 35% open-air RHA-pure HDPE where it appears between 2872.45-2986.23 cm^{-1}. These intense broad peaks are associated with CH_2 asymmetric stretching. The broad peak is also associated with –OH group and chemically absorbed water in the RHA. The prominent peak at 2074.07 cm^{-1} which shifts to 2073.10, 2034.59, 2019.92 and 2028.35 cm^{-1} (5, 10 and 35% RHA spectra) is related to C=C or C=O stretching vibrations. Other smaller peaks were noted between 1500-1800 cm^{-1} for 25% fRHA-pure and recycled HDPE, 50% fRHA-recycled HDPE, 50% oRHA-recycled HDPE and 50% oRHA-pure HDPE.

These were also associated with C=C or C=O stretching vibrations. Between, 1400-1500 cm^{-1} there appeared sharp peaks which were much longer for 0, 25 and 50% RHA. A broad band with slightly sharp peaks at the end appeared between, 1300-1350 cm^{-1}. These are associated with C-H in plane bending. The broad band between 970-1090 cm^{-1} for all RHA-HDPE samples is associated with Si-O-Si stretching modes. Other smaller sharp peaks appeared between 750-910 cm^{-1} and much sharper and longer peaks between 700-730 cm^{-1} for the 25% and 50% RHA samples. These are related to Si-O-C interactions in the RHA-HDPE blends. For these samples, a broad peak is also noted between, 440-470 cm^{-1}. It is highly contributed by the RHA and is associated with O-Si-O bending vibration in the ash samples.

SEM analysis

Figure 10 shows the SEM micrographs of HDPE both pure and recycled at a higher magnification of 20 kx. There is evidence of 'smooth' homogeneous matrix in both, but with voids that could easily be filled by filler materials. Figure 11 shows SEM micrographs for 25%

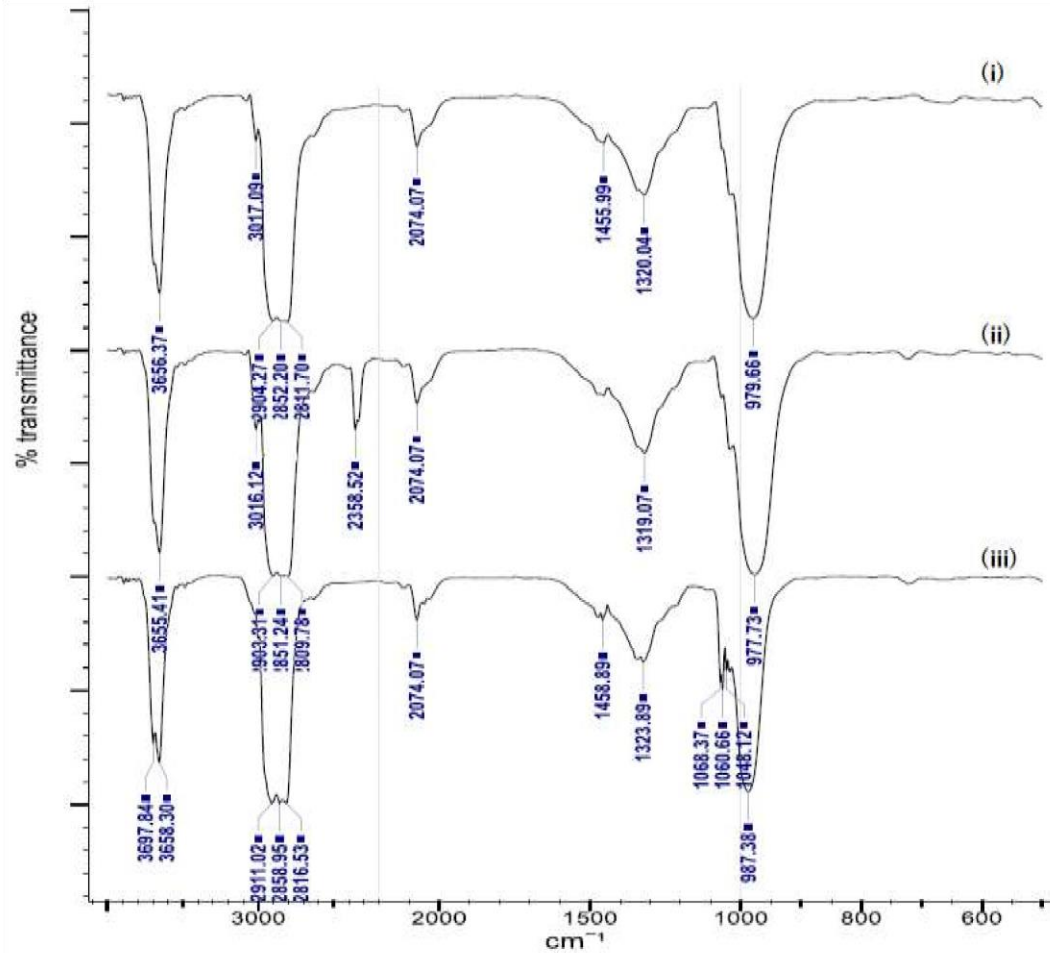

Figure 8. FTRI spectra for fRHA-MA-HDPE (recycled) composites (i) 5 % fRHA (ii) 10% fRHA and (ii) 35 % fRHA.

magnifications. There is evidence of agglomeration of RHA on the surface of HDPE composites. Lower magnification spectra for these samples show distinction between RHA particles and HDPE particles. RHA particles of irregular shapes and non-uniform distribution are also observed. Voids are also seen on the surface indicating its roughness. The surfaces also tend to appear smooth as the magnification increases. There is a homogenous dispersion of RHA particles in the matrix showing uniform composition of RHA-MA-g-HDPE. Due to its low percentage in these composites, RHA could not fill all the voids in the HDPE. Also, at this magnification there appear no clear gaps between the RHA and HDPE matrix indicating good interface bonding.

Figure 12 shows micrographs for 50% RHA-HDPE composites (magnification 20000×). The micrograph shows thermal eradication of the organic matter in the rice husk ash by forming residual pores within the ash sample. This is an ultimate indicator of porous active silica with large internal surface area. The color of the silica is grey, with loose flakes showing plate-like morphology (Farooque et al., 2009).

Conclusion

FTIR analysis for the RHA-HDPE samples showed that there were structural changes due to addition of RHA into the HDPE. It was also observed that the sharp peaks in the HDPE converted to broad peaks in these samples. All the 5% RHA- HDPE blends had a common transmittance at 2074.07 cm^{-1} which shifted to 2073.10 cm^{-1} for the 10% fRHA-pure HDPE. The 25% RHA-HDPE spectra were similar to those of HDPE with very slight shifts in peaks. For the 50% RHA-HDPE blends, the spectra appeared as a combination of both the RHA spectra and HDPE spectra. This meant that they contributed equally to the structure formation. In terms of functional groups, sharp peaks appeared between 3655.41-3708.44 cm^{-1} for the 5, 10 and 35% RHA samples showing the presence of O-H group in the RHA arising from silanol molecules. Broad bands are seen to appear in some of the 25% RHA and 50% RHA blends. These are related to intermolecular O- are associated to CH2 asymmetric stretching, -OH group H bonds. Some of the sharp peaks in HDPE appear as a and chemically absorbed water in the ash. broad band in the 5, 10 and 35% RHA samples. These Sharp peaks between, 1400-1500 cm-1 were observed for the 0, 25 and 50% RHA samples. These are related to C-H in plane bending of benzene rings. SEM analysis for the RHA-HDPE samples indicated that the surface for the 100% HDPE initially appeared smooth and uniform with a wavy texture. On addition of different percentages of RHA, SEM indicated that the surface texture for the samples roughens as the RHA percentage increases. RHA fills the voids in the HDPE making it to appear rough. Also, different magnifications show different surface textures for the same sample. Therefore, production of the oRHA is cheaper and has almost similar or better properties as compared to industrial made which is expensive and much involving. The production of oRHA through this method will also help control the pollution caused by RHA ash as sometimes the ash lacks deposition sites ending up becoming a menace in the society. The reinforcement of the HDPE with the oRHA is predicted to improve the mechanical properties of the HDPE. This study recommends the use of more equipment in the structural analysis of the composites. Also, specific and different temperatures can be considered especially for the fRHA. Other percentages of RHA can also be used to make more samples for more detailed analysis. Further studies on the mechanical properties are encouraged.

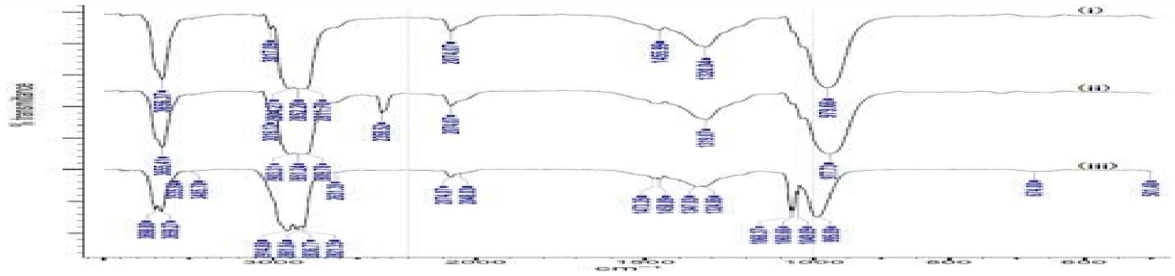

Figure 9. FTRI spectra for oRHA-MA-HDPE (recycled) composites (i) 5 % oRHA (ii) 10% oRHA and (ii) 35 % oRHA.

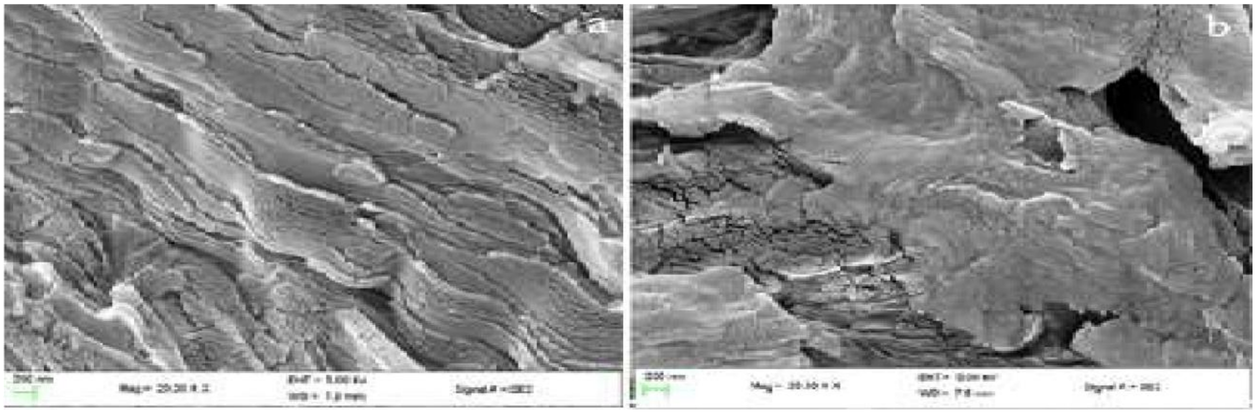

Figure 10. SEM micrographs for HDPE for a. Pure b. Recycled.

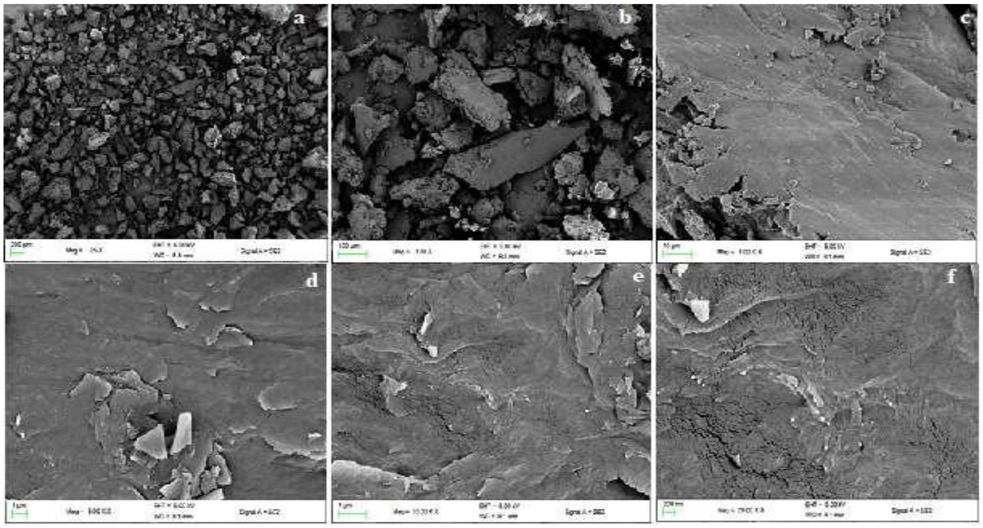

Figure 11. SEM micrographs for 25% RHA-MA-g-HDPE a. magnification at 25 X b. magnification at100 X c. magnification at 1 kX d. magnification at 5 kX e. magnification at 10 kX f. magnification at 20 kX.

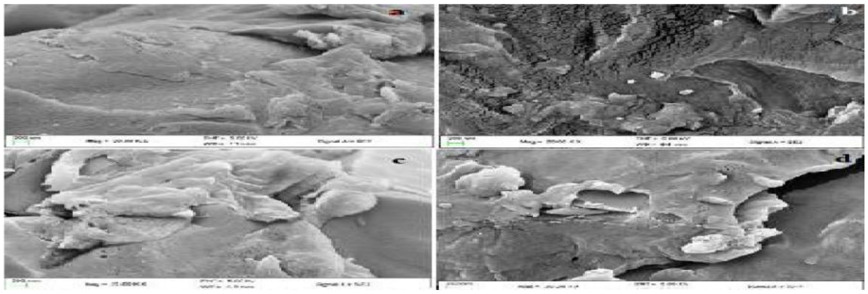

Figure 12. SEM micrographs for 50% RHA-HDPE samples (magnification 20000×) for oRHA with a. pure HDPE, b. recycled HDPE and for fRHA c. Pure HDPE and d. recycled HDPE.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests

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