PROPERTY EVALUATION AND ANALYSIS OF DRI SLAG FILLED POLYMER COMPOSITES

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Abstract: The present research focuses on developing a new class of composites consisting of epoxy resin filled with different proportions of Direct Reduced Iron (DRI) slag, commonly known sponge iron slag. These particulate composites are fabricated using conventional solution casting method with incorporation of 0, 10, 20 and 30 wt.% of the slag in the resin. The cured composite samples are subjected to various physical, mechanical and micro-structural characterization tests. Properties such as composite density, void content, tensile strength, flexural strength, micro-hardness etc. are evaluated under controlled laboratory conditions. It is found that while the porosity increases marginally with the increase in the slag content, the mean hardness of the composite improves substantially. A gradual decrement is noticed in case of the strength properties (tensile and flexural) of the composites with the filler content, which may be attributed to improper wetting and stress concentration arising out of the irregular shaped slag particles. The compositional/micro-structural features are identified with the help of electron microscopy, and X-Ray diffraction etc. Scanning electron microscopy reveals the shape, size and distribution pattern of the filler particles in the composites. X-Ray diffraction curve reveals the presence of hard phases such as silica, hematite etc. in the composite. This work suggests that successful fabrication of thermoset polymer composites is possible with addition of an industrial waste like sponge iron slag and that most of the mechanical properties improve with increase in the content of slag in the composite.

Keywords: DRI slag; epoxy; particulate composites; characterization

1. INTRODUCTION

Particulate filled composites are gaining popularity for the past few decades among the different polymer based composites which includes fibre reinforced, particle reinforced and laminated composites. Fillers are materials which are used to reduce cost, minimize the organic contents and to enhance the properties of the composites (1). There are different types of fillers which can be used as a reinforcement in polymer composites which includes natural, conventional and industrial fillers. However, the world is facing serious threats of environmental pollution and the disposal of the industrial waste is a big problem for any existing iron and steel industry. In this regard, polymer composites reinforced with various industrial wastes such as red mud, LD slag, copper slag, dolomite, waste marble dust, blast furnace slag have received attention of various manufacturing sectors such as medical, industrial packaging, aerospace and automobile sectors (2–10).
Generation and disposal of solid waste is posing a serious threat and is a major challenge all over the world. Direct reduced iron (DRI) slag is such a solid waste which is generated during the production of sponge iron in sponge iron industries. It is a by-product of the conversion process of production of sponge iron from iron ore and accounts for 55% of the total waste generated (11). These slags are dumped into various sites wasting acres of valuable land and causing environmental pollution. Disposal of DRI slag which accounts to a huge amount is a major concern, therefore there is an urgent need to use this slag so that it can be used judiciously in various materials (12). In view of this, previous investigators used DRI slag as a suitable material for the purpose of road construction. Sahoo et al. carried out the characterization study of DRI slag and found that it can be a good aggregate to be used in the construction of roads (13). The different possible usage of these slags are developed for the construction of low volume roads in different parts of our country (14). Apart from these reports no work have yet been reported on the use of these slags in polymer composites
Against this background, the present research work focuses on using this slag as reinforcement in epoxy polymer composites and aims on developing and characterizing a novel DRI slag filled epoxy based composite.

2. MATERIALS AND METHODS

2.1. Materials

Epoxy resin (LY556) with its corresponding hardener (HY951) is used as the matrix material in the present work. Epoxy resin (density 1.12 g/cm^3, viscosity 550 centipoise and glass transition temperature 104 ˚C) is supplied by Kumar traders, Bonfield Lane, Kolkata. Raw direct reduced iron slag is collected from local sponge iron plant in

Sambalpur, Odisha (Shyam Metalics and Energy Limited) and is crushed in a ball mill. The crushed powder are sieved to obtain an average size of 36 µm which is used as filler for the making of composites in the present work.

2.2. Composite fabrication

Four sets of epoxy composites are prepared with different compositions of DRI slag fillers (0, 10, 20 and 30 wt. %) using conventional solution casting method. The epoxy resin (LY556) is first mixed manually with its hardener (HY951) in a ratio of 10:1 as recommended to prepare a mixture. The sieved powder of DRI slag is added on this mixture to prepare a dough before being poured into a wooden mould of dimensions 250mm × 250 mm × 4mm and the cast slabs are cured at room temperature for 24 hours. These slabs are removed from the mould and are cut to required dimensions using a diamond cutter for physical and mechanical tests according to standard specifications. The designations of composites with respects to composition of DRI slag are given in Table 1.

Table 1: Designation of Composites

Sl.no	Designation	Compositions
1	CS-0	Epoxy + DRIS 0 wt.%
2	CS-1	Epoxy + DRIS 10 wt.%
3	CS-2	Epoxy + DRIS 20 wt.%
4	CS-3	Epoxy + DRIS 30 wt.%

3. CHARACTERIZATION

3.1. Physical characterization

3.1.1 Density and void fraction

The density of the DRI slag filled epoxy based composites are measured using Archimedes principle according to ASTM D792 test standard. The densities for 3 samples of each composition is tested and the average is reported as the measured density of the composite. However the theoretical density of the developed composites are measured using rule of mixtures as given by Equation [1](15). The void fraction of these composites are calculated by using Equation [2] (16).

$$\rho_{cth} = \frac{1}{\left(\frac{w_p}{\rho_p}\right)+\left(\frac{w_m}{\rho_m}\right)} \qquad (1)$$

$$\text{Void \%} = \frac{\rho_{cth}-\rho_{ce}}{\rho_{cth}} \times 100 \qquad (2)$$

3.2. Microstructural/compositional characterization

3.2.1 Scanning electron microscopy (SEM)

SEM is used to study the surface morphology of raw direct reduced iron slag. The powdered samples of DRI slag completely dried to remove moisture if any present in it and then a thin film of platinum coating is applied on it before being inserted into the chamber of JEOL JSM-6480L V SEM (Japan). The scanning electron microscopy is equipped with energy dispersive spectroscopy (EDS) for the elemental mapping of different chemical constituents present in DRI slag.

3.2.2 X-ray diffraction (XRD)

To identify the presence of hard phases, planes and mineralogical content in DRI slag and its composites, XRD test is carried out using BRUKER D8 ADVANCE X-ray diffractometer in 2θ range from 5˚ to 90˚ with a scan rate of 10˚ min^{-1} and a step size of 0.02˚.

3.3. Mechanical characterization

3.3.1 Tensile test

The tensile test of DRI slag filled epoxy composites are carried out on INSTRON 5967 with environment chamber according to ASTM D3039 standard (175 mm × 25mm × 4mm) for the evaluation of tensile strength and modulus of the composite samples. The cross-head displacement speed is maintained at 10 mm/min. The tests are performed on three specimens of each composition and the average of three values are reported as the tensile strength.

3.3.2 Flexural test

A three point bending test is carried out on universal testing machine (INSTRON 5967) with environmental chamber according to ASTM D 790 standard with a cross head speed of 10 mm/min and gauge length 48mm. Three replications of each composite samples are performed and the mean value is the flexural strength.

3.3.3 Compressive test

The compressive strength of the samples are evaluated using INSTRON 8862 at room temperature according to ASTM D3410. Cylindrical specimens of 12.5 mm diameter and 25mm length are prepared and are subjected to uniaxial compression with a cross head speed of 2 mm/min.

3.3.4 Impact test

An Izod impact tester is used to carry out low velocity impact tests on the composite samples as per ASTM D256 standard. The impact strength of the composites is indicated by the dial indicator of the impact tester.

3.3.5 Micro-hardness test

The shore D hardness of the composite samples is measured using a durometer DIN 53505 as per ASTM D2240 standard. The needle of the durometer is penetrated on different locations of the same composite samples by pressing the hand level arrangement. The micro-hardness number is indicated on the dial indicator of the durometer and the average of all the reported values is recorded as the hardness of the composite.

4. RESULTS AND DISCUSSION

4.1 Physical characterization

4.1.1 Density and void fraction

Table 2 presents the theoretical and measured densities of the epoxy composites filled with DRI slag along with the volume of void fraction present in them. It is seen that the volume percentage of void fraction increases with the increase in DRI slag fillers and the maximum void fraction is found to be around 5% for the composite with 30 wt.% filler content. This increased value is attributed to the entrapped and undissolved gases present inside the composites. Hence it is concluded that the amount of filler strongly influences the void fraction of the particulate filled composites.

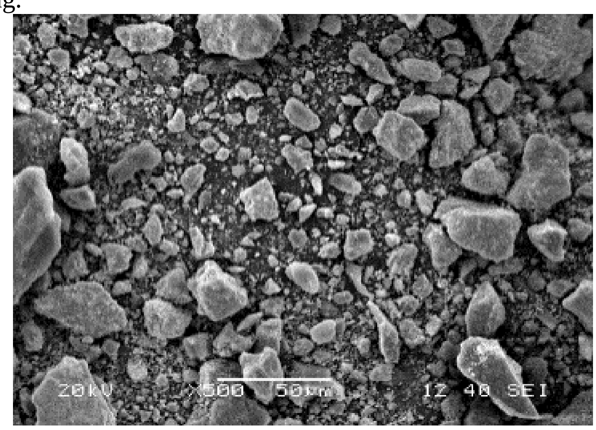
Table 2: Densities and void fractions of the composites with different filler content

Composite	Theoretical density (g/cm^3)	Measured density (g/cm^3)	Volume fraction of voids ((%)
CS-0	1.120	1.098	1.96
CS-1	1.196	1.158	3.17
CS-2	1.284	1.230	4.20
CS-3	1.385	1.315	5.05

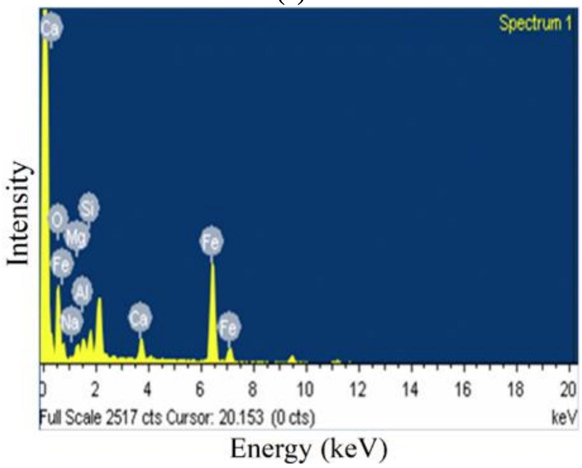
4.2 Microstructural characterization

4.2.1 Scanning electron microscopy

The SEM micrograph of raw DRI slag is displayed in Figure 1(a). The micrograph reveals that the slag particles are highly heterogeneous in nature and show variation of size and shape like oval, irregular and sub rounded shape. The EDS mappings of raw DRI slag is shown in Figure 1(b). The energy dispersive spectroscopy analysis suggests that O, Na, Fe, Al, Si, Ca and Mg are the major elements present in the slag. The mappings further suggested that the minerals present in DRI slag are quartz, albite, magnesia, alumina and hematite. It is estimated from the EDS detector diagrams that the slag contains 36.01 wt.% oxygen in the form of SiO_2, 0.50 wt.% of sodium in the form of $NaAlSi_3O_8$, 3.35 wt.% magnesium in the form of MgO, 2.86 wt.% of aluminum in the form Al_2O_3, 4.96 wt.% of silicon in the form of SiO_2, 4.05 wt.% of calcium in the form of $CaSiO_3$ and 48.28 wt.% iron in the form of Fe_2O_3 with its major constituent as iron since it is an iron slag.



(a)



(b)

Fig.1. (a) SEM image of raw sponge iron slag and (b) EDS mapping of raw sponge iron slag

4.2.2 X-ray diffraction

Figure 2a and 2b presents the XRD curves of raw DRI slag and its composite. The X-ray diffraction curves of the slag reveals the presence of chromate (CrO), hematite (Fe_2O_3), silica (SiO_2), spinel ($MgAl_2O_4$), pyrolusite (Mn_2AlO_4) and andradite ($Ca_3Fe_2(SiO_4)_3$). The analysis confirms the presence of chromium, iron, silicon, aluminium, magnesium, oxygen, calcium etc. Some phases may not be depicted because the quantity may be less. The diffractogram of composite is different from that of the slag as it contains epoxy which is amorphous in nature and therefore a hump is observed at an angle of $2\theta = 21.05^\circ$ which indicates that the slags are dispersed in epoxy resin. The phases present in DRI slag filled composites are hematite (Fe_2O_3), ferrosilicon ($Fe_{2.91}O_4Si_{0.09}$), magnesioferrite ($Mg_{0.64}Fe_{2.36}O_4$) and quartz (SiO_2). The presence of iron, silicon, oxygen, magnesium is confirmed in this analysis.

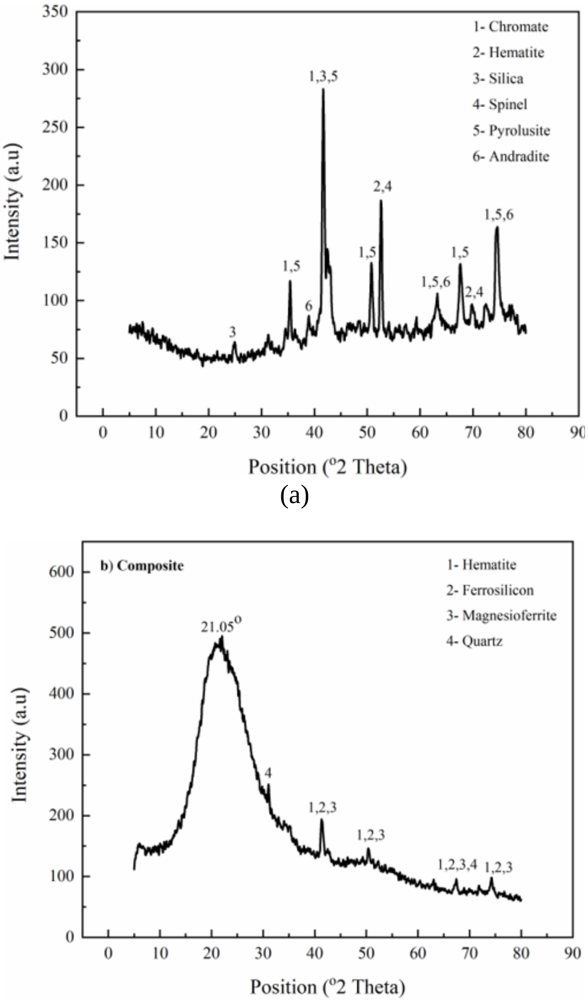


Fig.2. X-ray diffraction curves for (a) DRI slag and (b) DRI slag-epoxy composite

4.3 Mechanical characterization

4.3.1 Tensile strength and tensile modulus

Figure 3 displays the variations of tensile strength and tensile modulus of the composites with respect to different compositions of DRI slag as filler. A gradual decrement is noticed in case of tensile strength of the composites with the filler content as depicted from the figure, which may be attributed to improper wetting and stress concentration arising out of the irregular shaped slag particles. Similar observations have been reported in previous investigations (17–19). Similar trend is observed in case of tensile modulus of the composites which can be attributed to the fact that the slag particles strongly restrain the deformation of epoxy matrix which results in the reduction of strain rate. Moreover the decrease of both the tensile strength and strain of the composites, a synergistic effect leads to small reduction of the tensile modulus.

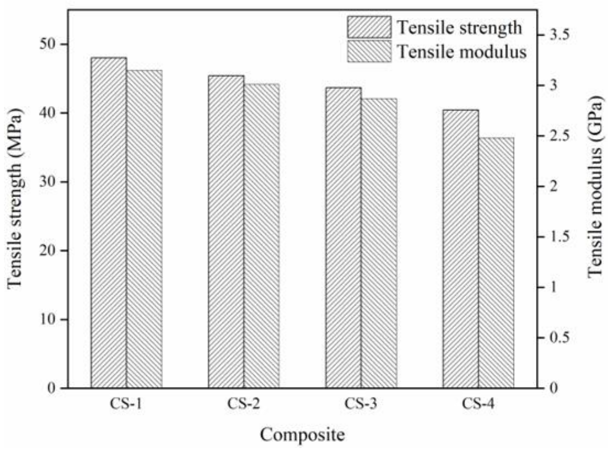


Fig.3. Tensile strength and tensile modulus of the composites

4.3.2 Flexural strength

The variation of flexural strength of slag filled epoxy based composites are presented in Figure 4. The strength gradually decreases with the increase in DRI slag content from 0 to 30 wt.% . as evident from the figure. This decrement is due to presence of voids, poor interfacial bonding and filler dispersion problems. The results indicate that there is a substantial influence of DRI slag fillers on the flexural strength of the composites.

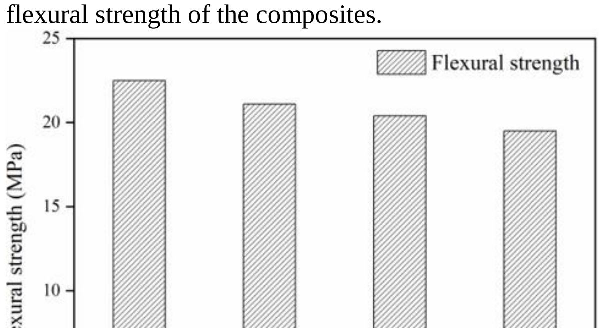


Fig.4. Flexural strength of the composites

4.3.3 Compressive strength

Figure 5 displays the effect of DRI slag content on compressive strength of the composites and the figure reveals that the compressive strength increases with filler content although the increase in strength is marginal. It is very clear from the figure that the compressive strength increases from 90 MPa to 92.67 MPa with the increase in slag content from 0 to 30 wt.%. There is a marginal improvement of about which is attributed to the presence of DRI slag particles. The presence of slag particles in the resin resists the buckling phenomena and hence increasing the compressive strength.

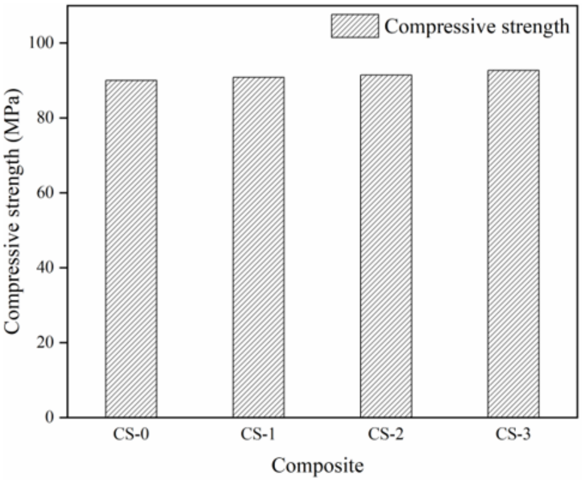


Fig.5. Compressive strength values

4.3.4 Impact strength

The effect of slag content on the impact strength of the composites is shown in Figure 6. The impact strength of the composites increases with the increase in filler content. This is because the hard fillers reinforced in the matrix material absorbs the impact energy and hence resulting in a higher value of impact strength.

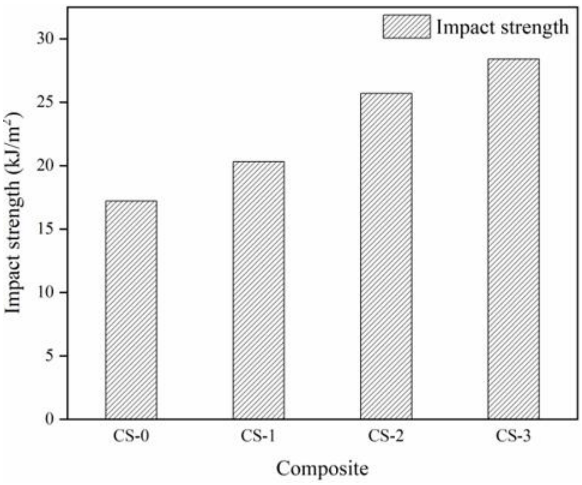


Fig.6. Impact strength values

4.3.5 Composite hardness

The measures values of micro-hardness (Shore D) of DRI slag filled epoxy based composites are presented in Table 3. It is clearly observed that with the increase in filler content in the composites, the hardness increases. The hardness of the composites varies from 80 to 87.92 in Shore D scale, showing an improvement of about 10%. This improvement is due to the presence of hard DRI slag particles and their uniform distribution in the epoxy matrix. Moreover, the distance between two neighboring particles reduces with the increase in filler loading resulting in improved resistance of the composites when subjected to indentation. Some similar observations are recorded in the past investigations.

Table 3: Shore D hardness values of Composites

Composite	Shore D hardness value
CS-0	80
CS-1	83.76
CS-2	85.94
CS-3	87.92

Conclusions

This investigation explores the possibility of developing a new class of composites consisting of epoxy resin filled with different proportions of Direct Reduced Iron (DRI) slag by conventional solution casting method and shows that DRI slag can be reinforced with epoxy as polymer. The microscopic analysis of the slag filled composites reveals the shape, size and distribution pattern of the filler particles in the composites and that the slag particles are irregular shaped and heterogeneous in nature. The compositional analysis confirms the presence of hard phases such as quartz, silica, hematite etc. It is observed that the density and void fraction of these composites varies with the variation in filler content and is found only about 5% which is good as far as the strength of the composite is concerned. A gradual decrement is noticed in case of the strength properties (tensile and flexural) of the composites with the filler content, however the micro-hardness, impact strength and compressive strength of the composites improved substantially.

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