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Thermal Behaviour of Epoxy Composites Filled with Micro-sized LD Slag Particulates

PriyankLohiya[a], Alka Bani Agrawal[a], Alok Agrawal[b*], Abhishek Kumar Jain[c]

[a]*Mechanical Engineering Department, UIT-RGPV, Bhopal-462036, India*
[b]*Mechanical Engineering Department, Sagar Institute of Research & Technology, Bhopal-462041, India*
[c]*Mechanical Engineering Department, MATS University, Raipur-493441, Chhattisgarh, India*

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Abstract

This article reports an investigation of the thermal behaviour of the epoxy/LD-slag composites prepared by hand layup method at varied loading of filler from 0 wt. % to 40 wt. %. The main aim of the work is to establish hazardous industrial waste as a filler material for the development of polymer composites.The thermal conductivity of neat epoxy is 0.15 W/m-K and the same improves to 0.292 W/m-K with the addition of 40 wt. % LD-slag. The addition of LD slag appreciably improves the epoxy matrix's thermal stability by increasing its degradation temperature at different levels along with the enhancement in the char residue. The maximum glass transition temperature of 74.74 ^{o}C is attained for a filler loading of 20 wt. %, whereas, a negligible reduction is observed at higher filler loading. Further, the material's specific heat capacity also progressed with the inclusion of LD slag and reached 1.115 kJ/kg-^{o}C showing an increment of 57.7 over the neat epoxy resin for a combination of epoxy/40 wt. % LD slag composite. The coefficient of thermal expansion gainfully declines with filler loading and decreases by 14.18 % for 40 wt.% of LD slag. The minimum coefficient of thermal expansion value registered is 59.3×10^{-6} /^{o}C.

Keywords: Epoxy, LD slag, Thermal conductivity, Thermogravimetric analysis, Differential scanning calorimetry, Thermal mechanical analys.

1. Introduction

With the rapid growth in the industrial sector all across the world, the amount of waste generated from the sector is also huge. This generated waste has a major problem associated with it, i.e., its disposal as it is very hazardous to the environment [1].Among the total waste generated, around 90 % belongs to the category of industrial waste. Steel-making is one of the largest businesses and the generation of waste through that process is also huge. Management of such waste materials has become very important for protecting the environment. One way to achieve this objective is to reuse/recycle the waste materials for the development of new materials [2]. Blast furnace slag, LD slag and LD sludge are the by-products generated in large quantities from the steel-making industries. Blast furnace slag is obtained during the production of iron in the blast furnace whereas, LD slag and LD sludge are obtained during the process of the LD converter. Increasing attention towards sustainable development and environmental awareness forces researchers to explore ways to either minimize it or reuse it [3]. Recently, these wastes have attracted the scientific community to utilize them as filler material in polymeric composites. Blas furnace slag is being utilized mostly as a filler material in the polymeric resin for the development of polymer composites [4-6]. LD slag and LD sludge are also explored as a single filler and in combination with other reinforcement for the development of a hybrid composite [7, 8]. Among the LD slag and LD sludge, the generation of LD slag is higher. Hence, in the present work, the main focus is on the utilization of the LD slag. In India, the estimated production of this by-product is nearly 160-170 kg per ton of crude steel depending upon the quality of the raw material [9]. Out of this, India utilized only around 25 % and the rest was discarded on neighbouring land of the site. This discarded slag is a prime source of soil, groundwater and air pollution near the dumped area [10]. The fine particles of the slag are easily carried away by the flowing air which may cause health problems to the humans and animals of the locality as it may be inhaled by them. Hence, judicial management of this huge quantity of waste is of prime importance.

Thus, considerable efforts for proper consumption of this waste have been put in as recycling and reuse is the only possible solution to this problem. LD slag has been used in the past in the infrastructural sector. It is gainfully used as raw material for the production of concrete and the construction of roads [9]. LD slag is also been used in railway ballast by combining it with stones. They are used in cement manufacturing as a replacement for clinker. Some works are also reported to utilize this slag for soil conditioners and fertilizers. Despite that, it is observed that the utilization is not up to the mark. Hence, a new avenue had been opened up for proper utilization of the LD slag. On that note, few works are

* Corresponding author e-mail: alokag03@gmail.com.

carried out to study the utilization of this waste material as filler in polymers. Pati and Satapathy [11] were the first to introduce LD slag in a polymeric resin and prepared a composite specimen. They studied the physical, mechanical, and tribological properties of polypropylene/LD slag composites. In their experimental analysis, they reported that the addition of LD slag in the polypropylene matrix appreciably enhances energy absorption during impact and the hardness of the material, whereas, the other mechanical properties under investigation decrease. They also tested the composites for erosion wear. For that, they planned the test as per Taguchi's experimental design. From the analysis, they concluded that the filler loading is the most significant factor that affects the wear. Pati and Satapathy [12] also studied the sliding wear behaviour but this time they used epoxy as the base matrix. The experiments are again designed using the Taguchi design of the experiment method. They conclude that the content of LD slag has a major influence in minimizing the wear rate among all the factors under investigation. Recently, Lohiya et al. [7] studied the physical (density, particle size analysis, water absorption) and mechanical properties (Tensile strength, flexural strength, hardness, compressive strength) and performed sliding wear tests on epoxy/LD slag composites and found similar results as obtained in an earlier investigation. Further, they successfully built a forecast model that is based on an Artificial Neural Network (ANN). They proved that the model successfully predicts the wear behaviour of the composites under investigation over a wide range of filler loading which may either be within the experimental domain or outside of it.

Apart from using LD slag as the only filler, little work is available where LD slag is used as filler material in the hybrid combination of short glass fibre. Pati and Satapathy [13] studied the solid particle erosion wear behaviour of the epoxy-based hybrid composites having constant content of short glass fibre along with varied content of LD slag micro-particulates. They conducted the experimental run according to Taguchi's method and implemented the soft computing technique based on an artificial neural network for the analysis. Their study exposes that the filler loading has maximum implication on the wear rate of the hybrid composites. They further validated the developed ANN model by comparing the measured values with the predicted values. Pati et al. [14] studied erosion wear behaviour with polypropylene as the base matrix reinforced with short glass fibre and micro-sized LD slag. In their work, they implemented a teaching-learning-based optimization technique to choose the appropriate amalgamation of process parameters to achieve an optimized wear rate under erosion. With the help of this optimization technique, they reveal that the least wear is obtained with the finest parametric amalgamation.

From the above discussion, it has been observed that the effect of the inclusion of LD slag in the polymer matrix is similar to that of the inclusion of ceramic fillers into the polymer matrix. The few known ceramic fillers are Aluminium oxide, aluminium nitride, boron nitride, titanium carbide, titanium oxide, silicon carbideetc [15-19]. The performance of such composites is also good, but the only problem with them is that such composites are costly as the filler used is conventional. Hence, the usage of such industrial waste as a filler material in the polymeric resin can be an impactful decision for future development.

It has been observed that the work reported on the polymer/LD slag composites deals mainly with mechanical and tribological behaviour, whereas, the thermal properties of the combination have not been explored in the past by any researcher. In that regard, the present work fills the gap and presents the different thermal properties of the epoxy/LD slag composites for a different combination by weight of epoxy and LD slag. The thermal properties under investigation are thermal conductivity, thermal stability, glass transition temperature, specific heat and coefficient of thermal expansion.

2. Material Used

In the present work, epoxy (Lapox L-12) is selected as a matrix material. The selected matrix material is cured with the tri-ethylene-tetramine (TETA) hardener. The two are combined in a ratio of 10:1 as per the recommendation of the supplier. Atul India Limited, Gujrat is the supplier of it. It is selected as the base material because of its low density (1.189 gm/cm^3), reasonably good mechanical properties and appreciable thermal stability. The filler material used in the present work is LD slag having a density of 3.45 g/cm^3. The same is obtained from the Rourkela steel plant; in Odisha. The raw LD slag collected is converted into the form of particles with the help of a crusher. Later, the particles obtained are sieved to get uniform-size particles. The particles obtained after the sieve are in the range of 1 micron. LD slag mainly comprises oxides of calcium, ferrous and silicon with a small content of oxides of phosphorous and aluminium. The traces of oxides of sodium, silicon, potassium, manganese and magnesium are also present in LD slag [7].

3. Specimen Preparation and Characterization

A simple open moulding technique is implemented for the preparation of the composites. The epoxy required its corresponding hardener for curing. As per the recommendation of the supplier, the ratio used is 10:1 by weight for epoxy and hardener. Later a certain wt. % of LD slag is combined in the blend of epoxy and hardener. The blend of epoxy, hardener and LD slag is then hand-stirred for approximately 10 minutes. After that, the combination is slowly decanted inside the mould keeping in mind the dough reaches every part of the mould to give defect-free composites.

Table 1. Samples prepared in the present investigation

S. No.	Composition
1	Neat Epoxy
2	Epoxy +5 wt. % LD slag
3	Epoxy +10 wt. % LD slag
4	Epoxy +15 wt. % LD slag
5	Epoxy +20 wt. % LD slag
6	Epoxy +25 wt. % LD slag
7	Epoxy +30 wt. % LD slag
8	Epoxy +35 wt. % LD slag
9	Epoxy +40 wt. % LD slag

Curing time is around 6-8 hours but the samples are removed from the mould after 12 hours to ensure proper curing. By following the mentioned steps, nine samples are prepared of which one sample is of neat epoxy and 8 samples are of composites. The different weight fraction samples prepared are shown in Table 1. Along with that, a few pictures of the fabricated samples are also presented in Figure 1.

The thermal conductivity is measured with a thermal conductivity tester Unitherm 2022. The standard followed for measuring thermal conductivity is ASTM E-1530. The coefficient of thermal expansion (CTE) is measured according to ASTM D 696 with the help of a Perkin–Elmer thermal mechanical analyser. The glass transition temperature and specific heat are evaluated through Differential Scanning Calorimetry (DSC) analysis as per ASTM D 3418 standard using DSC-4000 from Perkin-Elmer. The thermal stability is

determined using Thermogravimetric analysis (TGA) analysis as per ASTM E1868 standard using Perkin Elmer thermogravimetric analyser 8000.

4. Results and Discussion

4.1. Scanning Electron Microscopy

SEM micrographs are used to understand the size and shapes of the filler, surface asperities and microstructural voids on the surface of the developed samples [20, 21]. Scanning electron microscopy images of LD slag particles along with the epoxy/ LD slag composites are presented in Figure 2. Figure 2 (a) displays the microscopy picture of LD slag particles. It is clear from the image that the size of particulates under investigation is around one micron with very few particles of a bigger size. So, we can consider the average particle size of the particles as one micron in our study. It is further observed from the image that the shape of the particles is not uniform. Figure 2 (b) presents the cross-section morphology of the composite. It is confirmed from the picture that the distribution of particulates in the epoxy resin is even and the wetting between them is appropriate. There is no gap visible at the interface confirming that the mechanical bonding between the two phases is reasonably good. Good adhesion is established between the epoxy matrix and LD slag particulates because of the rough surface present over the filler material.

Figure 1. Pictorial view of the few fabricated samples

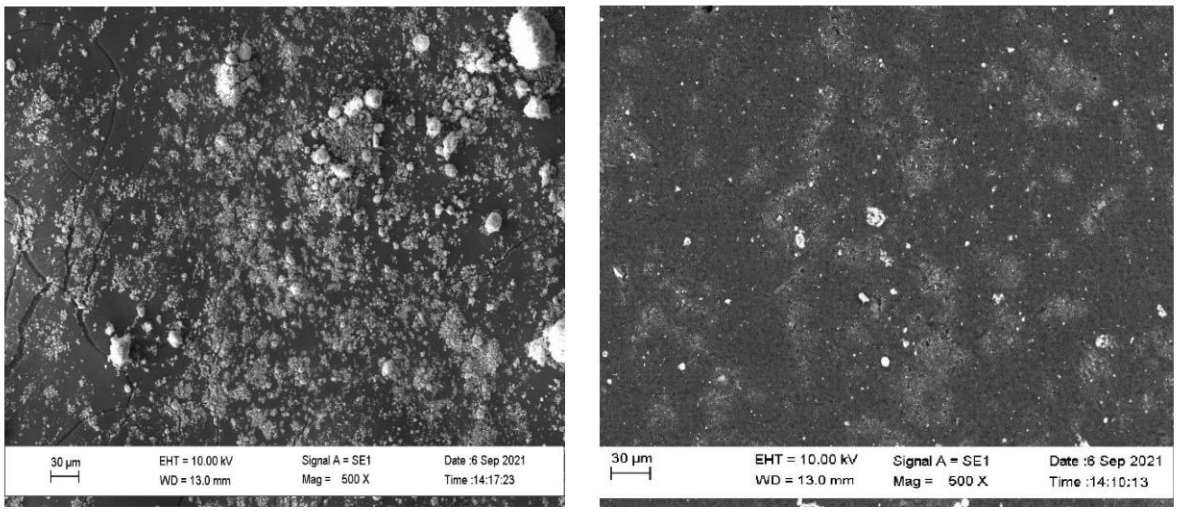

Figure 2. SEM images (a) LD – slag particulates, (b) Epoxy/LD slag composite

4.2. Thermal Conductivity

Thermal conductivity is an important property of the polymer composite material as it governs the other thermal properties of the material as well [22]. The effective thermal conductivity at various levels of filler loading is plotted in Figure 3. It can be observed from the figure that the effective thermal conductivity of the epoxy/ LD slag composites increases as the content of LD slag increases. The intrinsic thermal conductivity of the neat polymer used in the present investigation is 0.15 W/m-K. With the addition of the filler, the effective thermal conductivity of the composites increases. The improvement is because of the relatively high thermal conductivity of the filler against the polymer matrix. When the filler content is limited to 25 wt. %, the enhancement of thermal conductivity is quite low i.e. the value of thermal conductivity improves by only 32 % and attains a value of 0.198 W/m-K. When the filler loading is low, the conductive LD slag particles are randomly distributed and unable to form a conductive chain. Due to this, the enhancement in thermal conductivity value is limited. But as the filler loading increases above 25 wt. %, it is observed that the increasing rate of thermal conductivity improves significantly. For a filler loading of 30 wt. %, the effective thermal conductivity of composites attains a value of 0.276 W/m-K confirming an improvement of 84 % against the neat epoxy. There is a sudden jump from 32 % improvement to 84 % improvement, just with a difference of 5 wt. % filler is a result of the formation of the conductive path within the composite body at 30 wt. % of the LD slag. When the LD slag content increases above 30 wt. %, the rate of increases of thermal conductivity again declined and at the highest filler loading, the composite effective thermal conductivity is 0.292 W/m-K registering an overall improvement of 94.67 % against the unfilled epoxy. This phenomenon may be due to two reasons. First, at 30 wt. % filler loading, the conductive path is already formed and because of that, the thermal conductivity improves [23].

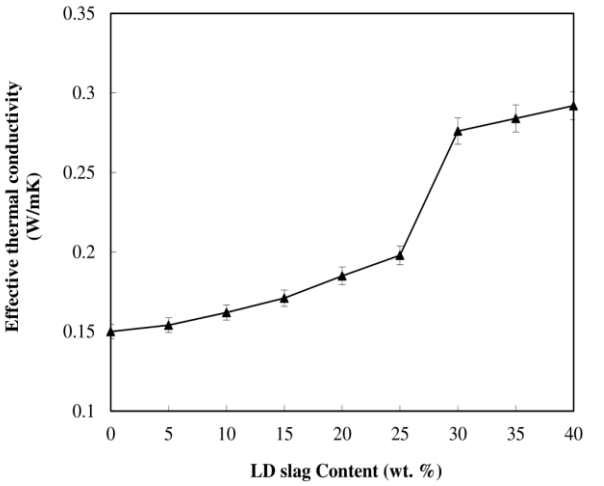

Figure 3. Effective thermal conductivity of epoxy/LD slag composites

With the further addition of the filler, the path remains unaffected and the improvement is pure because of the filler loading. Second, with an increase in filler loading, the void content also increases as observed and explained by Rajput et al. [24] when they used a similar fabrication technique for the preparation of the composite body. The voids have low thermal conductivity value and hence the improvement in thermal conductivity is restricted again at high filler loading.

4.3. Thermogravimetric Analysis

Thermal stability is a significant property of polymeric composites in various practical applications where the material has to face elevated temperatures during its life cycle [25]. The thermal stability of unfilled epoxy along with the epoxy/LD slag composites is present in Figure 4. The TGA analysis is conducted at a heating rate of 10 ^{o}C/minute in the nitrogen atmosphere. The parameters like thermal decomposition temperatures at 5 % of the weight loss (T_{d5}), at 10 % of the weight loss (T_{d10}), at 50 % of the weight loss (T_{d50}), the temperature at the maximum rate of the weight loss (T_{dm}) and char yield are used to study the thermal stability of the polymeric composites [26].From the figure, it is clear that the stability of the neat epoxy as well as its composites negligibly changes till the temperature reaches 200 ^{o}C. This is mainly because of the large binding energy of the epoxy matrix [27]. Also, the curve either of neat epoxy or filled epoxy, nature is similar or the decomposition profiles are similar in a given temperature range used for the measurement. This shows that the incorporation of micro-particulates does not alter the degradation mechanism of the epoxy matrix. It is also seen that with an increase in filler loading, the thermal stability of the composite material improves as a function of filler loading. From Figure 4 (a) it can be seen that the temperature at 5 % weight loss (T_{d5}) is 279.5 ^{o}C for pure epoxy and the same increases to 342.2 ^{o}C for epoxy/40 wt. % LD slag composites. Similarly, Figure4 (b) is marked for the temperature at 10 % weight loss (T_{d10}). For unfilled epoxy, it is 333.1 ^{o}C and the same enhances to 368.2^{o}C for epoxy/40 wt. % LD slag composites. Again, the temperature at 50 % weight loss (T_{d50}) is 394.5 ^{o}C and 492.4 ^{o}C for unfilled epoxy and epoxy/40 wt. % LD slag composites showing a remarkable improvement as shown in Figure 4 (c). However, the temperature at the maximum rate of weight loss (T_{dm}) was not much affected by the inclusion of filler. There is a slight modification in it as shown in Figure 4 (d). For unfilled epoxy, the value is 372.8 ^{o}C and for epoxy/40 wt. % LD slag composite it is 381.8 ^{o}C. The residue of unfilled epoxy is the lowest and is only 1.4 % because of the absence of char, whereas the char yield of filled epoxy is in the range of 4.2 % to 35.4 % as it increases with filler loading.

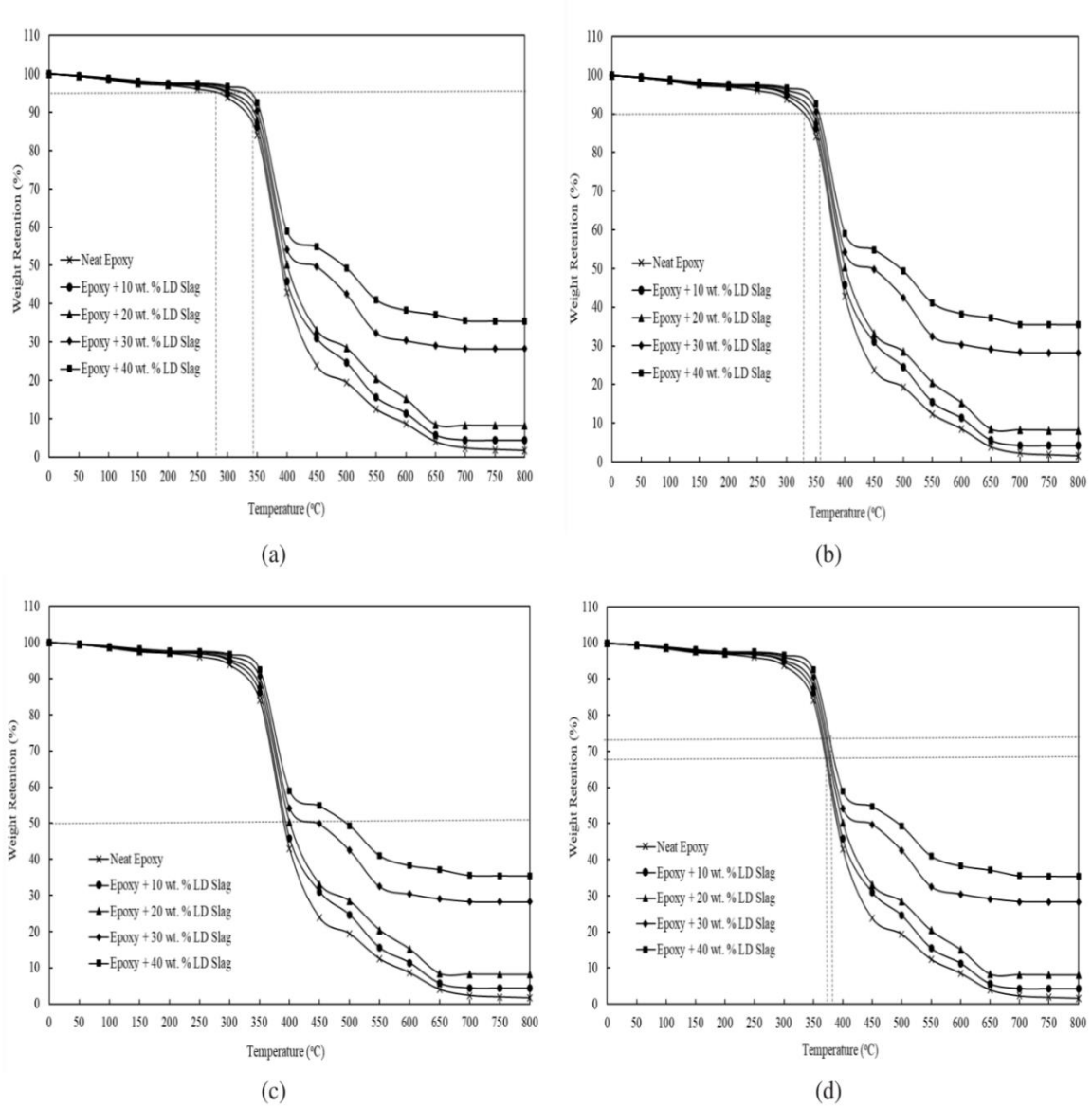

Figure 4. TGA analysis of epoxy/LD slag composites (a) 5 % wt. loss (T_{d5}); (b) 10 % wt. loss (T_{d10}); (c) 50 % wt. loss (T_{d50}); (d) Maximum rate of weight loss (T_{d5})

It is found that the epoxy when added with LD slag, the thermal stability improves and thus the char residue. Char formation is considered to be an important aspect at the time of the fire because higher char residue signifies higher protection to the core of the material. It is also helpful in protecting the structural integrity of the material. The cause for such enhancement is that the LD slag has a relatively higher thermal conductivity than the epoxy matrix and because of that the epoxy filled with LD slag absorbs more heat and the same results in the composite degradation at a higher temperature [28]. Apart from that, the fillers when added to the polymer act as a barrier that reduces the volatile by-product formed during the process of pyrolysis. This restricts the thermal motion of the polymer chain present near the surface of the filler material. This is a result of the physical interlocking provided by the filler to the polymer [27].

4.4. Coefficient of thermal expansion

The CTE of epoxy/LD slag composites is shown in Figure 5. Figure signifies that the inclusion of particulates gainfully modifies the CTE of the epoxy composites as the CTE decreases with filler loading. The low CTE is a desirable characteristic in the present application to avoid thermal fatigue. The measured CTE of unfilled epoxy is 69.1 × 10^{-6} /ºC. As the LD slag is added to it, the CTE reduces.

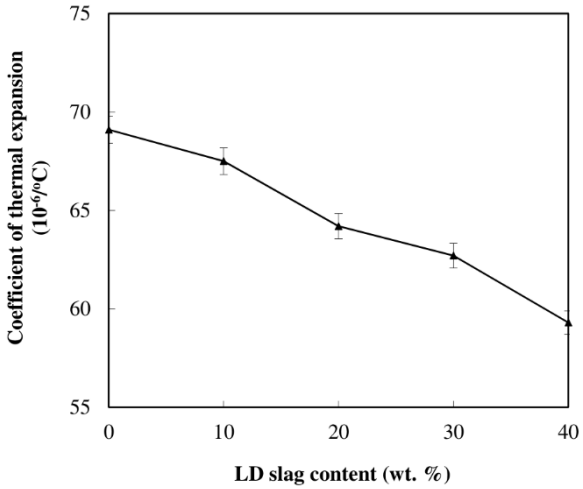

Figure 5. Coefficient of thermal expansion of epoxy/LD slag composites

For a filler loading of 40 wt. %, the CTE appreciably decreases by 14.18 % and attains a new value of 59.3×10^{-6} /ºC. The reduction in CTE is attributed to the fact that the CTE of fillers is very low as compared to the epoxy polymer and because of that inclusion of filler in polymer changes the behaviour of polymer from loosely bounded to tightly bounded. With this, the expansion of the polymer chain is restricted at a given temperature and reduces the CTE of the composites [29]. A relatively high temperature in that case is required to expand the polymer chain. A similar decreasing trend in CTE with filler loading is obtained by Ramdani et al. [30] while performing the thermal analysis of Polybenzoxazine/silicon nitride nanocomposites.

4.5. Differential Scanning Calorimetry

DSC analysis is used to determine the glass transition temperature, melting temperature, crystallization temperature and specific heat of the material. In the present work, the material is an epoxy-based composite and hence the melting and crystallization temperature is not possible to determine, therefore the glass transition temperature and specific heat of the composite at varied filler loading are shown in figure 6. The glass transition temperature of the epoxy matrix improves with the addition of LD slag particulates. The Tg of the unfilled epoxy is 66.45 ºC. This needs to be improved as per the requirement of microelectronic applications. As the LD slag particles are incorporated into the epoxy matrix, the free volume inside the polymer matrix reduces which otherwise exists in it. As free volume within the material reduces, the polymer chain mobility reduces and the glass transition temperature increases [31]. By adding 20 wt. % LD slag, the glass transition temperature increases by 12.5 % and attains a value of 74.74 ºC. It is further observed that when the filler loading increases above 20 wt. %, it does not contribute to further improvement in the glass transition temperature. It is seen that at 40 wt. % LD slag, the glass transition temperature of the composite is 74.11 ºC signifies a slight reduction at higher filler loading. This is because, at higher filler loading, the content of voids increases as well as micro-cracks within the composites grow and because of that further improvement of glass transition temperature gets restricted [32].

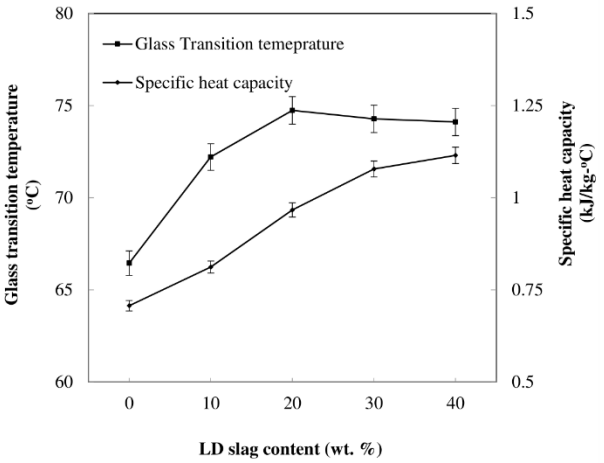

Figure 6. Glass transition temperature and specific heat capacity of epoxy/LD slag composites

A similar increasing trend in the glass transition temperature with filler loading is observed by Pattanaik et al. [33] during their investigation on epoxy-based composite filled with fly ash. Figure 5 also shows that adding LD slag in the epoxy advances the specific heat capacity of the composite body. The specific heat capacity illustrates the material's ability to absorb or release heat as the temperature fluctuates. Absorption of heat occurs for a positive value of specific heat capacity and release if heat takes place when the specific heat capacity has a negative value. In the present work, the specific heat capacity of epoxy improves when fillers are added. That means the LD slag can absorb more heat than the epoxy matrix. The specific heat capacity of unfilled epoxy is 0.707 kJ/kg-ºC and the same increases to 1.115 kJ/kg-ºC registering a huge improvement of 57.7 %. The increase in heat capacity means the material now requires more heat to raise its temperature. With this, the probability of failure of material due to high temperature is reduced as more heat is needed to attain a failure temperature.

5. Conclusion

The main focus of the work is to establish proper utilization of industrial waste i.e. LD slag. In the present study, epoxy-based composites are fabricated with micro-sized LD slag in varied content. The fabricated samples are studied for their thermal properties. The micrographs show that the affinity between the epoxy matrix and the LD slag filler is reasonably good. A noticeable improvement in the various thermal properties of the composites is observed. The maximum thermal conductivity achieved is 0.292 W/m-K, whereas the maximum glass transition temperature reported is 74.11 ºC in the present investigation for the composite prepared with epoxy and 40 wt—% LD slag content. Likewise, the specific heat of the epoxy matrix increases as a function of the loading of LD slag content. The maximum value of specific heat reported is 1.115 kJ/kg-ºC. The coefficient of thermal expansion reduces with filler loading and the minimum CTE of 59.3×10^{-6} /ºC is measured for composite prepared with a maximum filler loading of 40 wt. %. With the enhanced thermal properties, the presently fabricated composites can find potential applications in the field of microelectronic applications like in printed circuit boards, thermal interface material, electronic packaging, etc.

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