Silicon (2021) 13:1199–1207
https://doi.org/10.1007/s12633-020-00508-z

ORIGINAL PAPER

Role of Silicon Coupling Grafted Natural Fillers on Visco-Elastic, Tensile-Fatigue and Water Absorption Behavior of Epoxy Resin Composite

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Received: 18 January 2020 / Accepted: 28 April 2020 / Published online: 1 June 2020

Abstract

This work investigates the influence of silicon based coupling agents on visco-elastic properties of natural filler dispersed epoxy resin composites. Also this work attempts to explore the possibility of using silicon coupling grafted natural fillers as potential fillers for polymer composites. Ground nut shell power (GS powder), rice husk and saw dust were selected as reinforcement for this current investigation. The powders were silane surface grafted using silicon coupling agent 3-Aminopropyltrimethoxysilane via aqueous solution method. The composites were prepared via gravity casting method and post cured at 120°C. The visco-elastic behavior of silane surface modified rice-husk-epoxy natural filler composite gives improved results in storage modulus, and loss tangent. Similarly, the fatigue results revealed that the composites made with 10 vol.% of silane surface treated rice husk filler gives maximum fatigue life cycle of 1310. The sessile drop results show that the silane surface modified epoxy composites retains higher absorption resistance by offering higher contact angle even after the natural fillers are filled. Scanning electron microscope images revealed highly reacted phase and improved dispersion of natural fillers with matrix. These natural fillers strengthen epoxy composites could be right choice to replace many metallic based materials in engineering applications.

Keywords PMC · Natural fillers · Siliconization · Visco-elastic properties · Fatigue and water absorption

1 Introduction

The demand of natural fibre and filler composite is now evolving due to the current world's trend and eco-friendly nature. There are many researchers done their research study in natural fibre polymer composites and many more applications are now could see in automobile and other industrial sectors [1].

The natural fibre composites are capable for serving high load bearing applications like synthetic fibres and maintain eco-friendly nature. The natural fibre composites are better in strength, toughness and zero harmful effect. Thus they are widely prescribed by many automobile sectors. The world's famous German car makers also now replaced their automobile body with natural fibre composites due to their size to strength ratio, cheaper cost, considerable strength and eco-friendly nature [2]. But later twenty's the researchers from various regions focused their research on natural fillers since they offer good strength, dynamic mechanical properties and good repeated load bearing (fatigue) capacity. In this natural fillers like rice husk, egg shell, sea urchin, cow dunk, corn husk are lined up and lot more research studies have been carried out so far [3, 4]. Arun et al. [5] researched the role of adding a sea based novel bio filler sea urchin particle in neem epoxy hybrid composite. The researchers confirmed that the addition of sea urchin filler improved the mechanical, thermal and wear properties of composite. Similarly, Parivendan et al. [6] investigated the effect of dispersing the silane-modified micro egg shell particle into natural fibre-epoxy composite as a potential filler. The authors concluded that the egg shell

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particle dispersion improved the wear and mechanical properties of NF-epoxy composite. In any composite the role of matrix also plays a vital role. Based on this factor the selection of thermoset matrix is getting much attention. There are many thermoset plastics to prepare the natural filler composites. Among all the thermosets, the epoxy attracted the researchers view because of its excellent electrical, chemical and thermo-mechanical behaviors [7, 8]. Yixin et al. [9] studied the effect of adding novel hetero-structured silicon carbide-boron nitride nanosheets as SiC-BNNS/epoxy thermally conductive nano-composites for high thermal conductivity application. The authors have confirmed that adding SiC-BNNS improved thermal conductivity at the same time maintains high electrical insulation. Xutong et al. [10] studied the synchronously improved electromagnetic interference shielding and thermal conductivity for epoxy nanocomposites constructed using 3D copper nanowires and thermally annealed graphene aerogel framework. The outcome of the study revealed that the maximum electromagnetic interference shielding effectiveness (EMI SE) value of 47 dB and electrical conductivity (σ) of 120.8 S/m, ascribed to perfect 3D CuNWs-TAGA conductive network structures. Meanwhile, the corresponding elasticity modulus, hardness, glass transition temperature (Tg) and heat-resistance index (THRI) of the CuNWs-TAGA/epoxy nanocomposites increase to 4.69 GPa, 0.33 GPa, 126.3 °C and 181.7 °C respectively. Usually, the surface treatment on fillers may improve the dispersion of filler in matrix medium and contribute more on the thermo-mechanical properties [11, 12]. Moreover, other notable properties like electrical insulation and thermal conductivity also could improve for surface treated fillers in epoxy composites. The surface treated fillers dispersed epoxy composites may good in thermal conductivity behavior without losing their excellent electrical insulation [13, 14]. Thus in composite making the surface-modification of fillers or particles paid much attention by the researchers since surface-modification renders many notable improvements. The surface treatment process could be done by many ways. Acid, base and silane treatments are the three famous methods to produce surface treated reinforcements with simple process parameters. When the acid and base treatments are done on the reinforcements their cross section gets leached, which intern lead plastic deformation. But in silane surface modification the silane element covers the natural fillers without affecting their actual cross section. Hence the originality never disturbed and improved the performance of composite. On comparing with acid and alkali treatment the silane treatment is said to be better by many researchers. Tahir et al. [15] investigated the effect of surface modified MWCNTs and kenaf fibre in epoxy composite and their EMI shielding behavior. The authors concluded that the silane surface modified MWCNTs possess high shielding effect even the temperature is raised more than Tg of polymer. Similarly, Parthipan et al. [16] investigated the effect of silane modified kenaf fibre and silicon oxide particle in epoxy composite. The authors confirmed that addition of silane surface modified reinforcements improved the mechanical and machining behavior of composite. An amine based silane surface modifier could be selected for treating the surfaces of fillers since the base matrix used here is epoxy resin. To cure epoxy resin usually amine based curing agents are used. These amine curing agents supply electrons from nitrogen for making bonds. Similarly, the amino functional coupling agent has NH_2 functional group, which could react with epoxy resin during composite making. Based on the previous studies the role of natural fillers in polymer and need of silane surface-modification is explicated with quoted references. Very low numbers of researchers are done research on silane treated fillers and their effects in polymer composites. Moreover, their researches are mainly focused on thermal, mechanical and wear behavior. But still more important properties like visco-elastic, fatigue and creep need to be studied when overall composite properties are required for any specific applications. In this connection the present study aims to investigate the visco-elastic, fatigue and water absorption behavior of epoxy composites with silane surface-modified natural fillers. In this present study the natural filler like saw dust, ground nut shell powder and rice husk were selected since, they are cheap, abundantly available, easy processing methods and bio compatible nature [17]. Moreover, there are very few research only been done on these natural fillers, hence the interst on these fillers getting higher. The natural fillers volume may be varied for knowing the optimum quantity to be used for not making any adverse effects on the visco-elastic, fatigue and water absorption properties. The natural filler-epoxy composite could be prepared using blending-casting method since; it doesn't require any specific process parameters [18]. These natural fibre strengthened polymer composites could be useful in engineering applications such as automobile body parts, driver cabin essentials, light weight drown aircraft making and domestic applications [19].

2 Experimental

2.1 Materials

In this present study a quick set Diglycidyl ether of Bisphenol-A (DGEBA) araldite epoxy resin with 190.1 g/mol molecular weight with density of 1.19 g/cm3 used as base matrix. An aliphatic hardener Triethelenetetramine of density 0.9 g/cm^3 was used as a curing catalyst and purchased form Huntsman India Ltd. The silane surface modifier APTMS having density of 1.027 g/cm^3 is purchased from Sigma Aldrich, USA. The natural fillers ground nut shell powder saw dust and rice husk of equal size 5 μm and density as 1.12, 1.14, 1.11 g/cm^3 were purchased from Gogreen, India, Pvt. Ltd. The attachment chemicals like ethanol, acetic acid, filter paper and distilled water for surface treatment process were purchased form MERCK India Ltd. Figure 1 shows the scanning electron microscope image of ground nut shell powder saw dust and rice husk used in this present study. The morphology revealed that the particles are in uniform shape and not in agglomerated form.

2.2 Silane Surface Modification

The proposed silane surface modification was done using ethanol-water aqueous solution. In this the aqueous solution was prepared using ethanol of 95 wt.% and distilled water of 5 wt.%. The water is added with ethanol to dilute it for silane modification process. Acetic acid was added with the ethanol-water solution to maintain the pH as 4.5–5.5. The pH fixed aqueous solution was then mixed with 3-4 wt.% of silane agent as drop by drop and mixed gently. The silane mixed solution was then stirred for 10 min to get homogeneous solution. The natural fillers are then briefly immersed into the silane solution and waited for 10 min. After condensed silane surface modification over the immersed fillers are separated from the silane solution and dried in a hot oven at 110°C for 10 min to form Si-O-Si structure [20]. Figure 2 shows the scheme of silane surface modification on filler surface.

Figure 3 shows the FT-IR spectral analysis of silane surface modified natural fillers. The peaks at 3185 cm^{-1} indicates the presence of NH_2 amine group on natural filler surface. Similarly, the peaks at 2925 and 1410 cm^{-1} indicates the presence of C-H stretch on particle surface from attached silane. A peak at 1028 cm^{-1} indicates the presence of Si-O-Si structure on the particle surface from post curing of silane surface modified natural fillers. Thus the process of silane surface

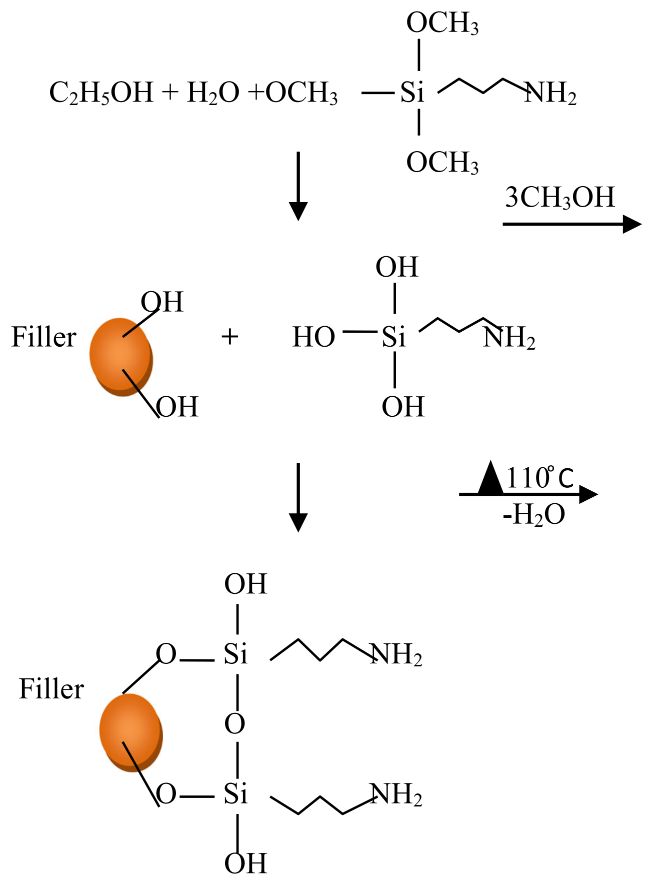

Fig. 2 Scheme of silane surface-modification

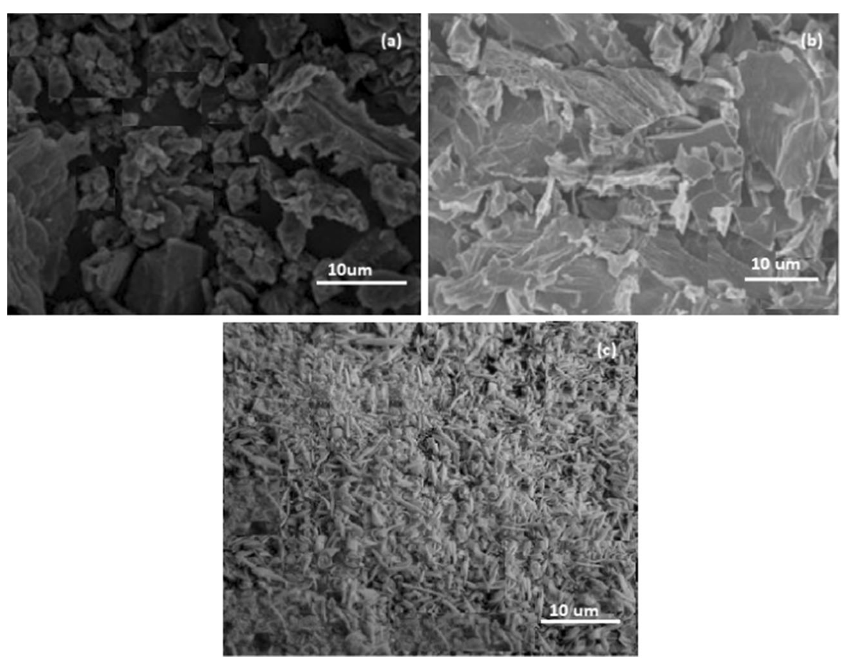

Fig. 1 SEM image of **a** groundnut shell, **b** saw dust and **c** rice husk powders

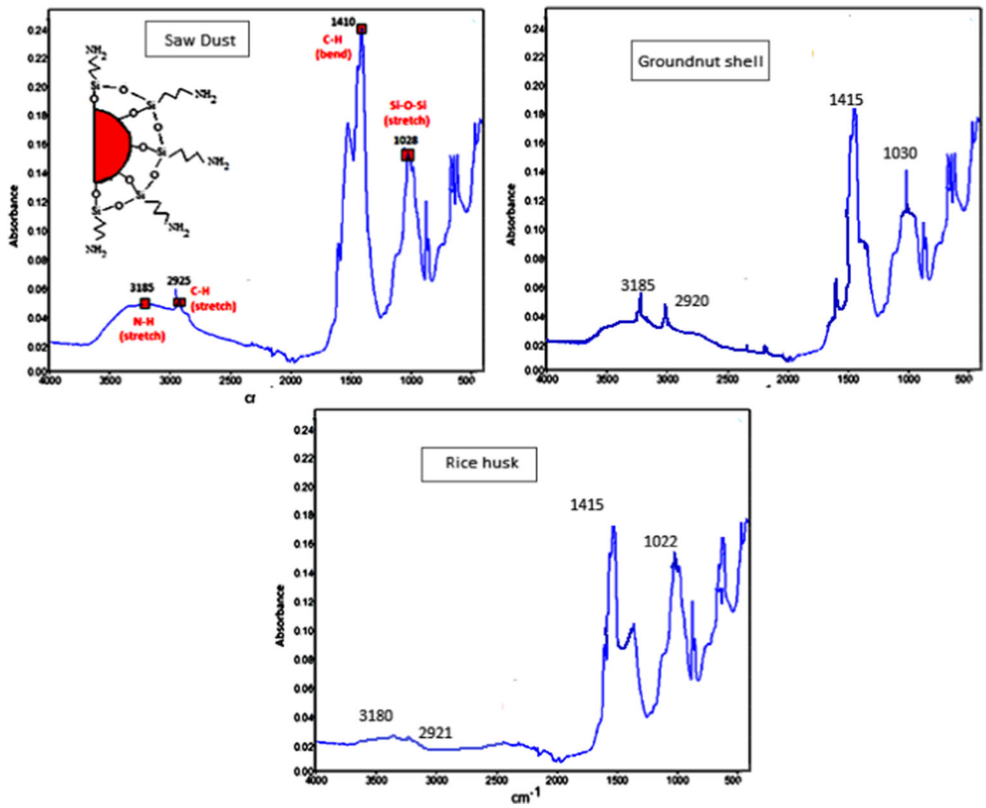

Fig. 3 FT-IR spectra of silane surface treated natural fillers

modification induced the amino group (NH_2) on the filler surface, which may react with epoxy resin during composite making [21].

2.3 Composite Making

The silane surface treated natural fillers of suitable volume percentages were mixed with epoxy resin of fixed quantity and stirred completely until a homogeneous solution was formed. The resulted suspension was then mixed with curing agent and continues in stirring for 10 min to get complete mixing of hardener with matrix. The resulted solution was then poured into a silica rubber molds and allowed for curing at room temperature for 24 h and post cured again at 120 °C for 48 h to get complete cross linking to be taken place [22]. The post cured samples were checked for visual defects and cleaned with cloth. The cased test specimens were made accurate to their dimensions based on ASTM standards. Table 1 shows the designations and compositions of natural filler composites fabricated.

3 Characterization

The gravity casted epoxy-natural filler composites are tested based on ASTM standards. The visco-elastic properties have been evaluated based on ASTM D 4065 with temperature sweep mode. The temperature variation applied here was between 30 °C to 240 °C at a constant frequency of 1 Hz with dual form cantilever fixtures with a heating rate of 5 °C/min. The fatigue behavior of natural filler-epoxy resin composite was tested based on ASTM D 3479 by using a tension-tension fatigue testing machine (MTS Landmark 370, USA). The R value was maintained as 0.1 with loading frequency of 5 Hz. The load of 1KN, which is equal to 50% of highest stress and tensile modulus of 6.0GPa at 25 °C working ambience, was set as process parameters. The micrographs of natural filler epoxy composite were characterized using a scanning electron microscope (HITACHI 1500 and JEOL JEM 2100 JAPAN). The sessile drop test of natural filler-epoxy resin composite

Table 1 Designation and composition of composites

Composite Designation	Epoxy (vol.%)	Groundnut shell (vol.%)	Saw dust (vol.%)	Rice husk (vol.%)
E	100	0	0	0
EG_1	90	10	0	0
EG_2	80	20	0	0
ES_1	90	0	10	0
ES_2	80	0	20	0
ER_1	90	0	0	10
ER_2	80	0	0	20

E, Epoxy; *G*, groundnut; *S*, saw dust and *R*, Rice husk

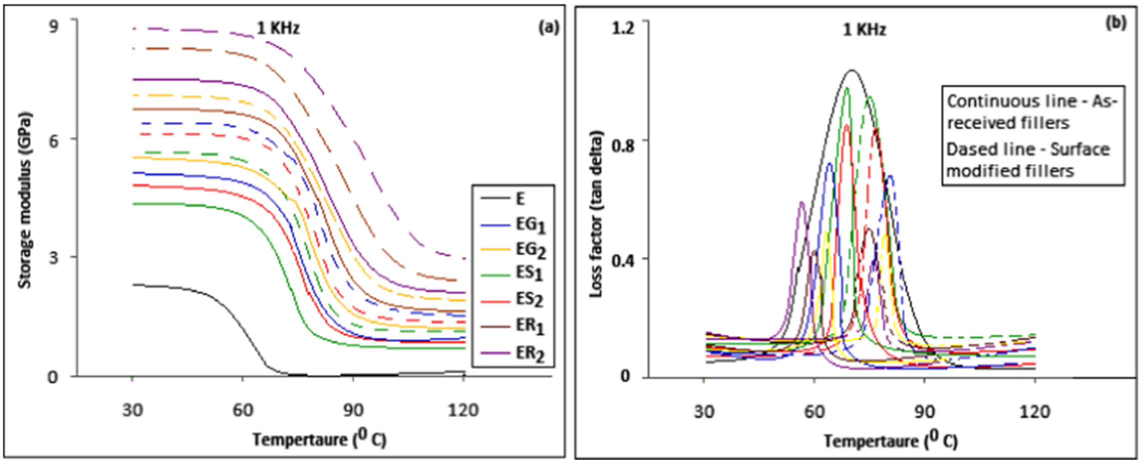

Fig. 4 Storage modulus vs. temperature of as-received natural filler-epoxy composite

was investigated via contact angle test in-accordance with ASTM D 2578 (sessile drop method, Holmarc India). In this a drop of water is placed on the composite and its contact angle with composite was calculated using a CMOS assisted high-performance aberration corrected imaging lens video capturing system.

4 Results and Discussions

4.1 Visco-Elastic Behavior (DMA)

Figure 4 illustrates the viscoleastic properties of (storage modulus and loss factor) various epoxy composites prepared with as-received and surface modified saw dust, groundnut shell and rice husk natural fillers. It is noted that the pure epoxy gives storage modulus and loss of 2.23 GPa and 0.85. The lesser storage modulus and higher loss factor is the cause of no stress absorbing elements in epoxy resin in continuous load and temperature. It is observed that the further additions of saw dust, groundnut shell and rice husk in the form of as-received and surface modified condition into epoxy improved the visco-elastic properties. A highest storage modulus of 5.2 GPa was seen for NF composite with 20 vol.% of rice husk. This improvement in storage modulus is the outcome of void filing of saw dust natural filler in the epoxy matrix. The natural fillers in voids arrest the mobility of secondary polymer molecular chains thus lot of energy is required to obtain the un-steady state of molecules. Thus higher storage modulus is observed for natural filler dispersed epoxy composite [23]. It is observed that the loss factor of ER_2 composite designation is very lower (0.74) than other composites. Since the addition of fillers improves the toughness of composite. The toughness improved composites never hold any residual stress whenever it gets stretched up. The smooth stress release reduces the chances of plastic strain in the composite material thus very lower loss factor is observed [24]. It is observed that when temperature and frequency increases the storage modulus increases and get decreases. This phenomenon is the reason of an increase of temperature the molecules get free volume due to the stretch of matrix molecules thus more energy could store in the composite. But when the temperature and frequency is beyond Tg and natural frequency the epoxy molecules start vibrate and move out of primary chain thus the stored energy is liberated. Thus decrement in storage modulus is observed during improvement of frequency and temperature [25].

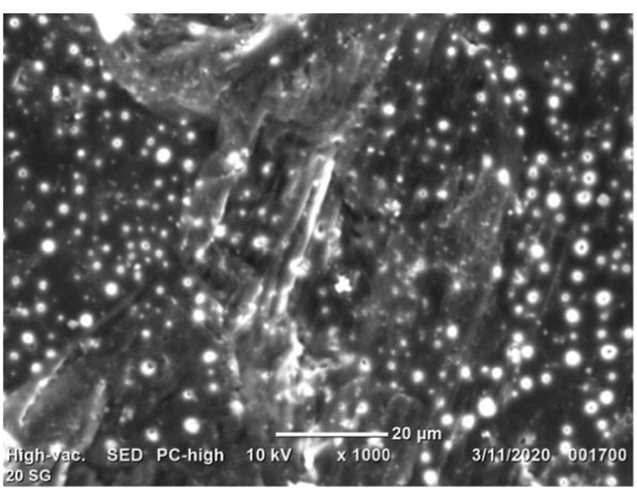

Fig. 5 SEM image of rice-husk natural filler in epoxy matrix

It is noted that the silane surface-modified natural fillers in epoxy composite gives improved results than composites with as-received fillers. The silane surface-modified rice husk filler gives higher storage modulus of 8.9 GPa and lower loss factor of 0.52 at 20 vol.%. This improvement is the cause of improved adhesion of silane surface-modified natural fillers with epoxy resin. The NH_2 functional group enhanced natural filler (rice husk) chemically bonded with epoxy resin during curing process. Thus, when temperature increases the filler bonded polymer molecular chains experience high resistance to rotate freely about the C-C polymer chains. Thus lot of energy is

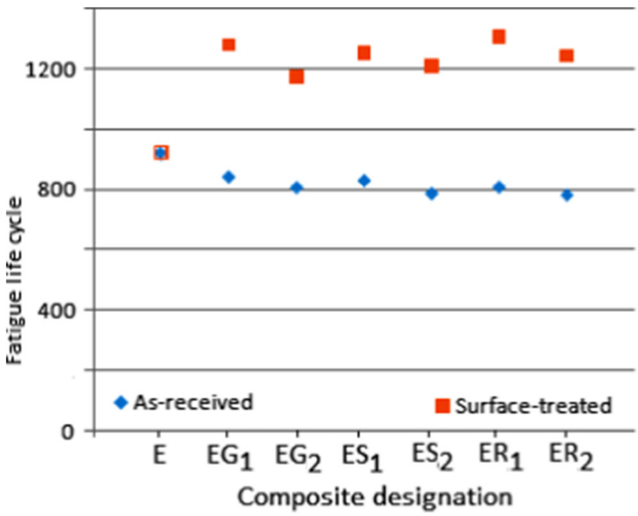

Fig. 6 Fatigue behavior of natural filler-epoxy composites

needed to activate them to rotate. Thus the silane surface treatment on natural fillers improved the visco-elastic properties irrespective of temperature and frequency [26]. Figure 5 shows the SEM image of silane surface-modified rice husk natural filler dispersed epoxy resin composite. The image reveals highly reacted phase of filler with matrix and possess uniform dispersion.

4.2 Fatigue Behavior

Figure 6 describe the tensile fatigue behavior of epoxy and its natural filler composites. It is noted that the neat epoxy produce very lower fatigue life cycle of 920 counts. This low fatigue count is the reason of high brittleness of epoxy resin. The neat epoxy does not hold any strengthening mechanism in it thus poor fatigue count is observed. It is observed that the further additions of as-received groundnut shell saw dust and rice husk in epoxy composite decreases the fatigue count. A decrement of 8, 12, 9, 14, 12 and 15% were noted for natural filler-epoxy composite designations EG_1, EG_2, ES_1, ES_2, ER_1 and ER_2 respectively. This reduction in fatigue strength is the reason of particle agglomeration and poor dispersion of fillers in matrix, which intern reduces the fatigue behavior. When the particles agglomerate in one place on the matrix, the stress intensity builds up and led the composite to deform plastically [27].

Whereas in surface-treated fillers dispersed epoxy composites are good in fatigue behavior. The improved fatigue life count of 28, 22, 27, 24, 30 and 37% were observed for composite designations EG_1, EG_2, ES_1, ES_2, ER_1 and ER_2. This improvement in fatigue strength is the reason of effective bonding of natural fillers in epoxy resin, which effectively share the load and reduces the pop-up of micro crack in matrix. The suppression of micro cracks in the matrix couldn't propagate further thus improved the fatigue behavior. Moreover, the silane bonded natural fillers in epoxy matrix reduces the acquirer of residual stresses when repeated load is applied. The toughness improved epoxy composite does not hold any plastic strain during repeated tensile fatigue load. Thus the residual stress free matrix offers more fatigue life counts [28]. It is observed that on comparing with all natural fillers the rice husk filled epoxy composite offered higher fatigue life cycle. This improved fatigue life count is the reason of intrinsic silica content of rice husk, which improves the adhesion behavior with matrix. Thus improved result in fatigue is observed for rice husk compare than groundnut shell and saw dust [29]. Figure 7 shows the scanning electron microscope image of rice husk natural filler dispersion in epoxy matrix. Figure 7a shows the as-received and 7b shows the silane-modified saw dust filler in matrix. It is observed that the as-received fillers show agglomeration whereas the silane modified fillers show uniform dispersion.

4.3 Water Absorption Behavior

Figure 8a-g shows the sessile drop image of pure epoxy resin and its natural filler dispersed epoxy composites. It is noted that the pure epoxy resin (Fig. 8a) gives very high contact angle of 112° which indicates very lower surface energy of epoxy resin and highest form of hydrophobic nature [30]. It is noted that the addition of saw dust, groundnut shell and rice husk into epoxy resin (Fig. 8b, d and f) gives reduction in contact angle, which

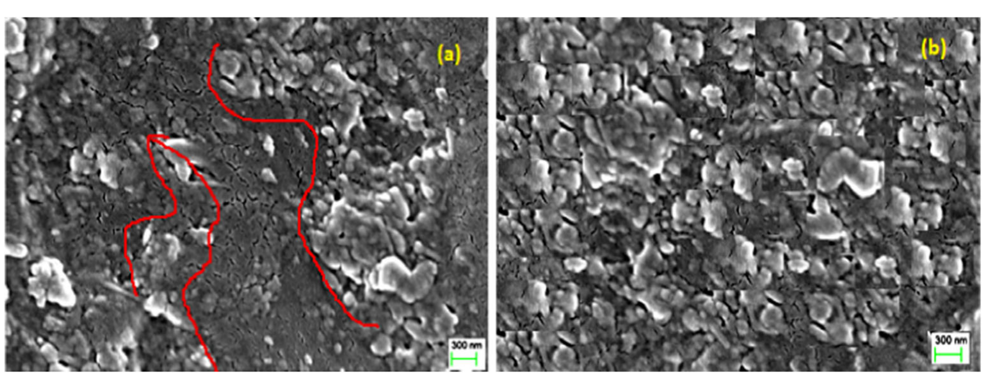

Fig. 7 SEM images of dispersion of fillers in epoxy matrix **a** as-received and **b** surface-modified condition

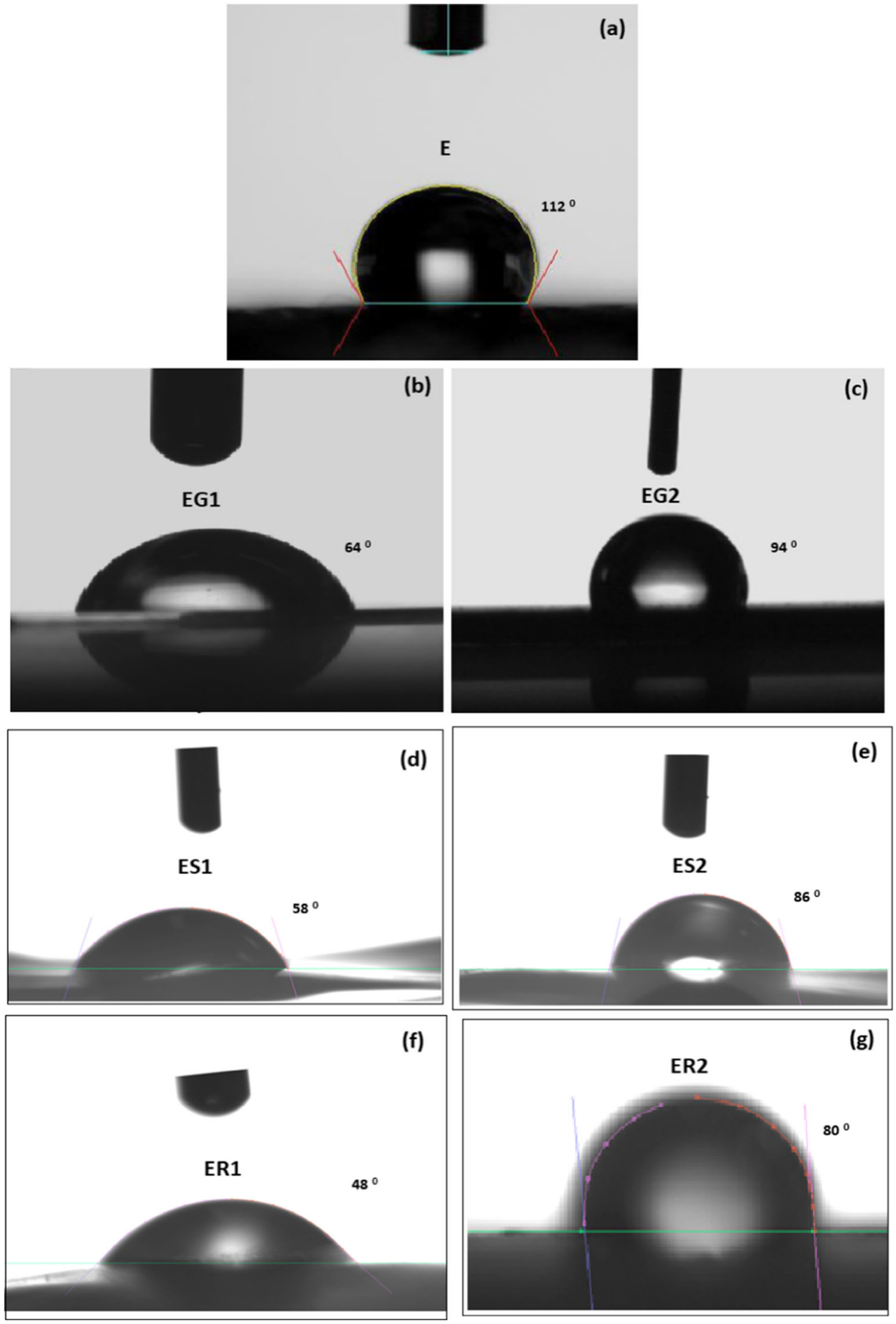

Fig. 8 Sessile drop contact angle images of natural filler-epoxy composites

indicates improved surface energy due to the addition of fillers. These fillers are highly borne to water molecules and absorbing them. It is noted specifically that the as-received natural fillers groundnut, saw dust and rice husk of 20 vol.% gives higher reduction in contact angle as 64^0, 58^0 and 48^0 respectively. This lack in water resistance is the cause of water borne natural fillers in epoxy resin composite.

In general, all the natural fillers have soft lignin and high porous water tender OH molecules in their surface. These OH molecules easily react with water and acquire more OH molecules, thus the composites lost their absorption resistance. Whereas in surface treated natural fillers the silane is covered the fillers as a coat and reduce the direct contact of soft lignin into water molecules. Thus the chances of water absorption in the composites are significantly reduced and retains higher contact angles of 94^0, 86^0 and 80^0 for 20 vol.% groundnut, saw dust and rice husk fillers in epoxy. These high contact angles revealed that the surface-modified natural filler in epoxy composite (Fig. 8c, e and g) posses low surface energy due to silane surface modification to acquire more OH molecules [31]. Thus the process of silane treatment on fillers retains high water absorption stability (resistance) during immersion rather than as-received natural fillers dispersed epoxy composites.

5 Conclusions

The as-received and silane surface treated natural fillers on epoxy resin and their effects on viscoelastic, fatigue and water absorption behaviour were studied. The effectiveness of silicon coupling grafting are summarized below.

a) The silane surface modification on fillers has been done via aqueous solution method and the same has been confirmed through FT-IR spectral analysis.
b) The as-received natural fillers on epoxy resin give lower values in storage modulus and loss factor. But the silane surface grafting on natural fillers produced improved viscoelastic properties like storage modulus and loss factor. The rice husk particle of 20 vol.% gives better result among three.
c) The fatigue results revealed that the silane surface-modified rice husk particles of 10 vol.% gives higher fatigue life cycle than other natural fillers whereas the as-received fillers in epoxy resin reduces the fatigue life cycle even less than pure epoxy resin. When the particle loading beyond 10 vol.% the fatigue life cycle counts are decreased.
d) The water absorption behavior of silane surface-treated natural filler-epoxy composites hold up the water uptake resistance as like as epoxy resin whereas the as-received natural fillers increased the moisture absorption.
e) Thus the study reveals that the silane surface modification produced desirable results in natural filler dispersed epoxy composites despite of as-received fillers produced dislike behaviors. Thus the silicon coupling grafting on natural fillers and the process is highly recommended to produce significantly good results in both property and application wise.

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