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Mechanical, Crystallographic, and Microstructural Analysis of Polymer Composites Developed from Iron Filings and Polystyrene Wastes

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KEYWORDS

Polymer composite;
Polystyrene;
Iron filings;
Wastes;
Properties.

ABSTRACT

The development of polystyrene composites, by solvent casting method, with iron filling waste as fillers is considered to improve the mechanical, crystallographic, and microstructural properties for definite uses. Tensile tests were conducted based on the ASTM D638-10 standard. X-ray Diffraction (XRD) analysis and microstructural analysis were also conducted. The Young's modulus increased (from 335.2 N/mm² to 1131.3 N/mm²) with increasing filler concentration (0 - 15 wt%) and vice versa occurred for elongation at break (from 4.9 mm to 1.6 mm). XRD revealed that there is a good structural interaction between the iron filings particles and the polystyrene based resin (PBR) matrix. The composites combine the amorphous and crystalline natures of polystyrene and the iron filings respectively. There was also no chemical reaction observed, but the development of synergistic structural reinforcement in the polystyrene matrix. Microstructural analysis revealed that a good dispersion and distribution of iron filings particles in the polystyrene matrix. The composite with 15% filler had the best interfacial adhesion and the proper mix of the particles-matrix system.

1. Introduction

In contemporary times, most of the metallic components of engineering systems and assemblies are being replaced by metallic particle reinforced polymer composites because of their unique array of properties such as high strength, hardness, corrosion resistance, aesthetics, and high temperature performance [1,2]. This invariably highlights how different materials can work in synergy. The use of micro-metal particles as filler in polymer composites is tantamount to inducing properties like electrical conductivity, magnetic permeability, sound absorption, and improved thermal conductivity in polymer matrices [3,4]. These metal-filled polymers are less costly and of lighter weight than the parent metals. They find applications in electromagnetic interference shields discharging static electricity, heat conduction, electrical heating, and converting mechanical signals to electrical

signals [5]. To make adequate use of metal-filled polymers, the variation of physical properties with the kind and percentage of filler materials must be known [6].

Metals and plastic waste materials are solid wastes of serious environmental problems. Since iron filings and polystyrene wastes are a pervasive category of non-biodegradable solid wastes derived as unused and unwanted materials from several industrial processes, the knowledge of their combined influence in the development of metal-polymer composite is worth finding out. Finding out the useful properties of composites is an indispensable task in engineering material development to foster effective applications [7,8].

Iron filings are predominantly derived from the grinding, filing, or milling operations on iron products; ultimately, they are obtained from the development of iron [9-11]. As a result, they have

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been a waste product of these operations. Conserving natural resources is a pressing matter of sustainable development to ensure sufficient resources for future generations and one major approach to achieving this is the reuse of solid waste as partial replacement in polymer composites thus reducing the demand for extraction of natural raw materials, saving landfill space and producing new products of comparative advantage and usefulness [12].

Extensive studies have been carried out with metal-filled thermosets (like epoxy) [7, 9, 13, 14], but corresponding reports on their thermoplastic (like polystyrene) counterparts are rare [15]. Adedayo and Onitiri [9] considered the reuse of waste material derived from the beneficiation of iron ore by reinforcing epoxy with iron ore tailings to produce Iron ore tailings epoxy composite, in which tensile properties of iron ore tailings filled epoxy composites was found improved with respect to the pure resin. Osman and Mariatti [16] evaluated the mechanical and microstructural properties of aluminium filled epoxy composites.

The reuse of waste polystyrene as PBR has been considered as a greener option of plastic recycling methods [11, 17]. The dissolution procedure is widely reported [12, 18-23]. Recently, polymer composites are being produced via solvent casting methods, with PBR being used as a matrix [24-30]. The usage has been adjudged as compatible and sustainable in plastic composite development. Its usage has been reported in combinations with many types of filler and found flexible in the production of a wide variety of composites [12, 18, 19, 25, 31-34]. Biomass and non biomass fillers like sawdust [21, 31, 35], clay [18], aluminium [11, 18, 24, 25], copper [11, 19], biochars [17, 18, 25, 30], plantain peel powder and fibre [17, 25, 27, 36-38], e.t.c. have all been previously explored as fillers in PBR matrixes.

Studies on iron filling reinforced polystyrene composites are unusual. None is reporting the mechanical, crystallographic, and microstructural properties for composites developed from polystyrene and iron fillings. This research group recently began investigating PBR as a matrix in metal-polymer composites. The use of aluminium [20] and copper [19] have

been investigated in the PBR matrix. Furthermore, The hardness and microstructural properties of iron and aluminium filled polystyrene composites were investigated [24]. As a continuation of this line of investigation, this study is aimed at evaluating the mechanical, crystallographic, and microstructural properties of iron filled polystyrene composites. This study will also help proffer an effective means of utilising solid wastes (polystyrene and iron filings) in lieu of the race for environmental sustainability.

2. Methodology

2.1. Materials

The main materials used were obtained from waste streams; the polystyrene was sorted and cleaned from the University of Ilorin plastic waste collection while the iron filing wastes were obtained from the Faculty of Engineering central workshop, where nodular cast iron of known composition was machined and was dried in an oven for 24 hours at 50°C to remove any free water present. The dried sample was sieved to obtain the powder of 150 µm size. The typical composition of iron filings from nodular cast iron is shown in Table 1 [39].

2.2. Preparation of Composites

Polystyrene Based Resin (PBR) was obtained as described in [Abdulkareem and Adeniyi [12], 35]. The PBR and the iron filing particles were thoroughly mixed into composites and then were pressed to increase with the aid of a single roller on a flat surface to eliminate the air bubbles and possible voids. The pressed composites were left to cure for 3 days. The mixing, pressing, and curing were all achieved at room temperature, as employed in previous studies [11, 18, 20, 32, 33]. At the end of the press cycle, the prepared composites were removed from the press and cut into specimens for characterisation. All these processes go for the compositions as scheduled in Table 2. Iron fillings powder had been used in a percentage below twenty to achieve optimal mixing and avoid agglomerations, as previously employed in related studies [40].

Table 1. Chemical composition of iron filings [39]

Elements	Carbon	Silicon	Magnesium	Sulphur	Phosphorus	Manganese	Iron
Composition (%)	3.53	2.67	0.05	0.01	0.03	0.31	93.4

Table 2. Composition of the developed composites

Composite Samples	Compositions (wt%)
S1	100% PBR matrix + 0% Iron filings
S2	95% PBR matrix + 5% Iron filings
S3	90% PBR matrix + 10% Iron filings
S4	85% PBR matrix + 15% Iron filings

2.3. X-ray diffraction analysis

The phase identification of polystyrene and iron filing, the crystalline state investigation of the PS/Iron filings composites, were both studied using the X-ray diffraction technique. The XRD diffraction patterns were obtained using Bruker D2 diffractometer within 2θ varies from 10 to 90° with a scanning speed of 2° min⁻¹. The studied samples were all films. A cobalt anode at ambient temperature was employed.

2.4. Mechanical and Microstructural Analysis

Universal testing machine (UTM: M500-50 AT) with tensile test fixture and different types of self-aligning grips were used for holding test specimen in a machine. It is fitted with a load cell and extensometer to record the test load and elongation accurately. Tensile tests were conducted according to ASTM D638-10. A computerised universal testing machine model was used to conduct a test at a constant crosshead speed of the order of 4 mm/min. Tensile loads were applied till the failure of the sample and load-elongation curves were obtained for all the composite materials produced. All tests were carried out at room temperature (25± 2°C). Three specimens were used for all the tests and the final results represent the average.

2.5. Scanning Electron Microscopy (Sem) Analysis Of Composite Samples

The Scanning Electron Microscope (SEM) Phenom Prox, manufactured by phenomWorld, Eindhoven, Netherlands was used to carry out the morphology analysis. The sample is placed on the Aluminium holder stub using sticky carbon tape. The sample is insulated using gold and then grounded electrically. The samples each are then labeled on their stub, then dried in the oven at 60°C for 3 hours. The nitrogen line is opened at 50 psi and the vent button is pressed to fill the area with Nitrogen for proper purging of the chamber.

The sample holder stub is then placed in the sample chamber holes and the door shut. At about 35 minutes, a vacuum of 5 x 10⁻⁵ Pa is created. The filament light and the monitor were switched on. At this stage, the accelerator voltage is 15 kV and the filament burned out. The lowest scan mode of 10x is picked and the TV scan is clicked. The magnification is then taken to 1000x at a slow scan and 1500. The image is then saved.

3. Results and Discussion

3.1. Mechanical Analysis

Generally, the tensile strength of composite materials is greatly enhanced by adding metallic fillers to a polymer matrix [11, 41]. This is possible since the metallic fillers have superior stiffness and strength values than polymer matrices [27]. The chemical stability and strength of the plastic matrix relative to the filler is an equally potent factor. A strong iron filings-PBR matrix interface bond is significant for considerable mechanical properties of the developed composite; this is confirmed in Young's modulus of the composites. The Young's modulus of the composite materials increased with increasing weight fraction of iron filing particles in the PBR matrix as shown in Table 3. Details of deviation parameters are included to establish the limit of the experimental errors and deviations. The observed inverse relation, between the Young's Modulus and elongation at break, is made more graphic in Fig. 1. It clearly indicates that the addition of iron filing particles increased the load bearing capacity of the composites. The introduction of iron filing reinforcements increases the tensile strength obtained from composite samples from 335.2 N/mm² to 1131.3 N/mm² at 15% reinforcement. This is 146.24% strength enhancement. The increment in tensile strength is also due to the small particle sizes (150 µm) of the fillers. The small sizes ensure a larger specific surface area of iron filings hence better particle-matrix interfacial adhesion. Proper interfacial bonding between filler and matrix leads to higher Young's modulus of polymer composites [12]. This is also confirmed from previous reinforcement of PBR with metallic particles [11, 18-20, 24, 42].

Table 3. Young's Modulus and elongation at break of the developed composites with deviation characteristics

Iron Filings Content (%)	Youngs Modulus (N/mm ²)	Elong. @ Break (mm)
0	335.72±0.63	4.75±0.02
5	484.41±0.96	3.81±0.05
10	630.15±1.47	1.48±0.03
15	1131.80±1.84	1.43±0.03

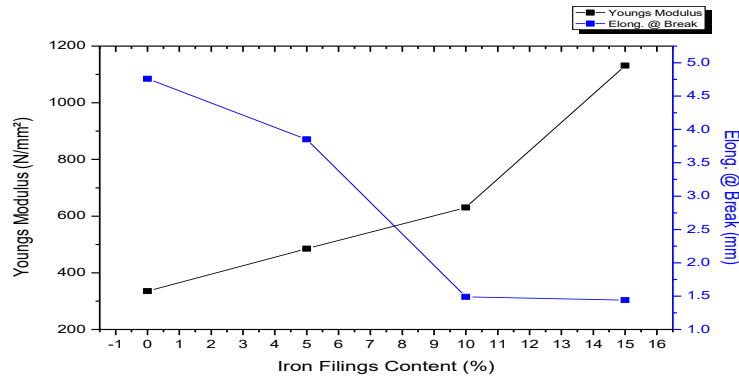


Fig. 1. Effects of iron filing content on Young's modulus and elongation at break

The elongation-at-break is a deformational index obtained from the definite mechanical loading of material [11, 24, 42]. It is usually a pointer to the estimation of the toughness in the plastic composite developed. Consequently, the toughness property of the iron filling reinforced composites developed is explained on the basis of the combined behaviour of the iron filling as a reinforcing element, PBR as a polymer matrix, and the filler/ matrix interface. At varying filler-PBR composition of the polystyrene composite, Fig. 1 demonstrates the effect of varying iron fillings concentration on the elongation-at-break.

The elongations pattern of the iron filling reinforced polystyrene composite in Figure 1 confirms the declining degree of elasticity of that composite. In Fig. 1, elongation-at-break decreases as iron filling concentration increases from 0 to 15%. This is because as the metallic loading increases, the matrix becomes increasingly reinforced, and the degree of stiffness of the composite increases. A similar result was obtained in the earlier study carried out [9, 32]. This study recorded a decrease in elongation as Aluminium powder concentration increases in the polystyrene composite.

The elongation at break of the composite material drops from 4.9 mm at 0% filler to 1.6 mm at 15% filler as shown in Fig. 1. In comparison with the foregoing, the increase in Young's modulus with increasing filler loading is expected, since the addition of iron fillings increases the stiffness of the composites, which in turn decreases the elongation at break as observed in this trend.

3.2. X-ray diffraction Analysis

In plastic composite development, one established route to thermal and electrical properties improvement is to reinforce the chosen matrix with metallic fillers [25]. Moreover, a good indicator of enhanced thermal and electrical properties in any developed plastic composite is its crystallinity. In this study, X-ray diffraction analysis was carried out to recognize

the crystalline contribution of the iron filling in the polystyrene matrix of the polymer composite developed.

The effect of iron filing powder in the PBR matrix is presented in XRD spectra of the composites at 5 wt %, 10 wt %, and 15 wt % of filler loading as presented in Figs. 2, 3, and 4 respectively. There are two sets of observable x-ray diffraction spectrum peaks in each of the figures under reference distinctively for polystyrene and iron filing, confirming the matrix and reinforcement material. The first set is the XRD pattern of neat polystyrene with a broad peak at 16° – 28° and one relatively less intense peak at 10° – 16° , this is common to all the patterns in Figs. 2, 3, and 4 which confirms the amorphous contributions of polystyrene to the composites' structure. Comparable instances were found in other studies [43, 44].

Another pattern in Figs. 2, 3, and 4 gives an idea about the presence of some iron filing powder reflections. These peaks, in the same diffraction pattern, confirm the formation of properly dispersed iron filing powder in the composite and the introduction of crystallinity in the Polystyrene matrix. At 5 wt % (Fig. 3) iron filing powder loading in the composite, a strong and diffuse crystalline within peaks 50° and 54° was observed, this trend is common to loadings at 10 and 15 wt % iron filings (Figs. 3 and 4). All of these are attributed to the crystalline nature of iron filings which was similarly observed in the past contributions [45, 46].

Furthermore, the comparison among X-ray diffraction patterns of iron filing reinforced polystyrene composites of all iron filing loading between 5 % and 15 % (Figs. 2 to 4) confirms the development of crystallinity in the polystyrene matrix. All the three composites' peaks match with peaks of iron filing, indicating the good dispersion of iron filing powder in the matrix crystalline composite. Also noticed is the reduction of polystyrene amorphous character and no alteration is reported in the crystal structure of iron filing. It is also seen that no

reaction takes place between iron filing powder and polystyrene during fabrication of composites but rather the development of synergistic structural reinforcement in the polystyrene matrix is achieved. Their combinations bring more rigidity to the studied samples. This

position is further confirmed by the mechanical results. These results are indications of developed iron filling reinforced polystyrene composites with excellent thermal and electrical properties.

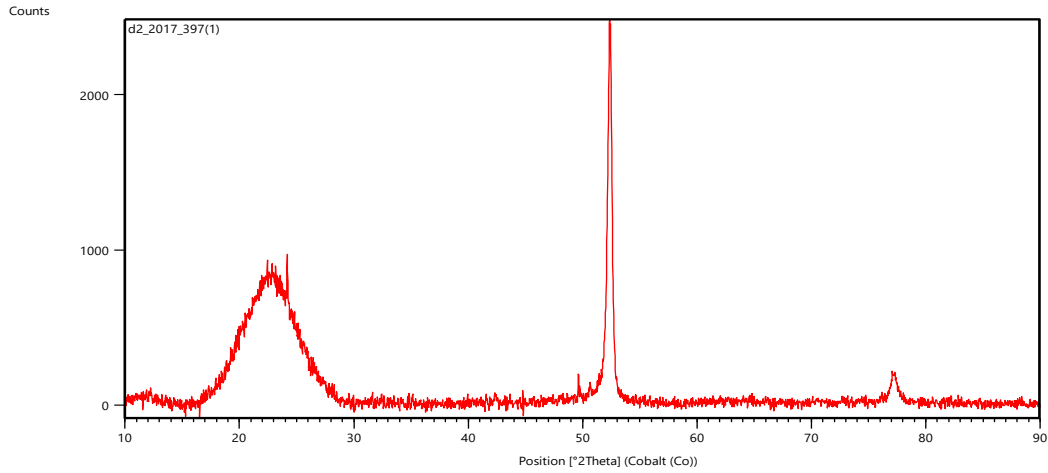


Fig. 2. XRD spectra of the composites with 5 wt % of iron filing loading

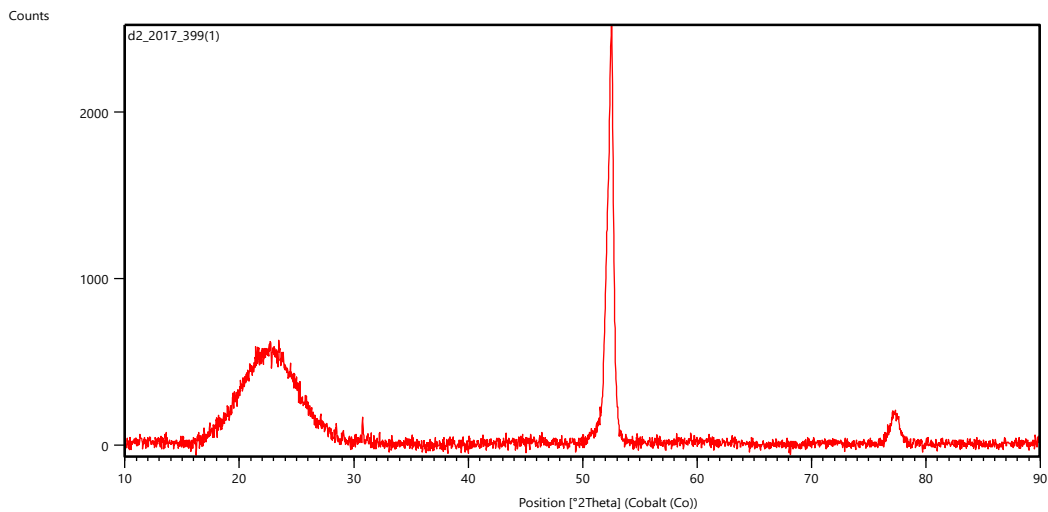


Fig. 3. XRD spectra of the composites with 10 wt % of iron filing loading

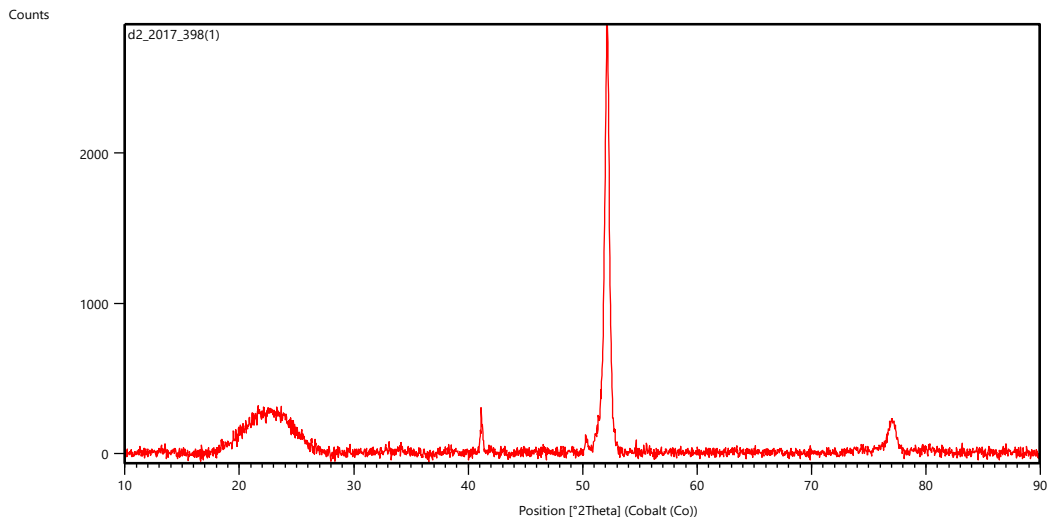


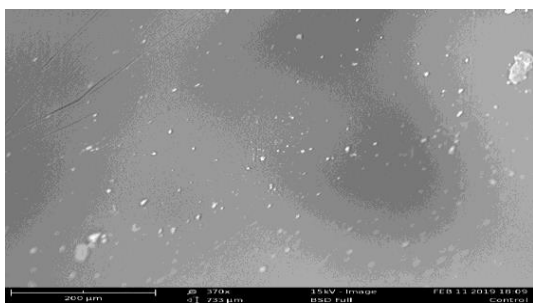
Fig. 4. XRD spectra of the composites with 15 wt % of iron filing loading

3.3. SEM Analysis of Polystyrene-Iron (PS-Fe) composites at various resolutions.

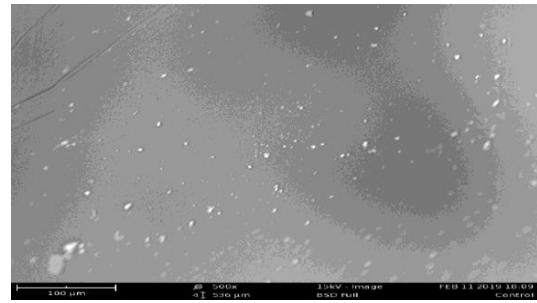
The assessment of the microstructural properties of the developed iron filling reinforced composite materials is indispensable. This is because both the resins of polymer composites and the reinforcing fillings in this regard are hydrophobic. The evaluation of their mixing uniformity and compatibility in resin/filler systems are equally significant and can be deduced from this analysis. Microscopic images of Polystyrene based resin (PBR) polymer prepared through solvolysis were taken at various resolutions to assess the morphology as a control sample before the injection of metallic fillers. As shown in Fig. 5 (a-d) a visibly clear and transparent polymeric product with some fine strata behind randomly scattered white particles on the morphological surface of the sample.

Moreover, microstructural images (Fig. 6) of the iron filled polystyrene composites (PS-Fe) showed a visibly rugged and cracked material surface with white indentations. These cracked surfaces depict low interfacial interaction between the metal particles and the polystyrene resin matrix. This weak cohesion between filler and resin generally weakens the composite and has been shown to negatively impact virtually all mechanical properties of the composite from moderate to low flexural, tensile, and hardness strength.

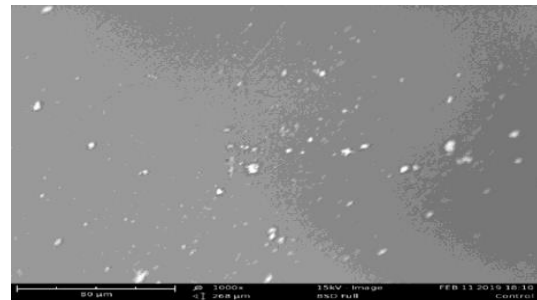
The PS-Fe metallic composite under microstructural analysis revealed poor interfacial interaction between the Iron particles and the polystyrene resin which can result in a relatively moderate to low tensile strength and hardness of the composite. Similarly, Abdulkareem and Adeniyi [19] observed the composite with the highest filler content as having the best mixing. The relationship between the microstructure and the properties of the developed composite agrees with the conclusions of other researchers [3, 4].



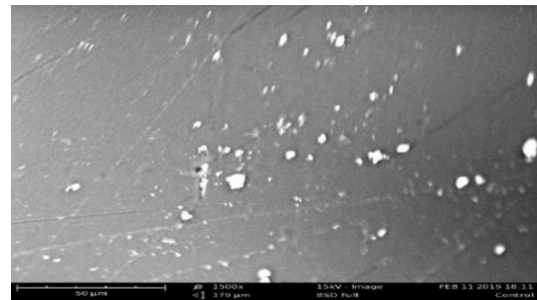
(a)



(b)

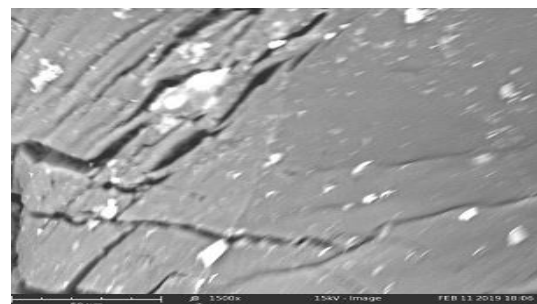


(c)

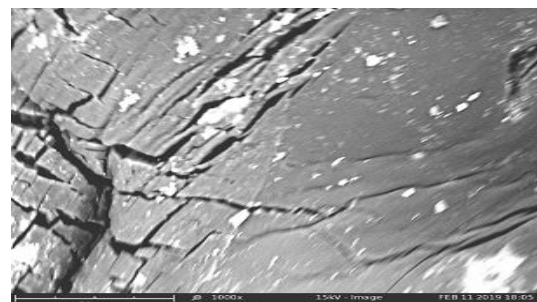


(d)

Fig. 5 (a-d). SEM images showing an overall morphology of the (Polystyrene polymer-PBR) before the injection of fillers (a: X370, b: X500, c: X1000, d: X1500)



(a)



(b)

Fig. 6 (a,b). SEM image of Polystyrene-metal (PS-Fe) composite at high resolutions with 15% filler content at particle size of 100microns

4. Conclusion

In the present work, metal-filled polymer composites were produced from iron filings powder and polystyrene from waste streams using a cold process. The mechanical properties of the composite were found to be a function of the iron filings powder content. There was a significant increment in Young's modulus with an increase in the filler concentration and vice versa for elongation at break. XRD analysis showed that there is good structural interaction between the iron filings particles and the PBR matrix and no chemical reaction was found. Microstructural analysis revealed that the composite with 15% filler had the best interfacial adhesion and the proper mix of the particles-matrix system. This study has successfully elucidated the positive effect of iron filling as fillers in polystyrene composites for multiple applications.

Conflicts of Interest

No potential conflict of interest was reported by the authors.

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