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PROPERTIES AND CHARACTERISTICS OF ALKALI TREATED CALOTROPIS GIGANTEA FIBER-REINFORCED PARTICLE-FILLED EPOXY COMPOSITES

The presence of particles and fibers as reinforcement in a polymer matrix greatly enhances the mechanical properties. Agricultural residues and natural fibers are commonly used nowadays due to the fact that they easily decompose even after a longer period and they are eco-friendly in nature. f Fiber that was extracted from stem of Calotropis gigantea was selected as reinforcement in the present investigation. Initially the fiber was treated with a sodium hydroxide solution and CG fiber--epoxy composites were prepared. The properties of alkaline treated CG fiber-reinforced epoxy composites were further improved by the addition of particles such as chitosan, red mud and rice husk. Properties such as the tensile strength, flexural strength, impact toughness, hardness, water absorption, thickness swelling behaviour, specific wear rate and coefficient of friction were evaluated and compared. The XRD pattern of the chemically treated CG fiber-reinforced parrticle-filled epoxy composites was presented in the present study.

Keywords: **rice husk particles, tensile strength, flexural strength, impact toughness, hardness, water absorption, thickness swelling behaviour, specific wear rate, coefficient of friction**

INTRODUCTION

The green fibers and natural particles have been effectively used as reinforcement materials in polymer composites in recent years. The three different bio particles were selected for the present investigation after a detailed literature survey. Chitosan is a sugar that is enriched with natural calcium and obtained from the hard outer skeleton of shellfish, including crab, lobster, and shrimp. The reinforcement of composites with chitosan has been performed by various researchers [1, 2]. The introduction of red mud particles influenced the mechanical, damping and chemical resistance properties of banana/polyester hybrid composites, which was studied in [3].

The required properties are to be evaluated in polymer composites before their domestic and industrial use. In addition to the mechanical properties, the thickness swelling behaviour of particulate composites was also studied [4, 5] previously. Balasundar et al. [6] developed eco-friendly composites which initiated the introduction of bio particles in polymer composites. Agricultural residues also influenced the dimensional stability and tensile behaviour of green composites [7, 8]. The strength of the natural fiber reinforcement is improved by their chemical treatment.

Madhu et al. [9] studied the effect of various chemical treatments of Prosopis juliflora fibers as composite reinforcement as well as the physicochemical, thermal, mechanical, and morphological property of composites. The most recent studies used red mud particles as reinforcement in composites due to their high amounts of alumina and silica [10].

The friction and wear behaviour are studied in addition to the mechanical properties and water absorption studies to state the effective applications of composites [11, 12]. The mechanical and water absorption behaviour of natural fiber-reinforced hybrid biocomposites was studied by Sekar et al. [13]. A review of the treatment of natural fibers in polymer composites as carried out by Nurazzi et al. [14] proved the importance of interfacial bonding to improve the mechanical properties, and the applications in the automotive sector by recent researchers has paved the way for the development of hybrid composites [15]. In addition to the mechanical properties, the moisture content is measured in polymer composites to state the proper applications as suggested by Ramakrishnan et al. [16] and thermal characterization is measured to select the environment for use [17]. These previous studies created interest in the development of a new variety of natural fiber-reinforced epoxy composites with particles.

MATERIALS, MANUFACTURING AND TESTING

Fiber extraction and treatment

The CG fibers were extracted from the stem of one-year-old plants and the leaves were removed. The required length of the stems was cut and they were dried at room temperature for 5 days. The fibers were manually extracted from the dried stems by decortication which is removal of the cortex followed by extraction of the fiber by means of scraping, which was done with a knife. Finally, the fibers were dried for 2 days and cut into the required length to prepare the composite lamina. The CG fiber was washed with water and dried at room temperature for two days. The surface modification was done by alkali treatment of the fiber with a 5% sodium hydroxide solution for 30 minutes. This type of treatment eliminates a certain amount of the lignin, oil and wax covering the external surface of the fiber cell wall. The alkaline treatment increases surface irregularities, ensuing better mechanical interlocking by increasing the amount of cellulose exposed on the fiber surface, thereby increasing the number of possible reaction sites.

Preparation of particulates and epoxy system

Red mud was obtained from MALCO (Madras Aluminium Company Limited) at Salem, India. The presence of natural organic matter such as iron oxide followed by aluminium oxide and silicon dioxide in red mud enhanced the value of the mechanical properties when reinforced with epoxy matrix [18]. The use of calcium oxide enriched particles can improve the static and dynamic mechanical strength of composites. Natural calcium material such as chitosan is selected as particle reinforcement because it is abundantly available and is dumped in the sea as waste. The use of natural calcium will help to utilise bio waste effectively for engineering applications. Rice husk (RH) is a waste material generated in agricultural practices and abundantly available in rice based agricultural countries. It is a natural waste from the sheath that is formed over rice grains during its development and the same is removed during the refining of rice. Rice husk is a stringy material and it is employed as a filler material to make lightweight polymer composites. It is enriched with natural silica that enhances the values of the mechanical and thermal properties of composites.

The Calotropis gigantea (CG) plant grows up to a height of 3-5 m and consists of oval shaped light green leaves and a milky stem. The fibers extracted from the stem of Calotropis gigantea plants are used for composite manufacturing. The chemical analysis was carried out on the extracted CG fibers as per the Technical Association of the Pulp and Paper Industry (TAPPI) Standard. A cellulose content of 73.8% and hemicelluloses of 20.8% were observed in the CG fibers during the chemical test.

Epoxy offers excellent moisture obstruction qualities when used in composites. It bonds well with fibers when fabricating fiber-reinforced composites. The polymerization reaction was performed to transform the liquid resin to a solid and was conducted by adding a minimal quantity of a reactive curing agent just before adding the fibers into the liquid mixture. One of the curing agents was an amine based hardener (HY 951) and was utilized to prepare the composites with a mixing ratio of 9:1 as recommended. The epoxy resin (LY 556) and the hardener were supplied by M/S Covai Seenu & Company, Coimbatore, Tamilnadu, India.

Sodium hydroxide pellets supplied by M/S Spectrum Reagents and Chemicals Private Limited, Edayar, Cochin, India, were used for the alkaline treatment of the CG fibers. Approximately 30 grams of CG fiber was soaked in 800 ml of the sodium hydroxide alkaline solution. The fibers were removed from the solution, washed with fresh water several times and finally rinsed with distilled water to remove any excess NaOH sticking to the fiber. Chemical tests were conducted on the CG fiber after the treatment with the 5% sodium hydroxide solution.

Compression molding process

A 30 ton capacity ACE hydraulic compression molding machine was used to fabricate the CG fiber-epoxy composite plates with the dimensions 30 × 30 × 0.3 cm. The appropriate weight content of the CG fiber, particles and epoxy were mixed by mechanical stirring at 20 rpm for 10 minutes at room temperature. The particulate content of 20 wt.% and fiber content of 30 wt.% were selected as the reinforcement percentage as per the literature review. The fiber length of 30 mm was selected during the fabrication of short CG fiber-reinforced epoxy composites. A constant compression pressure of 2.6 MPa and temperature of 80°C for 45 min facilitated uniform curing of the composite sheets. The atmospheric conditions of 28°C and relative humidity of 55% were recorded during composite manufacturing.

Testing equipment

A Tinius Olsen H10KL dual column digital Universal Testing Machine equipped with a 5 KN load cell was used to perform the tensile tests as per the ASTM D 638 standard. Five specimens 165 × 13 × 3 mm were used for the testing. At the cross-head speed of 1 mm/min the average value of tensile strength of the composite specimens was recorded.

Flexural tests (three-point bending) were conducted with the Tinius Olsen H10KL, which is equipped with a load cell in the range of 5 KN, as per the ASTM D 790 standards. Five specimens were tested from each composite at the crosshead speed of 2.5 mm/min. The flexural test was carried out on specimens 125 × 12.5 × 3 mm and five specimens were tested to obtain statistically significant results for each condition.

The impact toughness (Izod) was conducted using a Tinius Olsen IT504 plastic pendulum impact tester as per ASTM D 256. The testing machine is equipped with a pendulum with potential energy in the range of 2.57 J. Five un-notched specimens 64 x 12.7 x 3 mm were tested for each case and the average value was noted as the impact strength. The specimens for the impact test were cut from the fabricated composite and finished to the accurate size using sandpaper. The maximum pendulum capacity of the impact tester is 25 J and the maximum impact velocity is 3.46 m/s. The specimen used for testing was made as a cantilever beam and was placed in the vertical axis. When the pendulum swings and strikes the face of the sample, the sample breaks. A total of 5 samples were tested.

The hardness test is an indicator of the mechanical properties of the material that not only elicits the importance of the surface properties but also the friction and wears processes. It is also a simple and quick way to measure the hardness of the material and to determine the mechanical properties of the samples. The principle behind the hardness test is to press an indenter on the sample surface and to measure the resultant dimensions of the indentation (depth or actual surface area of the indentation).

The hardness of the CG fiber-epoxy composites was measured using a Vickers hardness tester, with a ball diameter of 2.5 mm and a load of 10 kgf. The Vickers hardness test was conducted according to the ASTM E10 standard on specimens 76.2 ×76.2 ×3 mm. The hardness was measured at 10 different points on each specimen and the average value was noted as the specimen hardness and is presented in terms of the Vickers Pyramid Number (HV). A diamond indenter, in the form of a right pyramid with a square base and an angle 136° between the opposite faces, is pressed against the material under a load.

The water absorption behaviour of the CG fiber--reinforced epoxy composite specimens was investigated as per the ASTM standard D570. A digital scale with an accuracy of 0.001 mg was used to measure the weight of the specimens. The percentage of water absorption can be calculated as per the equation given below:

$$\text{Parcentage of water abortion } W\% = \frac{W2 - W1}{W1} \times 100$$

where: W1 – weight of the dry specimen, W2 – weight of the water absorbed specimen.

The tribological test was conducted as per the ASTM G-99 standard and specimens 30 × 4 × 4 mm were obtained from the samples. The counterspecimen was a hardened alloy steel disc with a surface roughness of 0.25-0.30 μm and the experiment was conducted at room temperature. Accurate sizes of the samples were obtained using an emery sheet to ensure proper contact between the pin and the disc. The pin was initially weighed using an electronic balance meter with an accuracy of 0.0001 g. The sliding wear was measured using the weight loss method, in which the difference between the initial and final weights of the specimen was calculated.

Specific wear rate

$$k_s = \frac{V}{L \times D} \ [\mathrm{mm^3/Nm}]$$

where V is the volume loss [mm^3], L is the applied load [N], D is the sliding distance [m].

Five specimens were tested for each material and thus the average of the readings was used to calculate the wear. The specific wear was measured by the loss in weight, which was converted into wear volume and then the specific wear rate was calculated using the above equation.

XRD set up

X-ray diffraction analysis was used to identify the organic materials present in the rice husk, red mud and chitosan particles. The XRD analysis was carried out with a PW3040/60 PANanalytical X’Pert Pro (CECRI-Karaikudi) instrument employing Cu Kα (λ = 1.54). The samples were placed in an aluminium holder and scanned at 45 kV and 40 mA, with diffraction intensity in the range of 10 to 90° at 2θ and the exposure time for each sample was 120 s at a step size of 0.06°. The diffraction patterns were analysed using X’Pert High Score software. Photographs of the XRD instrument, hardness tester and tribological tester are shown in Figure 1.

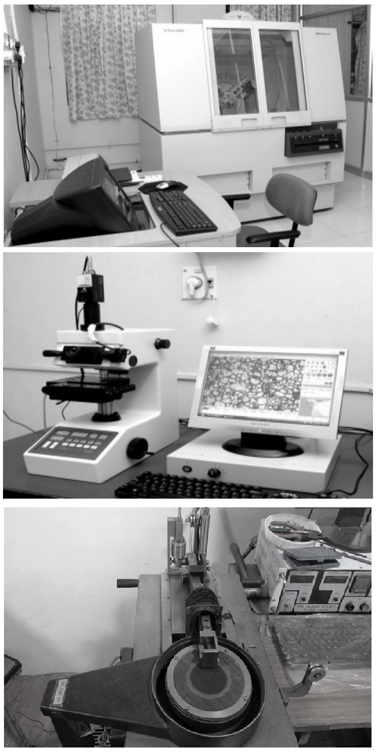

Fig. 1. Photographs of XRD instrument, hardness tester and tribological tester

RESULTS AND DISCUSSION

The mechanical properties of the alkali treated CG fiber-reinforced epoxy composites with particles are given in Table 1. The best values of tensile, flexural and impact toughness, 75.8 MPa, 107.1 MPa and 81.2 kJ/m^2, were obtained by the alkaline treated CG RH epoxy composites. The particles were prepared using high-energy ball milling equipment and then mixed with the epoxy system, which enhanced the mechanical properties of the alkaline treated CG fiber-reinforced epoxy composites.

TABLE 1. **Comparison of properties**

No.	Properties	CG epoxy	Alkaline treated CG epoxy	Alkaline treated CG CT – epoxy	Alkaline treated CG RM epoxy	Alkaline treated CG RH epoxy
1	Tensile strength [MPa]	62.7	64.3	67.9	69.3	78.2
2	Flexural strength [MPa]	60.5	80.3	87.4	89.6	107.1
3	Impact toughness [kJ/m^2]	43.5	56.2	63.8	66.1	81.2

Mechanical properties of chemically-treated CG EP particle filled composites

Tensile strength

The tensile strength results of the CG-EP composites and alkali treated (NaOH) CG-EP particle-filled composites are shown in Figure 2. The tensile value of the CG-EP composites shows low values (62.7 MPa), compared to the other composites. The sodium hydroxide treated CG-EP composite had a 2.6% higher tensile strength. The addition of particles, chitosan, red mud and rice husk, increased the tensile strength of the composites by 8.3, 10.6 and 24.8% respectively. The highest value of tensile strength (78.2 MPa) was attained by the 55 wt.% CG, 5 wt.% wt RH and 40 wt.% EP composite. Similarly, the Young's modulus of the CG-EP composites with RH was increased to 9.1 KPa. This is due to the good agreement of RH with CG fiber, and the higher amount of Si leads to a rise in the tensile strength of CG-EP composites [9].

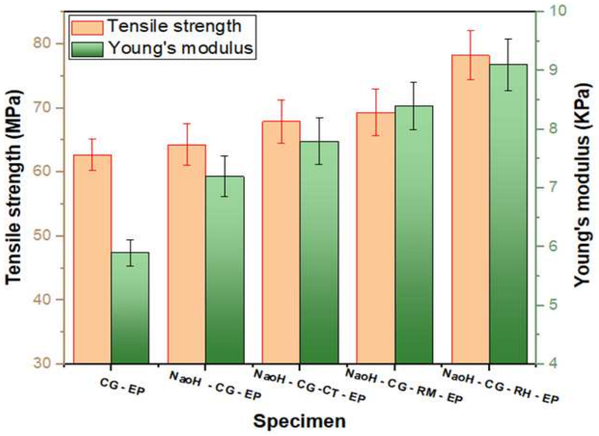

Fig. 2. Effect of particle addition on tensile strength of CG-EP composites

The reason behind the increasing tensile strength is the alkali treatment that increases the roughness of the CG fiber, which is attributed to the better interfacial bonding between the CG fiber and epoxy matrix, which enhances the tensile properties of the composites. This basically because the agricultural waste RH is a highly stiff particle; therefore, the addition of rice husk increases the strength [4, 5, 19]. Similarly, the CG-EP composites containing chitosan, exhibit improved Young's modulus and tensile behaviour; the same results were observed in grapheme/chitosan composites [1].

Flexural strength and impact toughness

The variations in the flexural and impact strength of the CG-EP particle-filled composites are presented in Figure 3. The CG-EP composites exhibited a flexural strength value of 60.5 MPa, whereas the incorporation of particle moderately increased the flexural strength: 87.4 MPa (CG-CT-EP), 89.6 MPa (CG-RM-EP), 107.1 MPa (CG-RH-EP) respectively, which is 44.5, 48.1 and 77.1% higher than the CG-EP composites. The addition of red mud particles to the polymer raises the flexural strength owing to presence of Fe_2O_3 and Al_2O_3 in the red mud particles, which were used as fine aggregates to fill the voids during the preparation of the composites [3]. On the other hand, RM serves as an excellent rubber reinforcing filler, which enhances the flexural properties of CG-EP composites [10].

In contrast, the alkaline treated CG-EP composites with RH exhibited the highest impact strength (81.2 kJ/m^2, SD 4.06) compared to the other composites and followed by the other particle-filled CG-EP composites. This was anticipated because RH particles should make the polymer more resistant to crack propagation [8].

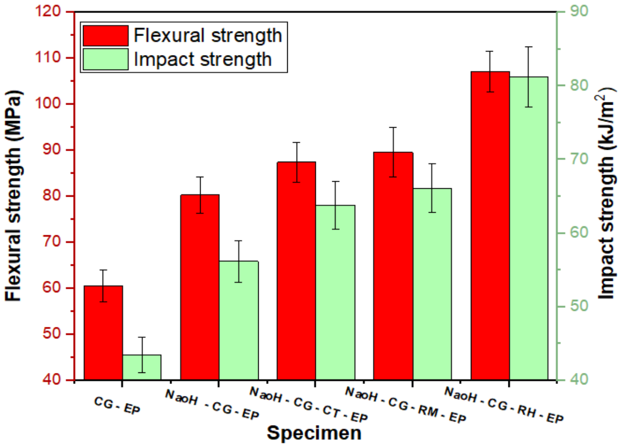

Fig. 3. Effect of particle addition on flexural and impact strength of CG-EP composites

Hardness

The variations in the hardness of the particle-filled CG-EP composites are illustrated in Figure 4. The hardness of the CG-EP composites increased from 19 to 28 HV with the addition of chitosan particles. Due to the uniform dispersion of the chitosan particles in the

CG-EP composites the hardness of the composites grew [2]. Similarly, the composites with the addition of RH, RM and NaOH treated CG fiber, the hardness values increased from 19 HV to 24 HV, 26 HV and 25 HV respectively. The increased hardness of the composite with the addition of red mud particles is primarily owing to the strong presence of mineral oxides in red mud, but it also increases the brittleness of the composites [12, 18].

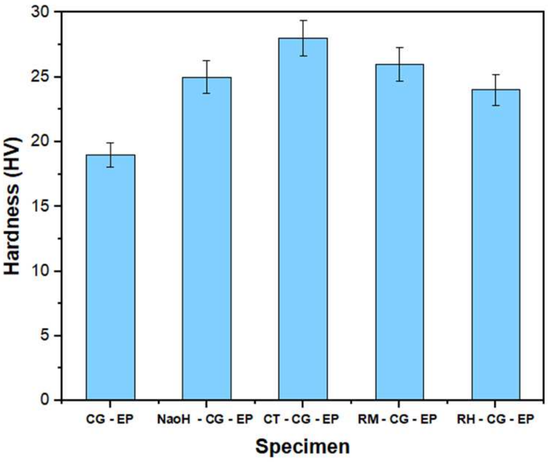

Fig. 4. Effect of particle addition on hardness of CG-EP composites

XRD analysis

The XRD pattern for the chemically treated and particle-filled CG–EP composites is presented in Figure 5. The pattern clearly indicates that the main crystalline peaks for the composites occurred at 2θ = 22.8° (CG-EP), 2θ = = 22.3° (NaOH CG-EP), 2θ = 34.2° (CT-CG-EP), 2θ = 28.2° (RM-CG-EP), 2θ = 34.1° (RH-CG-EP) respectively.

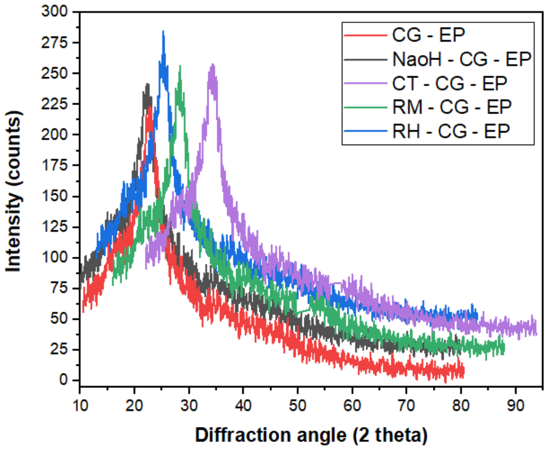

Fig. 5. XRD pattern of particle -filled CG-EP composites

The crystalline line (CI) value of the CG-EP composites was calculated using the Segal method. The value is 33.1% (CG-EP), followed by other composites 34.5% (NaOH-CG-EP), 48% (RH-CG-EP), 41.3% (RM-CG-EP) and 46.6% (CT-CG-EP) [6]. It is noticed that the addition of CT and RH particles in the CG-EP composites extended the crystalline peak to some extent.

Water absorption and thickness swelling behaviour

Figure 6 indicates the effect of CG on the water absorption of CT/RM/RH/EP composites at 24, 48, 72, 96, 120, 144, 168, 192, 216 and 240 h. The % of water absorption grew linearly with increasing the immersion time. After 7 days there was a gradual slowdown in the water absorption rate until it finally reached the level of saturation; this phenomenon is commonly called the equilibrium state. Similar results were noticed with the addition of rice husk to high-density polyethylene and Calotropis gigantea reinforced phenol-formaldehyde composites [13]. It was observed that in the composites soaked for 10 days the water absorption values were 26.5% (CG-EP), 25.5% (CT-CG-EP), 26.5% (RH-CG-EP), 27.5% (RM-CG-EP), 28.5% (NaOH-CG-EP).

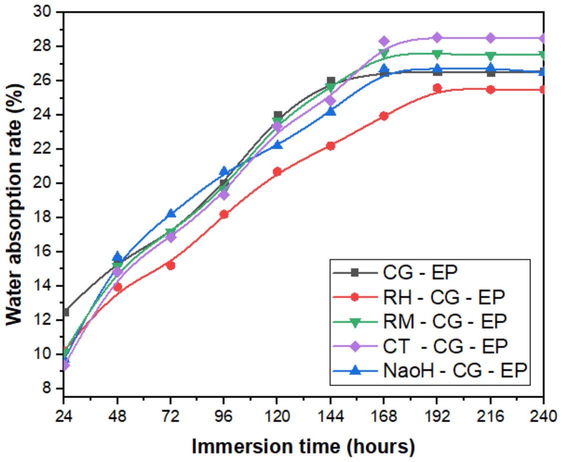

Fig. 6. Water absorption of particle -filled CG-EP composites

Higher amounts of water were absorbed by the NaOH treated CG-EP, RM-CG-EP composites. The CG fibers are plant fibers containing a large number of hydroxyl groups that have a strong water absorption capability, which leads to the higher water absorption of CG-EP composites [7]. That is the why the RM filled composites exhibit the lowest mechanical strength compared to the other composites.

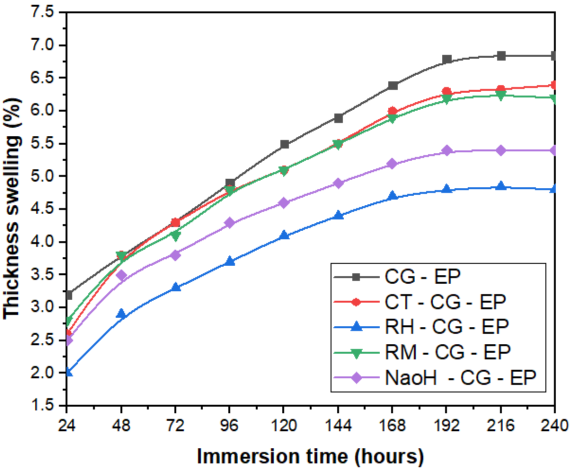

Fig. 7. Swelling behaviour of particle – filled CG-EP composites

Figure 7 displays the swelling behaviour of the particle – filled CG-EP composites. It was noticed that similar results were observed in the thickness swelling due to water absorption. The particles used in this investigation are plant fiber, agricultural and industrial waste based particles. Principally, they are kinds of cellulosic fiber and natural particles; in that way, water or moisture simply build up in the fiber wall, leading to thickness swelling and dimensional instability [4, 5]. Changes in the thickness can be seen in the CG-EP (6.8%), CT-CG-EP (6.3%) and RM-CG-EP (6.2%) composites. The particle – filled composites exhibit small reductions in swelling; that is the reason why the particle-filled composites possess better mechanical properties.

Specific wear rate and coefficient of friction

Figures 8 and 9 illustrate the wear behaviour and coefficient of friction of the different filled CG-EP composites. It was observed that increasing the sliding distance (250-1250 m), leads to a rise in the specific wear rate (mm^3/N-m) for all the composites. The smallest wear rate was observed in the NaoH treated CG-EP composites (0.0392 mm^3/N-m) and the highest wear rate in the CG-EP composite (0.99 mm^3/N-m), which is 96.1% higher than the alkaline treated composites. This is owing to the removal of the non-cellulosic components of of the CG fiber.

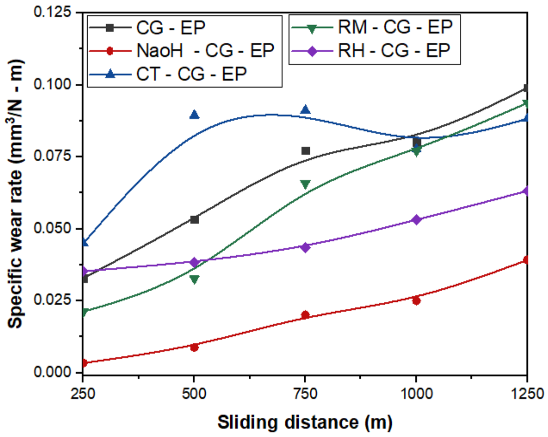

Fig. 8. Specific wear rate of particle -filled CG-EP composites

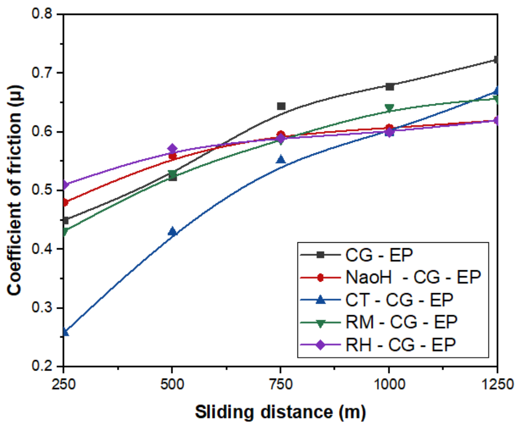

Fig. 9. Coefficient of friction of particle-filled CG-EP composites

As can be seen from the results, there is an appreciable reduction in the wear rate with the addition of particles; the RH particle-filled CG-EP composites exhibited a specific wear rate of 0.0632 mm^3/N-m, CT-CG-EP 0.0833 mm^3/N-m, RM-CG-EP 0.0938 mm^3/N-m at a 1250 m sliding distance. It is clear from the plot that the particle filled and NaOH-treated CG composites exhibited a lower wear rate compared to the untreated CG and unfilled composite. This is due to the higher load bearing capacity and good interfacial bonding between the particles and matrix, leading to a lower wear rate [11].

A similar trend was noticed in the coefficient of friction plot (Fig. 8); increasing the sliding distance leads to a rise in the coefficient of friction. The highest coefficient of friction was recorded for the CT filled (0.66 µ) and untreated unfilled composites (0.722 µ); this is owing to the abrasion of the fine CT particles, increasing the contact temperature, which leads to a higher coefficient of friction and more wear [20].

CONCLUSIONS

The addition of rice husk particles to the Calotropis gigantea fiber-reinforced epoxy composites extended the crystalline peak to some extent, which resulted in better mechanical properties. The better values of tensile strength, Young's modulus and impact toughness were obtained by the alkaline treated CG fiber-reinforced rice husk particle filled epoxy composites. The flexural strength is superior in the alkaline treated CG fiber-reinforced red mud particle filled epoxy composites due to the presence of natural silica and alumina in the red mud particles. The water absorption and thickness swelling behaviours were lower in the alkaline treated CG fiber-reinforced epoxy composites with rice husk, whereas the specific wear rate was lower in alkaline treated CG fiber-reinforced epoxy composites. It is observed that the CG fiber-epoxy composites will be successfully used for engineering applications by the inclusion of 50-100 micron particles.

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