Vinyl silane functionalized rice husk ash reinforced unsaturated polyester nanocomposites

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Vinyl silane functionalized rice husk ash reinforced unsaturated polyester nanocomposites.

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An organic-inorganic hybrid nanocomposites based on vinyl silane functionalized rice husk ash reinforced unsaturated polyester resin were developed and characterized. Rice husk ash (RHA) is an agro product resulting from the incineration of rice husk and was used as silica source. The present work involves the functionalization of rice husk ash using vinyl triethoxysilane and is reinforced with UP resin to enhance the thermo mechanical properties of UP resin. The physio-chemical, thermal, mechanical and morphological properties of the composite samples were analysed using FT-IR, differential scanning calorimeter, electron microscope, thermo gravimetric analyzer, XRD and goniometer. The mechanical properties i.e., tensile strength, modulus, flexural strength, impact strength and hardness were studied and reported. The values of dielectric constant, contact angle were also studied and discussed. The vinyl silane functionalized rice husk ash reinforced UP resin composites possess better thermo mechanical, dielectric and surface properties than those of neat UP matrix.

Introduction

Polymeric materials used for the fabrication of advanced composite components should exhibit desirable physical and chemical properties including light weight in combination with high stiffness and strength, good dimensional stability, better thermal stability and chemical resistance. Composite materials are widely used in engineering and aerospace applications not only due to their physico-mechanical and chemical characteristics but also to the capability of changing their properties according to the required applications. Unsaturated polyester (UP) resins are widely used in composite industry because of their low price, ease of processing under various conditions, and excellent chemical, solvent and salt water resistance. Unsaturated polyester resins have been used in the development of high performance light weight fibre-reinforced composites, for manufacturing marine crafts, automotive parts etc., because of their good mechanical, thermal and electrical properties, outstanding adhesion to various substrates and light weight properties. However, unsaturated polyester has high cross-linking density and high shrinkage during cure, which affects its impact behaviour. To improve these properties number of reinforcing agents were used as chemical modifiers[1-6]. Modifications of unsaturated polyester resin with filler content improved its impact behaviour but failed to increase the thermo-mechanical properties of the resin in the high temperature region. Hence, an attempt was made in the present study to modify the commercially available polyester resin using vinyl functionalized rice husk ash as silica source by forming an inter-crosslinked network of unsaturated polyester using benzoyl peroxide as curing agent and to study their characteristic properties in order to assess their utility for high performance applications. Among the commonly used inorganic reinforcements, different kinds of silica (SiO_2) are recognized as effective flame retardant filler in thermoplastic and thermoset polymers. Rice husk is one of the main lignocellulosic materials of the agricultural industry and a natural source for silica production. Because of its very low cost and high silica content (85 wt %), RHA offers many potential advantages, e.g. abundant renewable source, high specific strength, stiffness, non abrasive and less hazardous to health. [7-13]

Hence in the present work, the varying percentage weight of functionalized RHA has been incorporated into UP resin with vinyl functionalization, in order to improve thermo mechanical properties and an addition to improvement in water repellent behaviour. The properties of resulting nanocomposites have been analysed and discussed.

Experimental

Materials

Rice husk ash was used as the silica source and was synthesized as per the reported procedure[13]. VTES, ethanol and benzoyl peroxide were obtained from SRL (India) and were used as received. The matrix resin used in the present study was unsaturated polyester resin received from Ciba-Geigy Ltd.

Preparation of Rice husk ash (RHA)

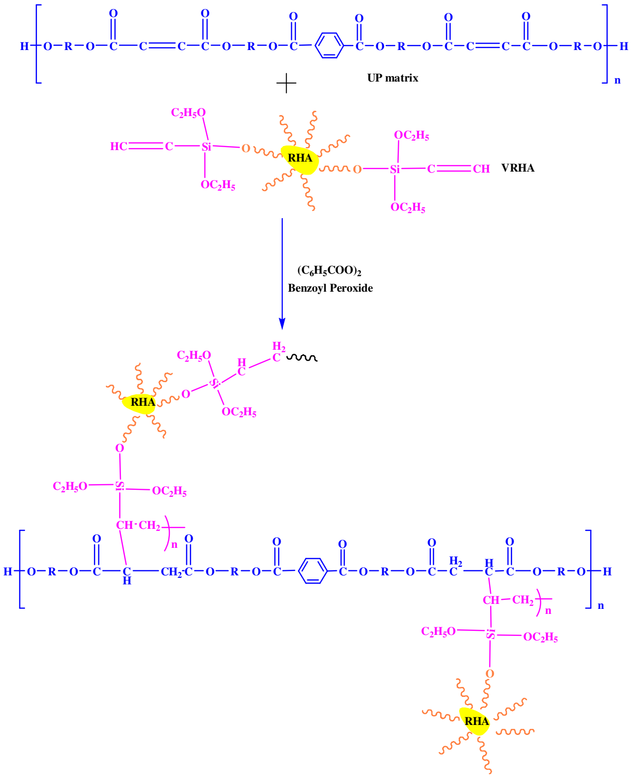
Rice husk ash is a solid powder obtained after burning of rice husk followed by washing with distilled water and dried in an oven at about 60 ˚C for 2h. Then washed with acetone and hydrochloric acid (2 M) to remove dirt and other contaminants present in them and then dried in oven at about 60 ˚C for 4h. Samples of rice husk were heated at 500 ˚C for 5h at a heating rate of 5 °C per minute in a muffle furnace, to obtain rice husk ash. The removal of surface impurities enhances the surface roughness thus generating more hydroxyl groups and other reactive functional groups on the surface.

Surface functionalisation of rice husk ash (VRHA)

To form a uniform dispersion of SiO_2 derived from rice husk ash into the matrix, VTES was used as a coupling agent to modify the surface of rice husk ash. The –Si (O $(C_2H_5)_3$) in VTES can be easily hydrolysed and chemically bound with the SiO_2 group in the rice husk ash. The functionalization of VTES with rice husk ash is as follows. 50 mM of VTES was mixed with 95% absolute ethanol and 5% deionised water under sonication for 15 min. The pH value of the solvent was initially adjusted to 4.5, using acetic acid, and subsequently sonicated for 1h in order to get the complete hydrolysis of VTES[14, 15]. 10g of treated rice husk ash was added to the mixture, which was then sonicated for 2h. The mixture was then refluxed for 24h, and centrifuged with the addition of water, followed by ethanol and hexane to remove the excess of VTES. The rice husk ash thus functionalized was further dried in a hot air oven at 100 ˚C for 24h, in order to remove the moisture.

Preparation of VRHA-UP nanocomposites

100 parts of unsaturated polyester resin and varying weight percentages of vinyl silane functionalised rice husk ash (0.5, 1.0 and 1. 5 wt %) were separately blended together by effective agitation using a mechanical stirrer for 24h until a homogeneous blend was obtained. Then, the stoichiometric amount of benzoyl peroxide corresponding to the UP resin equivalent was added. Then the product was subjected to vacuum to remove the trapped air and then cast, and cured at 120 ˚C for 2h. The castings were then post-cured at 160 ˚C for 3h and finally removed from the mould for characterization (Scheme 1).



Scheme 1 Schematic representation of VRHA-UP nanocomposites

Characterization

Fourier transform infrared (FT-IR) spectra were recorded on a Perkin Elmer 6X FT-IR spectrometer. The glass transition temperature (Tg) of the samples was determined, using DSC 200 PC differential scanning calorimeter (DSC) (Netzsch Gerateban GmbH). Thermogravimetric analysis (TGA) was carried out, using the DSTA 409 PC analyzer (Netzsch Gerateban GmbH). The tensile (stress–strain) properties were determined, using INSTRON (Model 6025 UK) as per ASTM D 3039. The flexural properties were measured by the INSTRON (Model 6025 UK) as per ASTM D 790. The un-notched Izod impact strength of each sample was studied as per ASTM D 256. The water absorption behaviour of the samples was tested as per ASTM D 570. The percentage of water absorbed by the specimen was calculated, using the following equation:

$$\% \text{ Water absorption} = (w_2 - w_1) \times 100 / w_1 \qquad (1)$$

where w_1 is the initial weight of the sample and w_2 is the weight of the sample after immersion in distilled water for 48h at 30 ˚C. The dielectric studies of the neat UP matrix and VRHA-UP composites were determined with the help of an impedance analyser. Contact angle measurements were carried out using 210 a Rame-hart Inc. goniometer (Succasunna, NJ, USA) with 5µl of deionised water and diiodomethane (DIM).

X-ray diffraction patterns were recorded at room temperature, by monitoring the diffraction angle 2θ from 10 to 70° as the standard, on a Rich Seifert (Model 3000) X-ray powder diffractometer. The surface morphology of the fractured surface of the samples was examined, using a scanning electron microscope (SEM; JEOL JSM Model 6360). A JEOL JEM-3010 analytical transmission electron microscope, operating at 80 kV with a measured point-to-point resolution of 0.23 nm, was used to characterize the phase morphology of the developed nanocomposites. TEM samples were prepared by dispersing composites under sonication in ethanol and mounted on carbon-coated Cu TEM grids and dried for 1h at 70 °C to form a film of < 100 nm.

Results and Discussion

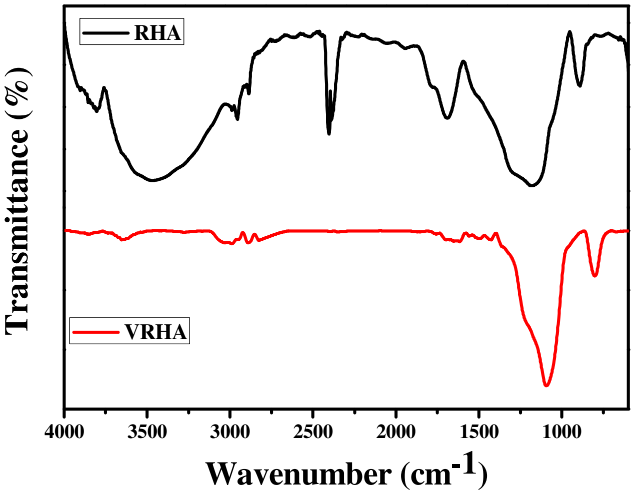


Fig. 1. FT IR Spectra of RHA and VRHA

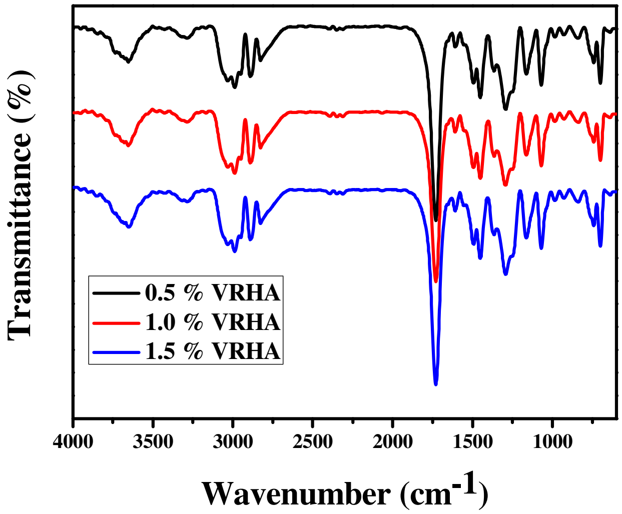


Fig. 2. FT IR Spectra of VRHA-UP nanocomposites

Fig.1 shows the FT-IR spectra of RHA and VRHA. The disappearance of peak at 3435 cm $^{-1}$ in VRHA is attributed to the coupling of vinyl triethoxy silane with the surface hydroxyl group of rice husk ash. Fig. 2 shows the FT-IR spectra of developed VRHA-UP nanocomposites. The appearance of peak at 1070 cm^{-1} is attributed to the cross linked network of vinyl terminated RHA and unsaturated polyester matrix[16]. The cross linking was achieved via copolymerisation reaction of vinyl group of RHA with the unsaturated bond in the UP matrix to form the resulted VRHA-UP nanocomposites[17].

The appearance of peaks at 1162 cm^{-1} and 747 cm^{-1} confirm the presence of Si-O-Si network in the composites. The peak appeared at 2948 cm^{-1} indicates the presence of an aliphatic CH_2 group, and confirms the intrusion of VRHA into the UP resin. The carbonyl group of the polyester resin and the siloxane linkage in the VRHA binds strongly which was clearly observed by the band occurred at 1728 cm^{-1} [18,19] .

The mechanical properties of varying weight percentages of VRHA reinforced UP nanocomposites are analysed and are presented in Table 1. The value of tensile strength of neat UP matrix is 32.0 MPa. The introduction of 0.5, 1.0 and 1.5 wt % VRHA into the UP matrix increased the values of tensile strength by 23.8, 39.1 and 69.7 %, respectively. The rigid silica particles of VRHA produce a stress concentration effect which initiates the surrounding resin to produce microgaps to absorb the deformation work when load is applied. The enhanced surface area of vinyl functionalised rice husk ash influences the particles to disperse homogeneously in the composites (Fig. 6 SEM micrograph). Hence, the adhesive forces operate between the filler and the matrix is strong, and thus the strength of the matrix is improved. The strength of the composites depends on the interaction occurring between the interfaces. The synthesized VRHA particles are in nanometer range, with size of 100 nm (Fig. 7 TEM image). This creates the atomic position gradient between the particles, which leads to an improvement of the tensile strength of the material.

Table 1 Mechanical Properties of neat UP and VRHA-UP nanocomposites

Sample	Tensile strength (Mpa)	Tensile Modulus (Mpa)	Flexural strength (Mpa)	Impact strength (Jm^{-1})	Hardness (HV1.2)
Neat UP	32.0	1002	40.1	23.3	80
0.5 % VRHA-UP	42.4	2080	66.6	78.13	83.5
1.0 % VRHA-UP	51.0	2147	72.8	93.75	89.2
1.5 % VRHA-UP	59.8	2348	80.75	109.38	96.7

The flexural stiffness is a criterion of measuring deformability. The flexural strength of neat UP matrix is 40.1 MPa, similar filler loadings of VRHA (0.5, 1.0 and 1.5 wt %) increased the flexural strength to 66.5, 82.4 and 92.3 % respectively. The silica groups present in the natural cellulose rice husk ash and the initiator benzoyl peroxide prevent and inactivate the development of fissures in the resin matrix which readily increased the values of tensile strength and flexural strength of the VRHA-UP composites[20].

As increase in the percentage weight content of VRHA in polyester matrix, it increased the values of both impact strength and the elongation at break continuously up to a maximum amount of VRHA is 1.5 wt % (2348 MPa tensile modulus and 109.4 J/m^2 impact strength) and then starts decreases gradually. The increment of reinforcement (VRHA) into the UP matrix starts to form agglomeration of particles along with the matrix and decreases the energy absorption character with the reduced values of impact strength. Agglomeration of particles actually decreased the distance between the particles which is near enough to affect each other parameters and results in the plastic deformation of the matrix resin.[21]

The tensile modulus is one of the important parameters characterizing the rigidity of a material. The strength, stiffness and fracture toughness of the VRHA reinforced UP composites are higher than those of neat UP matrix. The high specific surface area of VRHA provides a large contact area between the reinforcement and the matrix which consequently improves the ability of a material to resist fracture under stress applied at high speed. When materials are impacted they can produce more microgaps and absorb more amount of impact energy. In addition, the strengthening and toughening behaviour of the composites, are influenced by the interaction between the SiO_2 of VRHA and the unsaturated polyester matrix and can be ascertained on the basis of the glass transition temperature (Tg) obtained from differential scanning calorimetry (DSC). The largest aspect of surface area to volume ratio of SiO_2 has influenced the enhancement of tensile strength, flexural strength and impact strength, tensile modulus and hardness of VRHA-UP than those of neat UP matrix[22,23]. It was noticed that the value of hardness is increased with increasing weight percentages loading of VRHA into the UP matrix. Furthermore, the maximum value of hardness was found when loading of VRHA was at 1.5 wt %.

Table 2 Thermal, Water absorption and Dielectric properties of VRHA-UP

Samples	Tg (°C)	Degradation Temperature (°C) 40 %	60 %	80 %	Water absorption (%)	Dielectric constant (at 1MHz)
Neat UP	75.3	348.0	370	392.0	0.240	4.85
0.5% VRHA-UP	93.6	374.5	391	405.8	0.126	3.96
1.0% VRHA-UP	109.7	387.5	400.6	424.1	0.083	3.47
1.5% VRHA-UP	118.4	458.0	479.0	494.0	0.028	2.98

nanocomposites

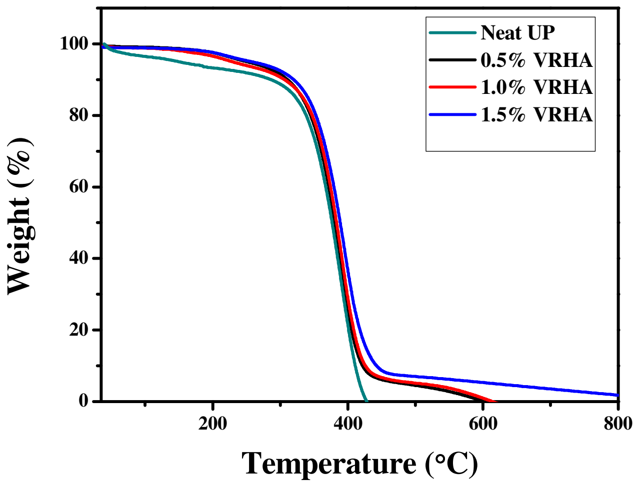


Fig. 3. TGA profile of VRHA-UP nanocomposites

The curing behaviour of neat UP matrix and the VRHA reinforced composites were characterized by DSC analysis and the results are presented in Table 2. The exothermic peak obtained at 200 ^{o}C generated from the copolymerization between vinyl group of VRHA and the active double bonds of the unsaturated polyester. Compared to that of neat UP matrix, the reinforced UP composites exhibit a wide processing window with both low onset temperature and exothermic peak, suggesting that the processing properties of UP matrix can be efficiently improved using the reinforcements. This is probably due to the formation of strong covalent bond between the vinyl moiety of VRHA and UP matrix. It also helps to restrict the chain mobility and consequently resulting in an increase in the value of T_g for the composite samples. Chain restriction is unlikely to play a significant role with regard to the value of T_g for the VRHA-UP composites. If an enhanced bonding between the filler and the matrix existed, a stronger crosslink system would be formed. Consequently crosslink density is a significant parameter which helps in characterizing the reinforcing extent of filler on matrix[24]. The thermal stability of neat UP matrix and the VRHA-UP composites were analysed by TGA and are presented in Table 2 and in Fig.3.

Neat UP matrix degrades completely at 400 ^{o}C whereas the VRHA reinforced composites extends the degradation up to 800 ^{o}C. The thermal stability of VRHA-UP composites consistently increased with increasing VRHA content due to the presence of high SiO_2 in the rice husk ash. It was also predicted that the rough surface of rice husk ash acts as protective barrier during thermal degradation of VRHA-UP composites, which slow down the rate of degradation via a diffusion process. This indicate that the uniform dispersion of VRHA in the composites which in turn contributes to an enhanced thermal stability[25].

When a liquid drop is brought onto the surface of a solid, the contact of liquid reaches an equilibrium condition and the liquid drop is in a static state. The angle with which the liquid subtends the solid is known as the contact angle (θ) as shown in Fig. 4 and the values are presented in Table 3.

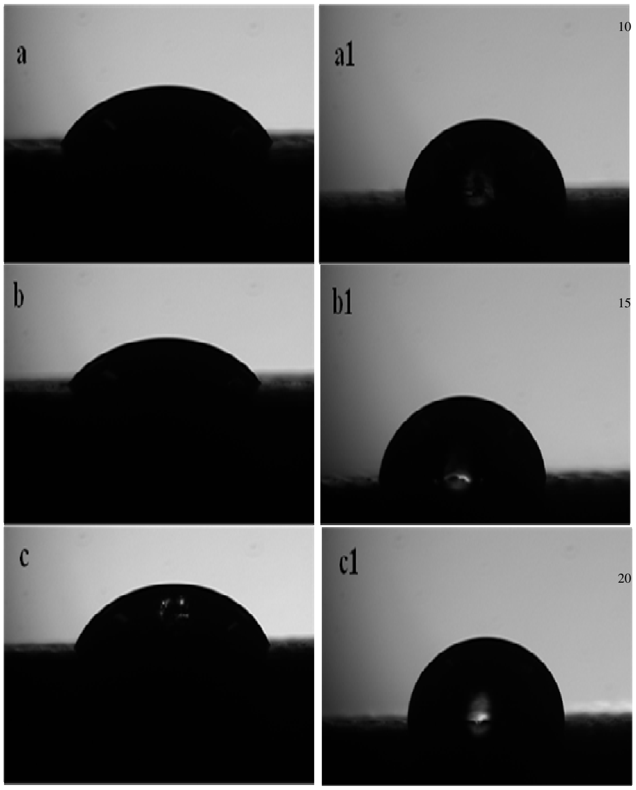


Fig. 4. Contact angle of VRHA-UP composites (a) 0.5 wt%, (b) 1.0 wt% and (c) 1.5 wt% using water as liquid and (a1) 0.5 wt%, ((b1) 1.0 wt%, and (c1) 1.5 wt% using DIM as a liquid.

Table 2 Contact angle and Surface free energy of VRHA-UP nanocomposites

Sample	Contact angle (θ)		Surface energy		J/m
	Water	Diiodo methane	γ^d	γ^p	Γ
0.5 % VRHA-UP	85	50.3	41.9	5.5	36.5
1.0 % VRHA-UP	91.2	60.3	37.1	3.0	34.1
1.5 % VRHA-UP	95.6	68.4	30.7	2.3	28.4

The reason was assumed to be the excess number of non-polar groups on the surface and the increase in the roughness of the surface. The effect of surface roughness brought about by the silica moiety in the rice husk ash reduces the wettability and hydrophilic behaviour of matrix. However, the presence of attractive hydrogen bonding between the filler backbone and the matrix changes the free energy of mixing and leads to an excellent dispersion and interfacial contact. A number of factors influence the degree of hydrogen bonding, including steric accessibility, spacing of hydrogen bonding functional groups and the ability of the polymer to hydrogen bond with its neighbours[26]. Consequently, -Si-O-Si linkages in the vinyl functionalised rice husk ash can be used to reduce the surface energy and enhance the substrate surface roughness and make the material surface hydrophobic.

XRD patterns for rice husk ash, VRHA reinforced UP resin composites are shown in Fig. 5 and it confirms the amorphous nature of the developed composites. The intense peak at 30 degree of RHA shifted to 19 degree and appeared as broad amorphous peak adjacent to diffraction pattern of UP matrix at 11 degree. This is due to the introduction of VRHA into the UP matrix which forms a strong covalent bonding interaction between the reinforcement and matrix through vinyl functionalization. After the introduction of UP matrix, the XRD data gives clear evidence that there is no intense peak observed, and the peak occurs only at 11 degree 2θ angle which confirms the exfoliation of VRHA groups into the matrix successfully without any deviation[27].

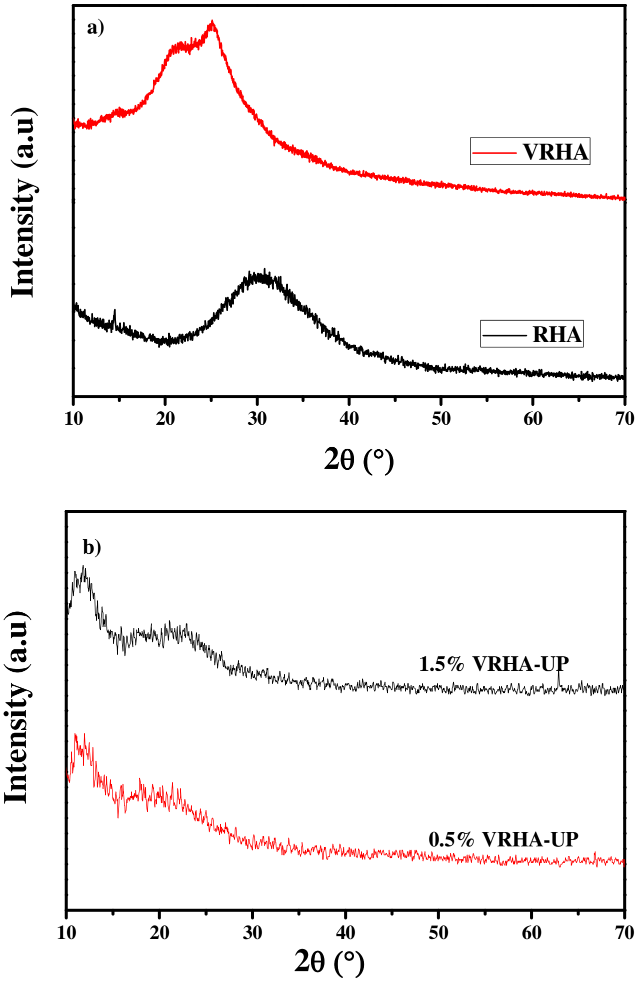


Fig. 5. XRD diffractogram of a) neat RHA, VRHA and b) 0.5% and 1.5% VRHA- UP nanocomposites

The morphology of VRHA-UP composites developed with vinyl functionalised rice husk ash and UP resin via benzoylperoxide initiator was investigated by SEM and the data are presented in Fig. 6(a–f). SEM photographs of fractured surfaces of the unmodified epoxy systems 6(a) indicated a smooth, glassy and homogeneous microstructure without any plastic deformation. There is no separate phases exist even after the incorporation of filler into the matrix, this clearly indicates the homogenous dispersion of VRHA particles. From the values of contact angle, mechanical and thermal properties it was found that the concentration of rice husk ash had a significant influence on the morphology of the resultant composites. From the fractured surface images (Fig.6 e & f) of 1.0 wt % of VRHA-UP, it is clearly observed that there is no agglomeration of rice husk ash particles and no possibility for pulling out of the particles from the matrix. This data support the higher values of tensile strength and Tg when compared to those of neat UP matrix[28].

VRHA reinforced composites were prepared by tethering VRHA molecules to the side chains of UP matrix using benzoyl peroxide initiator. The value of dielectric constant of the resultant VRHA-UP composites is lower than that of neat UP matrix due to the increased free volume and less polar nature of silica present in the rice husk ash**.** Another possible contribution to the lower dielectric constant arises from the fact that the alkyl groups on UP matrix are partly replaced by VRHA molecules, leading to a decrease in the polarity of the composites and also reduction in water absorption behaviour. Thus reduction in water absorption behaviour is attributed by the presence of –Si–O–Si– linkage present in the VRHA[29].

The morphology of VRHA-UP nanocomposites was analysed by TEM technique and the image are shown in the Fig.7. From TEM analysis, it was clearly seen that the VRHA particles were dispersed into the UP matrix completely. These results indicate that the VRHA-UP nanocomposites possess good interfacial interaction through the chemical bonding with the help of the initiator benzoylperoxide. The nanometer level dispersion of VRHA particles in the UP matrix influences on the thermal properties of the polymer.

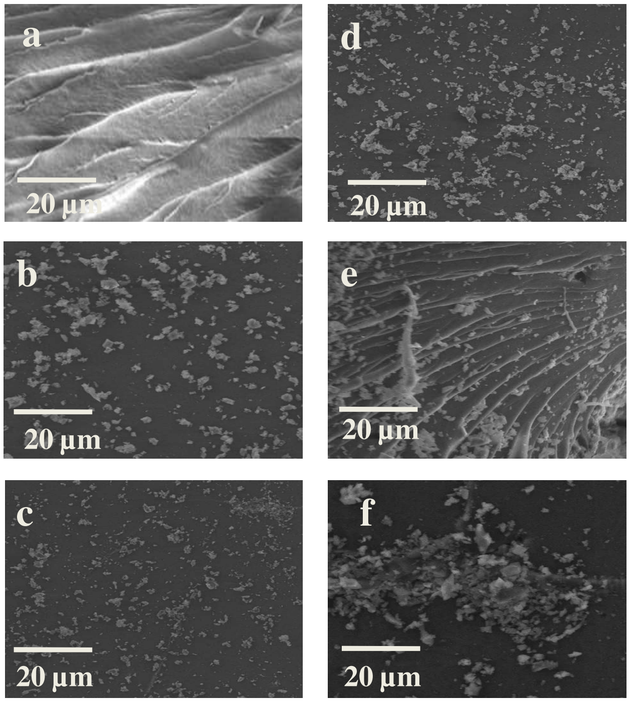


Fig. 6. Scanning electron micrograph of (a) neat UP, (b) 0.5, (c) 1.0 and (d) 1.5 wt % (e) and (f) Fractured surface of 1.0 wt. % VRHA-UP composites.

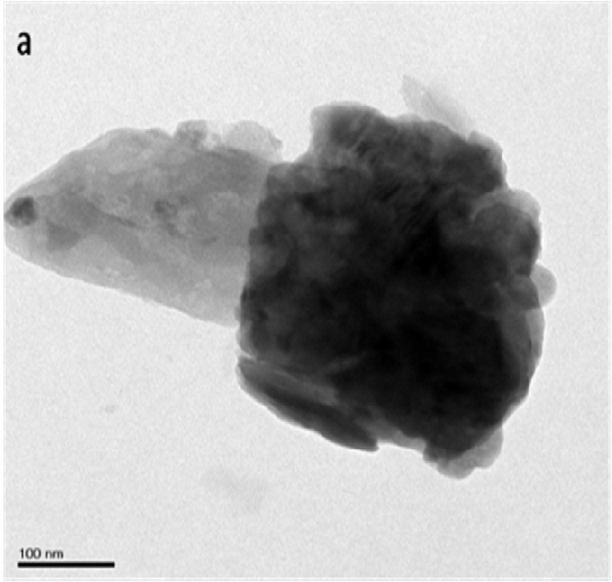


Fig. 7. TEM image of 1.0 %VRHA -UP composites

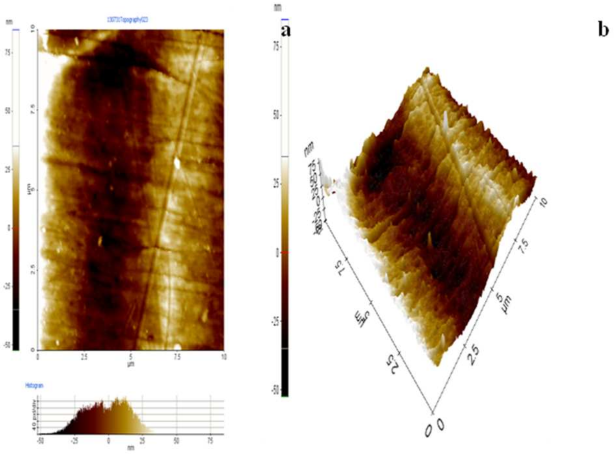


Fig. 8. AFM image of (a) 2D image and (b) 3 D image of 1.0 %VRHA -UP composites

The compatibility of organic polymer and the inorganic reinforcement will dominates its effect on thermal and mechanical properties. The AFM image (Fig 8) confirms the existence of strong covalent bonding between the polymer matrix and functionalised reinforcement. The AFM image of 1.0 wt % VRHA-UP nanocomposites shows a smooth and uniform morphology. From the image it was observed that the nanocomposites have a molecular level dispersion of organic and inorganic networks, and the silica particles are distributed uniformly at the nanoscale in the organic phase. The structural uniformity was also confirmed by SEM analysis, which shows no cracks or flaws were seen in the SEM micrograph.

Conclusions

The RHA obtained from natural source rice husk was functionalized with VTES and was used as reinforcement for UP matrix cured with benzoyl peroxide to obtain nanocomposites. VRHA reinforced composites exhibits higher thermal and mechanical properties than those of neat UP matrix. The values of higher contact angles possessed by the composites support the hydrophobicity which can be used as water repellent coatings. SEM, XRD, TEM and AFM studies further confirm the homogenous dispersion of VRHA reinforcements into the matrix which attributes to the improvement of properties.

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Notes and references

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