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Physical, Mechanical and Thermal Properties of Polyester/Kota Stone Dust Composite

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Abstract: In the current investigation, A waste of the stone industry, i.e. Kota stone dust, is explored as a filler material for the development of a polymer composite with polyester as a base matrix material. The different sets of composites are prepared using the hand lay-up method, where the samples are prepared by varying the content of filler from 5 wt. % to 40 wt. %. The properties investigated are physical, mechanical and thermal for all sets of composites and are compared with the values of the neat polyester to understand the effect of the addition of filler. It is observed from the experimentation that the inclusion of Kota stone dust in the polyester resin increases the density and water uptake rate. Further, the mechanical performance of the composites also increased when the filler loading was judiciously selected. The maximum tensile and flexural strength is obtained for filler content 30 wt. %, whereas compressive strength and hardness are reported to be maximum at 40 wt. %. The highest mechanical properties reported in the investigation are 66.2 MPa (tensile strength), 73.2 MPa (flexural strength), 109.4 MPa (Compressive strength) and 84.3 Shore-D number (Hardness). During the thermal characterization, it was observed that the inclusion of Kota stone dust improves the heat conduction behaviour of the fabricated samples. For a filler loading of 40 wt. %, the maximum improvement in thermal conductivity observed is 67.6 % against that of unfilled polyester. Also, a substantial improvement in the value of the glass transition temperature is reported along with the gainful reduction in the coefficient of thermal expansion. At maximum filler loading, the maximum glass transition reported is 89.4 ^{o}C and the minimum CTE reported is 52.3 × 10^{-6} /^{o}C which is a modification of 37.9 % and 33.1 %, respectively.

Keywords: *Polymer matrix composite, thermal conductivity, glass transition temperature, coefficient of thermal expansion, mechanical properties.*

Introduction

The utilization of natural resources is increasing as there is a continuous growth in the world population. Because of the limited resources, a gap between the demand and supply grows continuously. A reduction of this gap is of prime importance, and various industries are working on it by increasing their production as per the requirement. When the industries work on that, the waste generated from the industries also increases, which is generally hazardous in nature and is not suitable for our environment [1]. The hazard level of the waste depends on the type of industry. In the present discussion, we are concentrating on the waste obtained from the stone industries. The waste generated from the stone industries is solid waste which are originated during the processing of stone from raw stone to finished product. These wastes are generally in the form of particles of varying sizes and are disposed of in the nearby land, which causes problems like land, water and air pollution [2]. In India, Rajasthan is the state which has the largest stone industries, and because of that, the maximum stone waste is generated in that area. If the waste generated from the stone industries is utilised properly, it will help in reducing pollution, which makes the surroundings cleaner and greener [3]. An effective way of utilizing this waste is to use it as a filler material for the development of composite material with polymer as base matrix, which provides multiple benefits like low cost, sustainable and environmentally friendly material [4].

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Marble stone, granite stone and Kota stone industries are major industries present in Rajasthan. Hence, the dust generated from these industries is also large. A lot of work has been reported in the past with this industrial waste as a filler material for the development of composite materials. Khan et al. [5] investigate the recycling of marble waste as reinforcement in LDPE composites via injection moulding. Characterization and testing showed optimal mechanical and thermal improvements at 50% marble content, demonstrating strong potential for large-scale sustainable applications in construction and insulation products. Awad et al. [6]

also worked on a similar combination of matrix and filler material. They identified the optimal conditions for the combination, which shows improvement in different mechanical and thermal properties. They also observed that the inclusion of marble dust reduces the mass loss of the composites due to wear. In their other work, Awad and Abdellatif [7] used a combination of polypropylene and marble dust and studied the mechanical properties at different filler content and size. From the experimental investigation, they found that the inclusion of filler material enhances the mechanical properties of the composites when the content of filler is selected judiciously. Further, they reported that the inclusion of smaller-sized fillers creates more impact on the enhancement of the mechanical properties of the composites. Nayak and Satapathy [8, 9] used marble stone dust with polyester resin and investigated the mechanical and tribological properties as a function of filler loading and filler size. Results show slight reductions in tensile and flexural strength, but improved compressive strength, impact resistance, and hardness. Further, a dry sliding wear test using a pin-on-disc based on Taguchi design is performed. Optimal parameters for minimum wear were identified and reported. During the study of filler size, it was reported that the strength of the material is greater when composites are prepared with smaller filler, whereas hardness is better for their counterpart. Bakshi et al. [10] reported that the inclusion of marble dust in polypropylene increases the density and water-resistant characteristics of the composites. The optimized filler loading for mechanical properties and thermal conductivity is 20 wt. % as reported in their work. Lendvai et al. [11] added marble dust to the polylactic acid and reported significant improvement in tensile and flexural modulus, though slight reductions occurred in strength and ductility. Further, impact strength increases up to 10 wt. % filler before marginally declining at higher loadings. Wear resistance of the composites also improves with filler loading. From the DSC analysis, it was observed that the crystallinity of the polymer decreases with the increase in the content of marble dust. In other work, they reported that inclusion of the marble dust in the recycled polyethene terephthalate improves the hardness of the composites [12]. The thermogravimetric analysis and differential scanning calorimetry show that the inclusion of marble dust in the recycled polyethene terephthalate increases the storage modulus and thermal stability of the polymer [13]. Later, Singh et al. [14] applied a multi-criteria decision-making approach to optimize marble dust–filled PLA and rPET composites. Composites with 0–20 wt% filler were assessed for mechanical, physical, and wear properties. Marble dust improved modulus and hardness but reduced tensile and impact strength at higher contents. Wear resistance improved up to 10 wt. %. MCDM analysis identified 10 wt. % marble dust–filled PLA as the optimal composite with balanced overall performance.

Granite dust is another stone industry waste, which, because of its hard nature, can improve the hardness of the polymer along with its durability. As hardness increases, so does the wear resistance characteristics [15]. Garigipati and Malkapuram [16] used polybenzoxazine matrix with granite dust and reported similar enhancement in the hardness of the polymer with filler loading. Further, during the thermal analysis, they reported that the inclusion of granite dust increases the thermal stability of the composites, along with the limiting oxygen index. Arumugam et al. [17] observed that the hardness, thermal conductivity and dielectric strength increase, whereas tensile and flexural strength decrease when the granite dust is added to the epoxy resin. Granite dust is also explored with polybenzoxazine matrix, where it is reported that thermal stability and glass transition temperature of the polymer increase with the content of granite dust, whereas the electrical conductivity of the composite decreases [18]. Zhang et al. [19] studied the influence of surface modification on the granite dust using an aluminium ester coupling agent. They observed that the modified particle composites show significantly improved tensile, flexural, and impact strengths due to enhanced interfacial adhesion and stress transfer. Thermal conductivity is also increased when modified granite dust is used.

The solid waste from the Kota stone industry has also been utilized recently. Rajput et al. [20] investigated the mechanical properties of epoxy reinforced with micro-sized Kota stone dust. Gupta et al. [21] performed the sliding wear analysis of epoxy/Kota stone dust composites and reported a decrease in specific wear rate in filled composites. Singh et al. [22] also performed the sliding wear analysis of polyester/Kota stoned dust composites and found improvement in the wear resistance characteristics of the composites. Awasthi et al. [23, 24] performed the mechanical, sliding wear and thermal analysis of the Kota stone dust filled polymer composites and reported improvement in the different properties of the composites.

From the past work, it is noticed that a sufficient quantity of work has been performed on the utilization of the marble stone dust and granite stone dust, but a very limited study is available on Kota stone dust. As the composition of Kota stone is comparable to that of marble and granite, it has the potential to be used as a filler material in polymeric resins. Given this, the present work comprises the development of Kota stone dust filled polyester composites and their characterization. The properties investigated are physical, mechanical and thermal. The variation in these properties as a function of filler loading is present in this work.

Materials and method

An unsaturated isophthalic polyester resin is employed as the matrix material, supplied by Carbon Black Composites, Mumbai, India. The curing of the resin system is carried out using methyl ethyl ketone peroxide (MEKP) as the catalyst, while cobalt naphthenate is utilized as an accelerator to enhance the curing reaction. The reinforcing phase consists of Kota stone dust (KSD), which is obtained by drying the slurry generated during Kota stone processing. Polyester/KSD composite specimens are fabricated using a conventional hand lay-up technique. Initially, the required amount of KSD micro-particles is gradually incorporated into the room-temperature curing polyester resin and uniformly mixed by manual stirring. Subsequently, the cobalt accelerator (1 wt.%) is added to the mixture and stirred for approximately two minutes to ensure proper dispersion. This is followed by the addition of MEKP catalyst (1 wt.%), after which the mixture is again manually stirred for an additional two minutes. The prepared mixture is then poured into the mould and allowed to cure at ambient conditions for 4 hours. After completion of the curing process, the composite specimens are carefully demoulded. All characterization and testing are conducted only after allowing a post-curing period of 48 hours. A total of eight composite formulations is produced by varying the KSD filler loading up to a maximum of 40 wt.%.

The density of the prepared samples is experimentally determined using water immersion techniques following ASTM D 792-91 standard. The density is also evaluated theoretically with the rule of the mixture model [17]. The absorption of water by the composite is measured following the ASTM D 570 standard [18]. The tensile tests are performed following the ASTM D638 standard, the compressive test is performed following ASTM D695 and flexural tests are performed following the ASTM D2344-84 standard with the help of the Instron 3382 Universal testing machine. The hardness is measured by following the ASTM D-2240 standard using the PosiTector SHD Shore hardness Durometer. The thermal conductivity is evaluated following the ASTM E-1530 standard. The coefficient of thermal expansion and glass transition temperature are determined by following the ASTM D 618 and ASTM E 831 standards, respectively.

Results and discussion

Physical Properties

Table 1 presents the density of the composites along with the content of voids present inside the composites. The densities presented are both the measured density and the theoretically evaluated density. It is observed from the table that the density of the composite increases as the content of Kota stone dust in the polyester resin increases. This is primarily because of the higher density of Kota stone dust as compared to polyester resin. The density of neat polyester is 1.152 g/cm^3, whereas that of Kota stone dust, it is 2.65 g/cm^3. It is observed that there is a large difference between the density of polyester resin and Kota stoned dust, and because of that, the density increases with filler loading. With the inclusion of 5 wt. % Kota stone dust, the density increases to 1.173 g/cm^{3,} showing a marginal improvement of 1.82 %. The enhancement in density is continuous and reaches a maximum of 1.408 g/cm^3 for a filler loading of 40 wt. % showing an increment of 22.2 % over the density of neat polyester. It is also observed from the table that the theoretical density is higher than the measured density for a given loading of Kota stone dust. The primary reason for that is that during the theoretical calculation, the air trapped during the process of fabrication is not considered.

Table 1: Densities and void content of the fabricated samples

Kota Stone dust Content (wt. %)	Theoretical Density	Experimental Density	Void Content (%)
5	1.186	1.173	1.10
10	1.221	1.202	1.56
15	1.259	1.231	2.22
20	1.299	1.262	2.85
25	1.342	1.298	3.28
30	1.387	1.329	4.18
35	1.436	1.364	5.01
40	1.489	1.408	5.44

Further, with an increase in filler loading, the difference between the measured density and the theoretical density increases. This signifies that the void content increases with filler loading. For a filler loading of 40 wt. %, the maximum void content reported is 5.44 %. The increase in void content with filler loading is primarily because, as filler loading increases, the stirring process becomes more difficult, and as a result, the air content trapped increases, increasing the void content in the composite body.

The water uptake percentage of the polyester/Kota stone dust composites as a function of filler content is presented in Figure 1. The test is conducted for a duration of 7 days, and the reading is taken after that. It can be observed from the figure that the water uptake percentage of the composite increases as the content of Kota stone dust in the resin increases. For unfilled polyester, the water uptake percentage is 1.18 %, which increases to 3.12 % for a filler loading of 40 wt. %. It is observed that for neat polyester, the water uptake rate is very low, which is primarily due to the hydrophobic nature of the polymer. Against that, the stone dusts are relatively hydrophilic in nature. Because of that, the water uptake rate increases with filler loading. Apart from that, it is also observed that the void content increases with filler loading, which is another reason for the increment of the water uptake percentage. With an increase in void content, there is more space available in the composite to accommodate the water molecule and hence the water absorption rate increases.

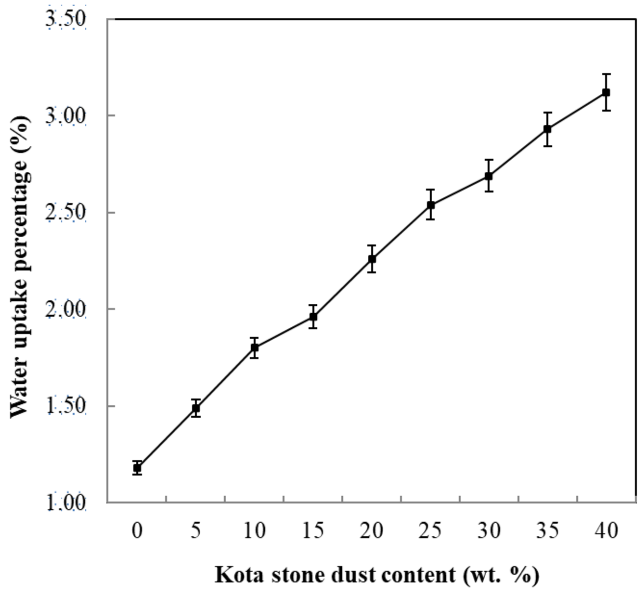

Figure 1: Water uptake percentage of the prepared samples

Mechanical Properties

The mechanical properties investigated in the present work are tensile strength, flexural strength, compressive strength and hardness. Figure 2 shows the variation in the tensile strength of the composites as a function of Kota stone dust. It can be observed from the figure that the inclusion of filler in the polyester resin improves the tensile strength of the composites. The tensile strength of unfilled polyester is 46.5 MPa, which increases to a maximum of 66.2 MPa for a filler loading of 30 wt.%. This is an enhancement of 42.4 % against the value of neat polyester. It is further observed that when the filler loading increases above 30 wt.%, the trend is reversed, i.e. the tensile strength decreases with an increase in the filler loading. For a filler loading of 35 wt. %, the tensile strength decreases to 61.4 MPa, which further reduces to 56.2 MPa for a filler loading of 40 wt. %. The decrement in tensile strength at high loading of filler is due to improper wetting of fillers along with their agglomeration with the matrix body. Also, an increase in void content with filler loading is another reason for the reduction in tensile strength of the composites.

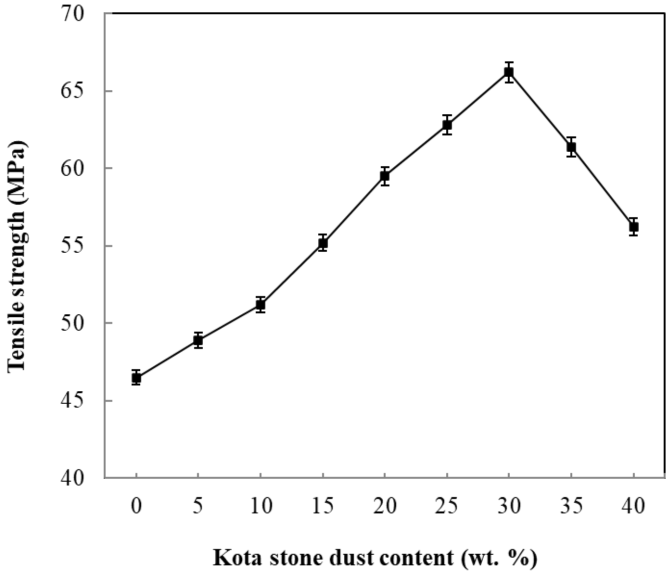

Figure 2: Tensile strength of the prepared samples

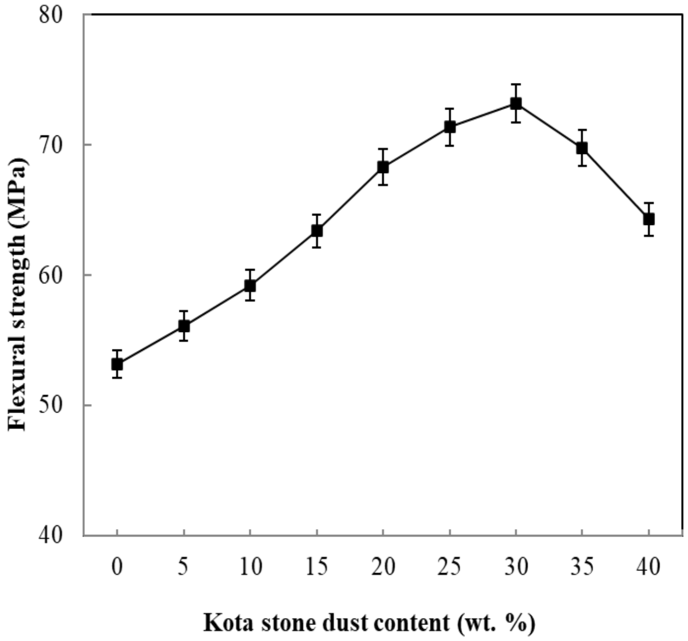

Figure 3: Flexural strength of the prepared samples

Similar to the tensile strength, flexural strength also shows a similar trend, which is presented in Figure 3. The flexural strength for unfilled polyester is 53.2 MPa and reaches 73.21 MPa (37.6 % higher than neat polyester) for a composite prepared with 30 wt. % of the filler material. For a filler loading of 40 wt. %, the flexural strength reduces to 64.3 MPa. This is a decrement of 12.2 % form the peak value. The reason for the decrement is similar to that discussed for tensile strength.

The compressive strength of the prepared samples is presented in Figure 4. It is observed from the figure that the trend obtained for compressive strength is different from that observed for tensile and flexural strength. Here, the compressive strength increases with filler loading till 40 wt. % Kota stone dust content. For an unfilled polyester, the compressive strength is 53.2 MPa, which increases to 64.3 MPa for a filler loading of 40 wt. % registering the highest value at maximum filler loading. This is an improvement of 20.8 % against the value of unfilled polyester. The increment in hardness of the composites with filler loading is primarily because of the high compressive strength of the Kota stone dust compared to the polyester resin. Further, the voids present inside the composite do not affect the compressive strength much, as the direction of load during compressive strength measurement is in the opposite direction from the tensile loading situation, which is helpful in nullifying the gaps generated due to the presence of air voids.

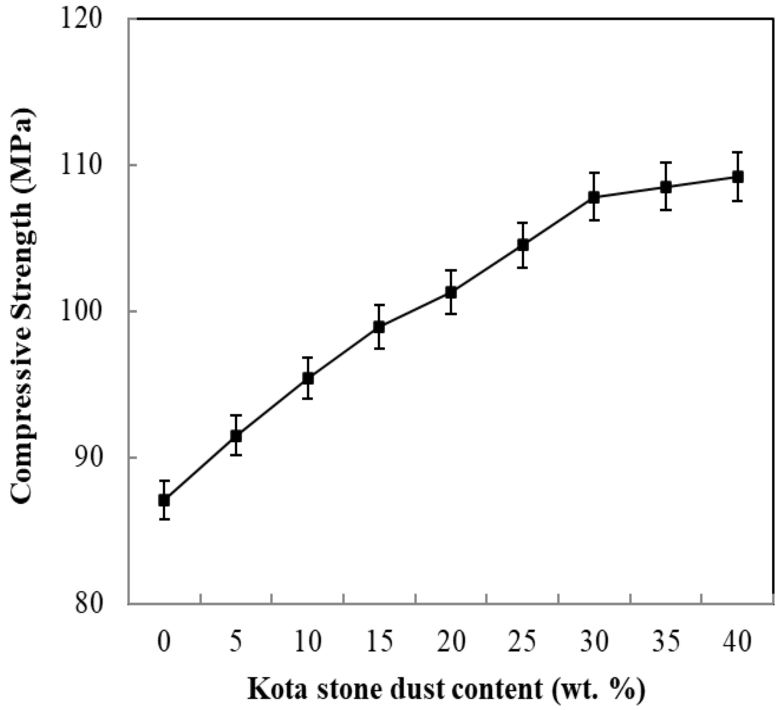

Figure 4: Compressive strength of the prepared samples

Hardness of the composites prepared also shows a similar behaviour as presented during the compressive strength discussion. The hardness also increases with the loading of Kota stone dust for the entire range of filler loading, as shown in Figure 5. The hardness of the unfilled polyester is 74 Shore-D. When the 5 wt. % Kota stone dust is added to it, the hardness increases to a very small value and reaches 74.6 Shore D. Even up to 15 wt. % of the filler, the improvement in hardness is limited and reaches 76.4 Shore-D. This shows an improvement of 3.2 %. At low filler loading, the improvement in hardness is low because the hardness is a surface phenomenon, and at low filler loading, it does not create much impact on the hardness. But when the filler loading increases above it, the rate of increment in hardness increases. For 40 wt. % of the Kota stone dust loading, the value of hardness reaches 84.2 Shore-D, registering an improvement of 13.78 %. The increment in the value of hardness is primarily due to the high hardness value of Kota stone dust. The improvement in hardness properties is mainly because the filler imparts a strengthening effect in the polyester matrix.

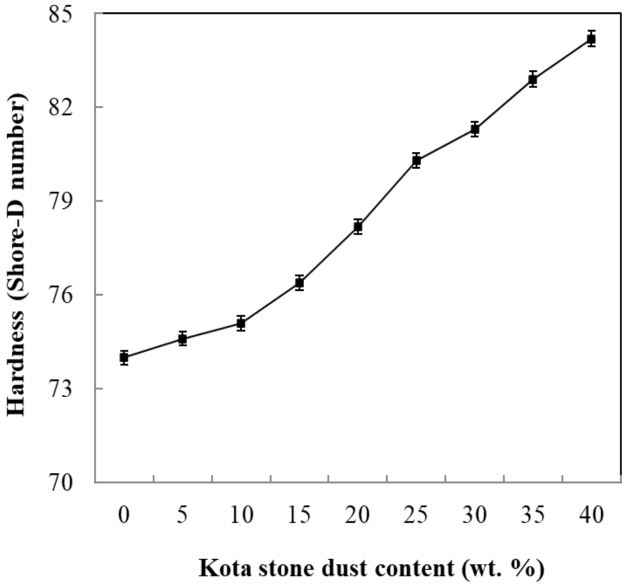

Figure 5: Hardness of the prepared samples

Thermal Properties

The thermal properties investigated in the present work are thermal conductivity, coefficient of thermal expansion, and glass transition temperature. The variation in the value of thermal conductivity with filler loading is presented in Figure 6. It is observed from the figure that the thermal conductivity of the composites increases as the content of the Kota stone dust in the polyester resin increases. Also, the increment is linear in nature. When the polyester is unfilled, the thermal conductivity measured is 0.235 W/m-K. The value increases to 0.269 W/m-K with the inclusion of 10 wt. % of the Kota stone dust showing an increment of 14.5 %. For a filler loading of 40 wt. %, the value of thermal conductivity increases to 0.394 W/m-K, registering an appreciable improvement of 67.6 %. The increment in thermal conductivity value with filler loading is primarily due to the higher thermal conductivity of Kota stone dust as compared to polyester resin. Further, the inclusion of Kota stone dust particles provides a crystalline and superior medium with a better path for heat transfer.

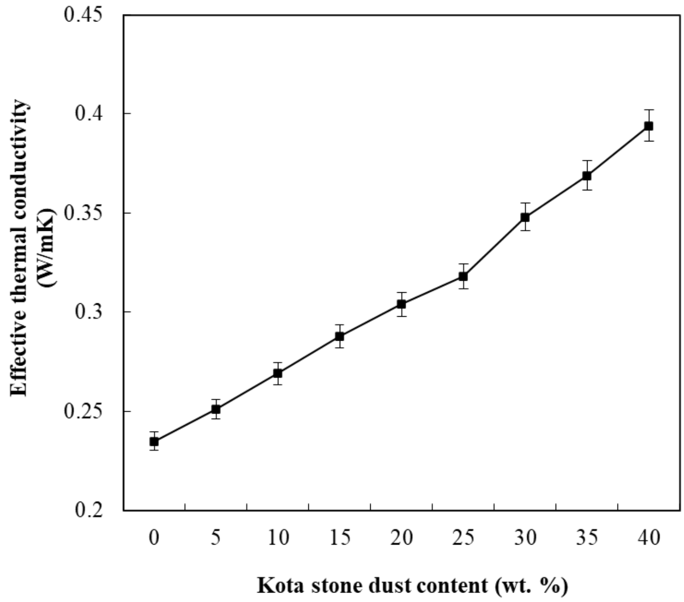

Figure 6: Thermal conductivity of the prepared samples

The variation in the value of the coefficient of thermal expansion of the polyester/LD slag composites at varied filler loading is presented in Figure 7. It can be observed from the figure that the inclusion of Kota stone dust in the polyester resin decreases the CTE of the composites as a function of filler loading. The CTE of unfilled polyester is 69.6 × 10^{-6} /ºC. This CTE is considered to be on the higher side, as high CTE is not desirable as it may cause the problem of thermal fatigue when the working temperature is fluctuating. With the inclusion of 10 wt. % of the filler, the CTE of the composite decreases to 66.4 × 10^{-6} /ºC showing a decrement of 4.6 %. With the further inclusion of the Kota stone dust, the CTE value further decreases and reaches a low value of 52.3 × 10^{-6} /ºC with a noteworthy decrement of 33.1 %. With an increase in filler loading, the CTE reduces primarily because of the low CTE of Kota stone dust. Further, the inclusion of Kota stone provides restriction in the movement of the polymer chain, which results in a decrease in the CTE of the composites.

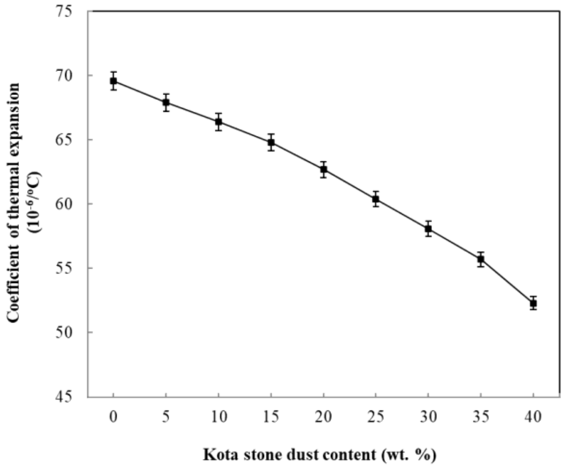

Figure 7: Coefficient of thermal expansion of the prepared samples

The glass transition temperature of the polyester composite at varied content of Kota stone dust is presented in Figure 8. It is clearly visible from the graph that the inclusion of the filler material increases the glass transition temperature of the composites, which is a desirable phenomenon. The glass transition temperature is defined as that temperature at which the material deviates from its brittle nature to ductile as it softens when the temperature rises. An increase in the glass transition temperature increases the working range of the material. The glass transition temperature of the polyester resin is measured to be 64.8 ºC. The maximum glass transition temperature was reached at 40 wt. % filler loading, which is 89.4 ºC. This value is an appreciable improvement of 37.9 %. The increment in glass transition temperature is mainly because the inclusion of particulate filler binds the polymer and restricts its mobility. Due to such a restriction, the glass transition temperature increases with filler loading.

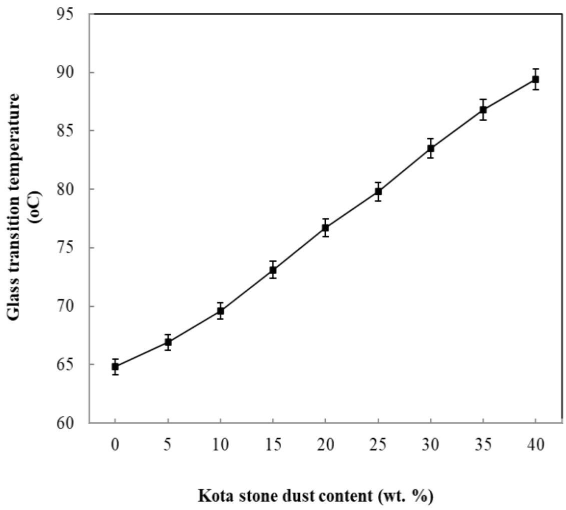

Figure 8: Glass transition temperature of the prepared samples

Conclusion

The physical, mechanical and thermal properties of the polyester composites filled with varied content of Kota stone dust (5 wt. % - 40 wt. %) are investigated. It is observed that the addition of Kota stone dust in the polyester results in an increase in the value of density, void content and water uptake rate as a function of filler loading. Further, the inclusion of Kota stone dust improves the mechanical properties like hardness and compressive strength for the complete range of filler content. Other mechanical properties, i.e. tensile and flexural strength, also increase with the filler content for a certain filler loading. The maximum tensile and flexural strength reported is for 30 wt. % of Kota stone dust loading, whereas compressive strength and hardness attain their maximum value at 40 wt. % filler loading. A noteworthy improvement in the thermal properties was also reported in the present work. The thermal conductivity increases from 0.235 W/m-K for unfilled polyester to 0.395 W/m-K with 40 wt. % Kota stone dust filled polyester. Similarly, other thermal properties like the coefficient of thermal expansion are reduced by 33.1 %, which is helpful in reducing the thermal fatigue and glass transition temperatures increase by 37.9 %, which enhances the working range of the material.

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