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**Abstract**

The aim of the present communication is to study the effects of the addition of the untreated and treated coconut shell particles on the mechanical properties of vinyl ester composites. Composite plates were prepared by hand lay-up technique with the six different content (5, 15, 25, 35, 45, and 55 wt%) of the coconut shell particles. Mechanical properties such as, tensile, flexural, and impact, of the coconut shell particle/vinyl ester composites were determined and compared at both the untreated and treated conditions based on the content of the coconut shell particles. The results revealed that the mechanical properties of the composites have increased with the addition of the coconut shell particles up to 35 wt% and then dropped at both the conditions. The treated composites show the high level of mechanical property values compared to the untreated composites. The optimum particle content to get the best combination of mechanical properties is 35 wt% in this composite. The fractographic studies were carried out to understand the failure of the composites. To understand the chemical compatibility between the particle and the matrix in the composite at both the untreated and treated conditions, the Pukanszky's model was used and correlated with the experimental results.

1. Introduction

Recently, the fibres and particles derived from the bio resources have been extensively used as potential reinforcements into polymer matrices instead of the commonly used man-made synthetic reinforcements like carbon, glass or aramid owing to their low weight and cost, abundant availability, high specific strength values, and biodegradability [1]. The natural cellulose fillers can be obtained from both the agricultural and forestry resources and they have been gaining acceptance in commodity polymer (thermoset and thermoplastic) [2, 3] from the last decades. Moreover, the waste materials derived from the agricultural resources are serving as suitable fillers for polymer resins in the past few years, with the benefit of improved performances and lower cost. The applicability of natural cellulose fillers in polymer matrix composites has been studied and reported successfully by many researchers [4]. The particles derived from the natural resources like plants, fruits or some other living species, are reinforced with the polymer resin matrix to enhance the properties of the polymer composites. Several works have been done on various bio particles reinforced polymer composites [5–7]. In order to diminish the negative effects on the environment created by the plastic structures, the use of agro crop residues as potential fillers was studied by several researchers [8, 9]. Nowadays, composites reinforced with natural cellulose fillers are in extensive demand because of their biodegradable and renewable nature. The usage of these fillers has reduced the use of high cost and synthetic fillers in several secondary application fields. The various natural bio fillers such as wood flour, groundnut shell particle, rice husk, wheat husk, coconut husk, etc are bio based residues from the agricultural resources and are available in plenty in countries like India, Indonesia, Malaysia, Thailand and Sri Lanka. Among various bio particles, the coconut shell particles are showing potential to be used as reinforcing agent in polymer matrix composites because they are relatively inexpensive and commercially and also easily available in various forms at the coconut industries.

Coconut shell is non-food part obtained from the coconut tree and one of the hard lignocellulosic agro wastes. Around the world, 90% of coconut shells were disposed as waste on the land and either burned in the open air. The coconut management issues are directed worldwide towards the utilization, storage and disposal for economic and environmental related issues. Many researchers have been made several attempts to develop and characterize the polymer matrix composites using bio natural coconut shell particles as reinforcement: CSP-Epoxy [10]; CSP and Barley Husk—Polypropylene [11]; CSP-Polyester [12]; CSP-Low Density Polyethylene [13]; CSP-Epoxy [14]; CSP-Epoxy [15]; CSP-Epoxy [16].

Even though natural cellulose fillers exhibit several venerable advantages over mineral fillers (mica, talc and calcium carbonate, etc) they have some disadvantages like incompatibility with the non-polar polymer matrix. The presence of strong polarized hydroxyl groups on the surface of natural cellulose fillers reduces the interfacial bonding with the polymer resin matrix when the hydrogen bonds have a tendency to prevent the wetting of the filler surfaces (lack of interfacial adhesion). Due to this, the polymer composites reinforced with the natural cellulose fillers show poor mechanical properties by the lack of interfacial adhesion. The better the level of interfacial adhesion between the fillers and the matrix in the polymer matrix composites can be enhanced by the suitable surface modification process. Presently, several methods of surface modification (alkaline treatment, silane treatment, esterification, compatibilizers and the other chemical compound) are used to improve the strength of the interfacial adhesion in the natural fillers reinforced polymer composites [17–20]. Based on the above literature, a very few works are carried out on the combination of the Coconut Shell Particles (CSPs) and the Vinyl Ester (VE) resin matrix. Therefore, in the present study, the CSPs were reinforced with the VE resin matrix to evaluate their mechanical properties at six different weight percentages. The effects of alkali treatment on the mechanical properties of the CSP/VE composite were also examined based the weight percentages of the CSPs. Composites were prepared by a simple hand lay-up technique and their mechanical properties, such as tensile, flexural and impact, were evaluated based on the content of the CSPs. The fracture surfaces of the composite specimens are examined by Scanning Electron Microscope (SEM)

2. Experimental details

2.1. Materials

The CSPs are hard particles compared to the other bio particles like groundnut shell, cashew nut and coir pith particles. Therefore, the CSPs were selected as reinforcing material as received in the present study. The locally disposed coconut shells were collected from the Devendrapuram Village, Rajapalayam, Tamilnadu, India, and then dried in sunlight for a week. After that, the refined coconut shells were crushed to produce coconut shell particles and simultaneously sieved to obtain required particle size of 100–200 microns. The VE resin with the hardener, accelerator, and promoter was used as resin matrix. Methyl ethyl ketone peroxide, Cobalt naphthenate and DI-Methylacetamide were used as the hardener, accelerator, and promoter. All chemical agents and materials were procured from GVR Enterprise, Madurai, Tamilnadu, India.

2.2. Reasons for the chemical modification of the CSPs

Generally, in natural bio fillers reinforced polymer composites, the fillers and the polymer matrix interface adhesion is crucial to the effective stress transfer between them. The hydrophilic nature of natural fillers leads to a heterogeneous system when they are reinforced with the polymer resin matrix and their properties are inferior due to poor interfacial adhesion between the fillers and the polymer matrix. Chemical modification (alkali, bleaching, acetylation, grafting of monomer, etc) of natural bio fillers (fibers and particles) helps to make it less hydrophilic and to improve the bonding between the fillers and the polymer matrix. Among various chemical modifications, the alkali treatment, also called mercerization, is one of the important methods to produce high quality fillers (fibers and particles). Alkali treatment reduces the filler's size and improves the interfacial bonding due to the removal of natural and artificial impurities. By reducing the filler's size, the aspect ratio of the fillers is simultaneously increased and also a rough surface topography is developed. They offer better interfacial adhesion between the fillers and the matrix and an increase in physical and also mechanical properties. Alkali treatment creates the number of possible reaction sites into the fillers and allows better filler wetting with the polymer matrix [21].

2.3. Alkali treatment of the CSPs

Alkali treatment of coconut shell particles was done by soaking in a 10% of solution of alkali agents for 1 h in a stainless beaker. Coconut shell particles were added into the stainless beaker and stirred well in mechanically. This process was done in room temperature for 1 h and followed by washing thoroughly with de-ionized water to remove the excess of alkali agents sticking on the particles. The treated particles were dried at room

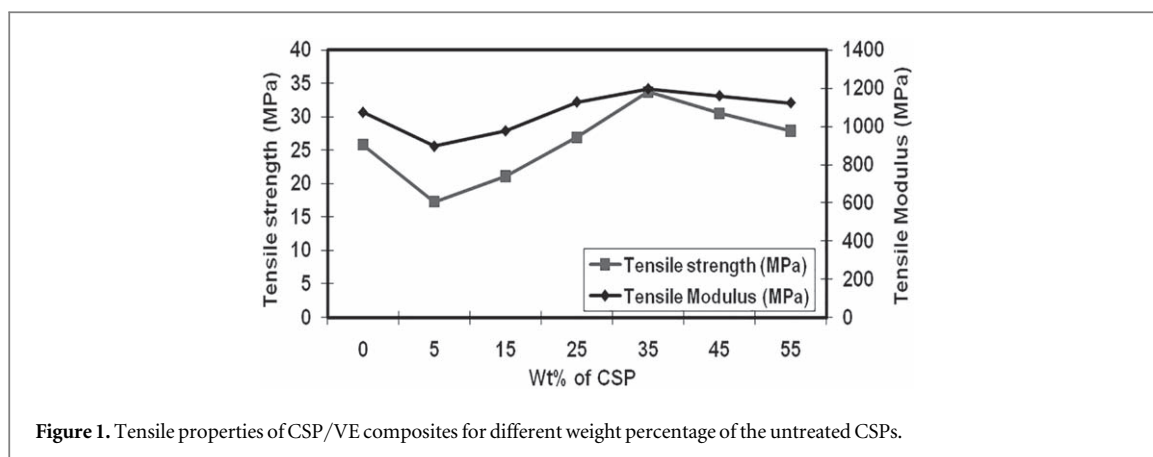


Figure 1. Tensile properties of CSP/VE composites for different weight percentage of the untreated CSPs.

temperature for 12 h. Alkali treatment has removed the natural and artificial impurities and also changed the surface morphology of the particles [22].

2.4. Preparation of composite plates

Coconut shell particles were reinforced properly with the vinyl ester resin matrix to prepare the particle reinforced vinyl ester composites. Composite plates were fabricated by a simple hand lay-up technique with a mould size of $150 \times 150 \times 3$ mm for six different particle loading (5, 15, 25, 35, 45, and 55 wt%). Prior to the process, a releasing agent (silicon spray) was applied for the easy removal of composite plates from the mould box after curing. The particles were mixed with the resin matrix by a mechanical stirrer at a speed of 800 rpm and poured into the mould box. Then, the mould was allowed to cure at room temperature for 24 h. After that, the cured composite plates were removed from the mould and cut with electrical cutter as composite specimens according to ASTM standards for performance studies.

2.5. Testing of composite specimen

Tensile tests were performed on a computerized FIE universal testing machine according to ASTM D638-10 at a crosshead speed of 2 mm/min. The flexural, three point bend, tests are conducted on the same universal testing machine in accordance with ASTM D790-10 at a crosshead speed of 2 mm/min. Impact tests were carried out on the Izod impact tester in accordance with ISO 180.

3. Results and discussion

3.1. Mechanical properties of the untreated CSP/VE composites

3.1.1. Tensile properties

Figure 1 depicts the variation of the tensile properties of the CSP/VE composites with different weight percentage of CSPs. It is clearly observed that the initial addition of CSPs had not contributed to the improvement of tensile properties of composites. The initial addition of the CSPs (5 and 15 wt%) gives the lower level of tensile properties compared to the neat resin specimen. Composite reaches the tensile properties of the neat resin specimen at 25 wt%. It may be due to the distribution of insufficient weight percentage of particles. The distribution of insufficient particles within the composite cannot contribute for better transfer of applied load from the polymer resin matrix to the particles. From the figure 1, it is also identified that both the tensile strength and modulus are improving with an increase in the content of CSPs up to 35 wt% and then dropped. The improvement in tensile strength was 30.62% at 35wt% composite when compared with the neat resin sample. This shows an effective and uniform stress transfer within the composite after the incorporation of 35 wt% of CSPs into the VE resin matrix. Beyond the 35 wt% of CSPs, composites reach the brittle behaviour due to the poor wettability between the CSPs and the VE resin matrix [23]. Because of the deformation of the polymer matrix is reduced due to the incorporation of higher weight percentage of particles (insufficient resin matrix). It is also observed that the composite with 25 wt% CSP content exhibits slightly less value of tensile strength and modulus as compared to the 55 wt% composite.

3.1.2. Flexural properties

The effect of the addition of the CSPs on flexural properties of the CSP/VE composites is illustrated in figure 2. Here also observed that the flexural properties of composite decreased at the initial addition of CSP's content (5 and 15 wt%) but further addition of CSP's content (25 wt%) in the VE resin matrix increase the flexural

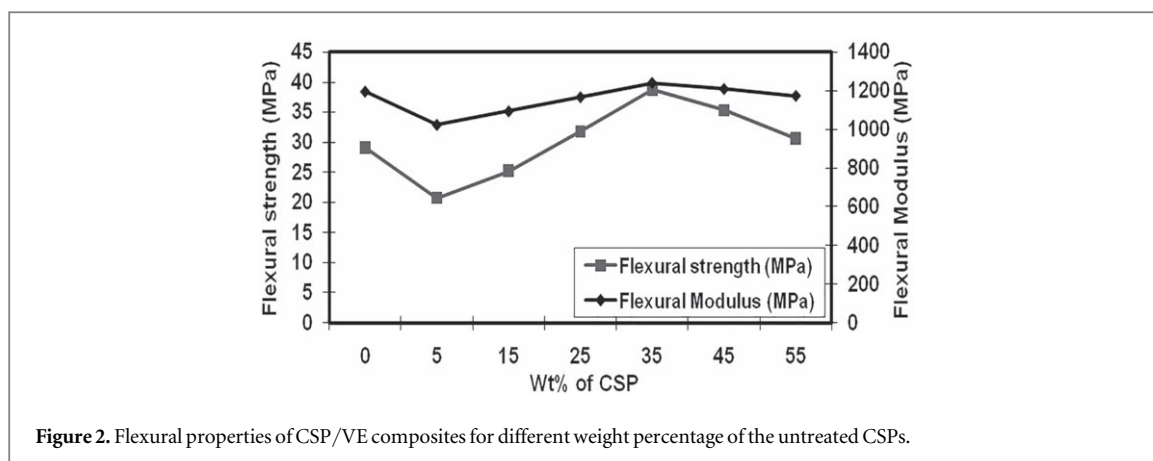


Figure 2. Flexural properties of CSP/VE composites for different weight percentage of the untreated CSPs.

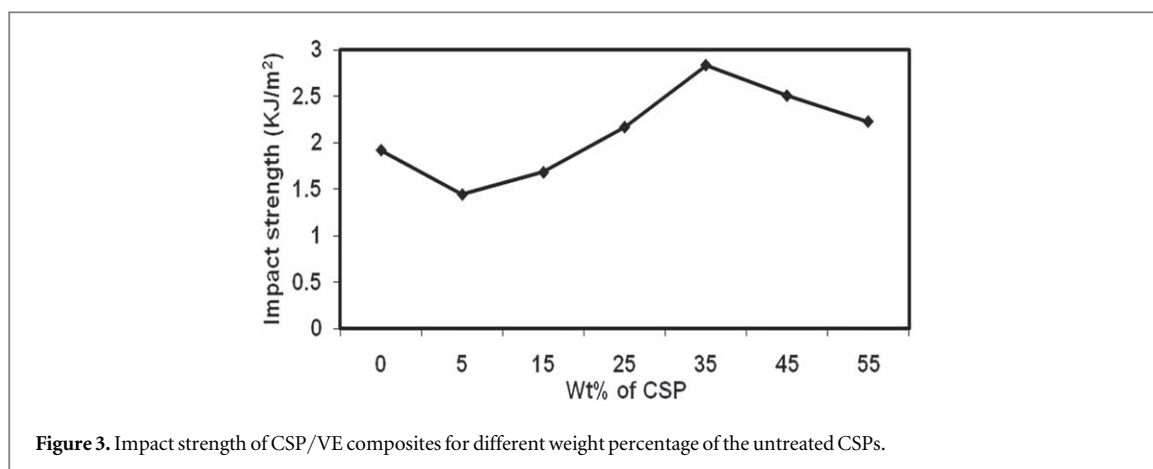


Figure 3. Impact strength of CSP/VE composites for different weight percentage of the untreated CSPs.

properties, which is continued up to 35 wt% and then dropped at 45 wt% and 55 wt%, as shown in tensile property values. The reason for the initial reduction in flexural properties of the CSP/VE composites may be due to the weak interfacial bonding between the particles and the polymer resin matrix. However, the addition of CSPs beyond the 15 wt% of CSPs improves the flexural properties of the composite up to 35 wt%. It may be due to the favourable dispersion and entanglement of the CSPs with the polymer resin matrix which increases the mechanical interlocking between the particles and the polymer matrix [24]. The flexural strength of 35 wt% composite was 38.7 MPa, which shows 32.99% of improvement as compared to the neat resin sample. Moreover, the addition of CSPs beyond the 35 wt%, composites show the reduction in the flexural properties which may be due to the existing of brittle behaviour into the CSP/VE composites.

3.1.3. Impact strength

The effects of the addition of the CSPs on the impact strength of composites are depicted in figure 3. From the figure 3, it can be seen that the impact strength of the CSP/VE composites increases with an increase in the content of CSPs up to 35 wt% and then dropped. An improvement of 47.92% was observed at 35 wt% composite when compared to the neat resin sample. The impact strength of composite reaches the impact strength value of the neat resin specimen at 25 wt%. The initial addition of the CSPs reduces the impact strength of the composites. It may due to the dispersion and distribution of fewer amounts (insufficient) of the CSPs into the VE resin matrix. The distribution of fewer amounts of particles within the polymer resin composites may lead to the weak transfer of applied load through the resin matrix, which creates the quick failure of the resulting composites. The maximum value in the impact strength of composite was obtained at 35 wt%. On the further addition of the CSPs (45 and 55 wt%), the values of the impact strength of composite are reduced. It is due to the insufficient amount of polymer resin matrix to wet the CSPs. The wettability between the particle and the polymer resin matrix is poor; therefore, the composite specimens are attaining the brittle nature. In this condition, composite specimens attain the quick failure by the brittle failure. The impact failure of the polymer composites reinforced with bio reinforcement agents (fiber or particle) occurs mainly due to the factors such as reinforcement agent's pull out, reinforcement agent and/or polymer matrix fracture and reinforcement agent / polymer matrix de-bonding. The pullout of reinforcing agents dissipates more energy compared to the fracture of the reinforcement agent.

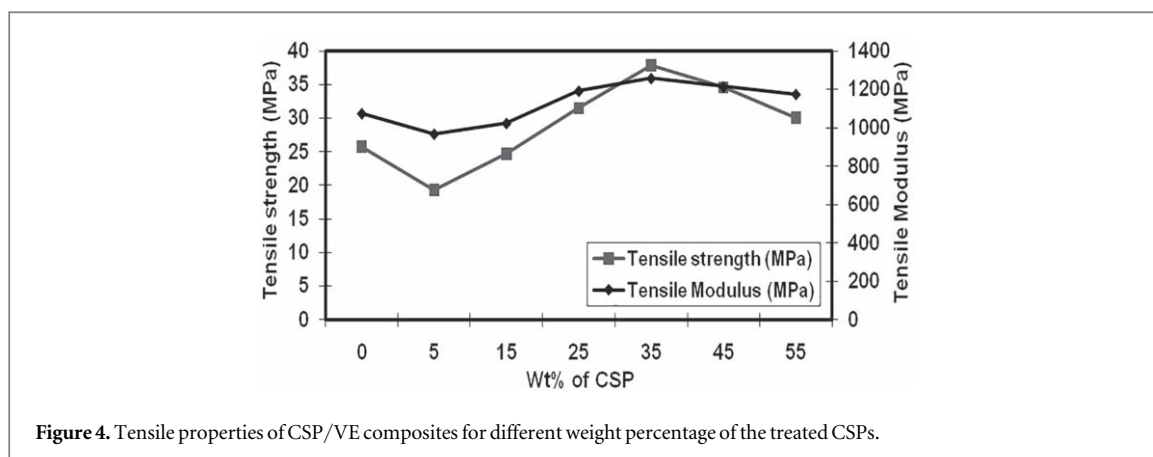


Figure 4. Tensile properties of CSP/VE composites for different weight percentage of the treated CSPs.

3.2. Mechanical properties of the treated CSP/VE composites

3.2.1. Tensile properties

The effect of CSP's content on the tensile properties of treated CSP/VE composites is illustrated in figure 4. From the observation of the untreated particle composites, it can be seen that the tensile properties of both untreated CSP/VE composites increased as the CSP's content increases due to the ability of the CSPs to support stress transfer from the VE resin matrix. In the case of treated particle composites also, increase of treated CSP content increases the tensile properties of the CSP/VE composites. The treated CSP/VE composites exhibit higher tensile properties compared to the untreated CSP/VE composites. It may be due to the removal of several components presented on the surface of the particles which increases the surface roughness and leads to the better mechanical interlocking between the treated particles and the resin matrix. During alkali treatment, the several bonding materials presented on the surface of the particles are removed and as a result the individual cellular elements are exposed on the particle surface. The NaOH solution is also comprised the interruption of hydrogen bonding in the structure of the CSPs which leads to the increase of surface roughness [25]. The tensile strength value of the treated particle composite at 35 wt% was 37.9 MPa which is 46.9% higher than the neat resin sample. The treated particle composite (31.5 MPa) shows 17.1% of the improvement in tensile strength compared to the untreated particle composite (26.9 MPa) at 25 wt%. As like in the untreated particle composites, the tensile properties of treated particle composite also increased with the addition of treated CSPs up to 35 wt% and then dropped. The tensile modulus of the treated particle composite is 1258 MPa which is 5.09% higher than the untreated particle composite at 35 wt%.

Some researchers [26, 27] have been used the following equation (1) to estimate the tensile strength of various particulate reinforced polymer composites through the quality of interfacial bonding strength between the particle and the matrix. The tensile strength of the composite falls along the straight line with zero interception, which represented by following equation:

$$\ln \sigma_{rel} = \ln \frac{\sigma_c}{\sigma_m} \frac{1 + 2.5V_p}{1 - V_p} = BV_p \quad (1)$$

where σ_c is tensile strength of composite, σ_m is the tensile strength of the particle. V_p is the volume fraction of the particle and B is an empirical parameter, which can be obtained directly as the slope of the linear regression. The value of B depends on the density and specific area of the particle and also depends on the interfacial bonding between the particle and the matrix. The tensile strength of the composite can be derived from the equation (1) as follows:

$$\sigma_c = \sigma_m \frac{1 - V_p}{1 + 2.5V_p} \exp(BV_p) \quad (2)$$

Moreover, the equation (1) can be used to estimate the relative tensile strength values of the untreated and treated CSP/VE composites, as shown in figure 5. The variations are observed in the B values (untreated = 0.2663, treated = 0.3276) of both the untreated and treated particle composites. For this reason, the relative tensile strength of the CSP/VE composite at treated condition is increased due to better mechanical interlocking between the particles and the matrix. Hence, it can be observed from this behaviour that there is a strong interfacial adhesion between the particle and the matrix due to the alkali treatment of particles.

3.2.2. Flexural properties

The flexural property values of the treated CSP/VE composites for different particle content are shown in figure 6. In the case of the untreated particle composites, increase of the CSP's content increases the flexural properties

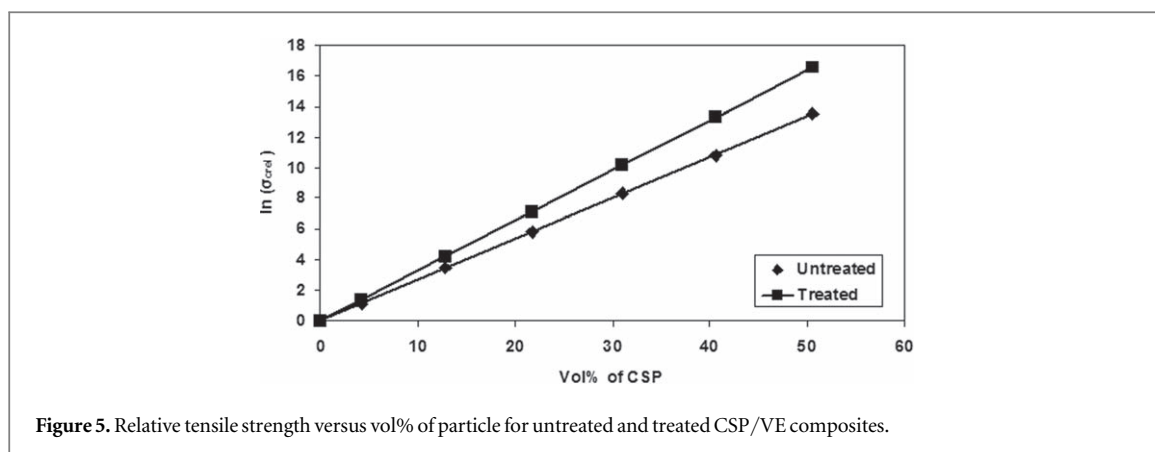


Figure 5. Relative tensile strength versus vol% of particle for untreated and treated CSP/VE composites.

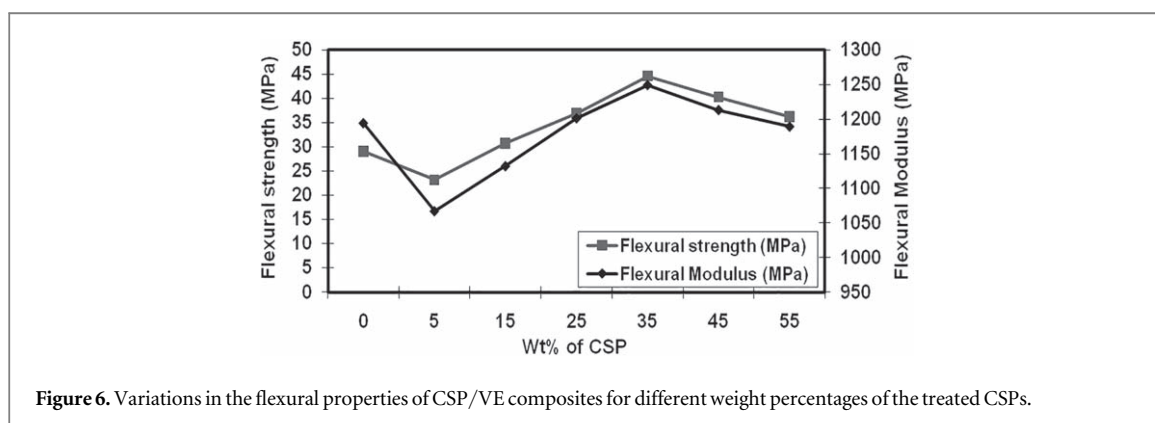


Figure 6. Variations in the flexural properties of CSP/VE composites for different weight percentages of the treated CSPs.

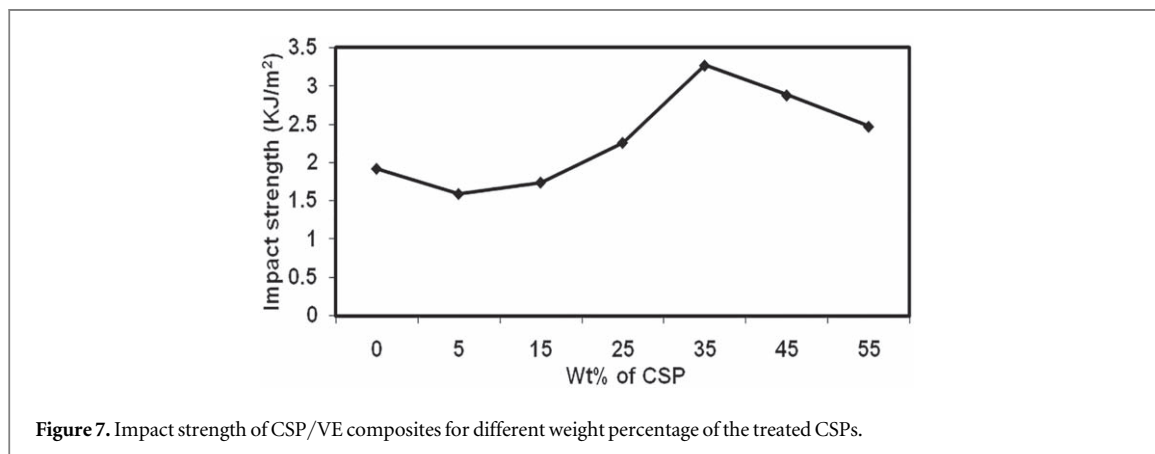
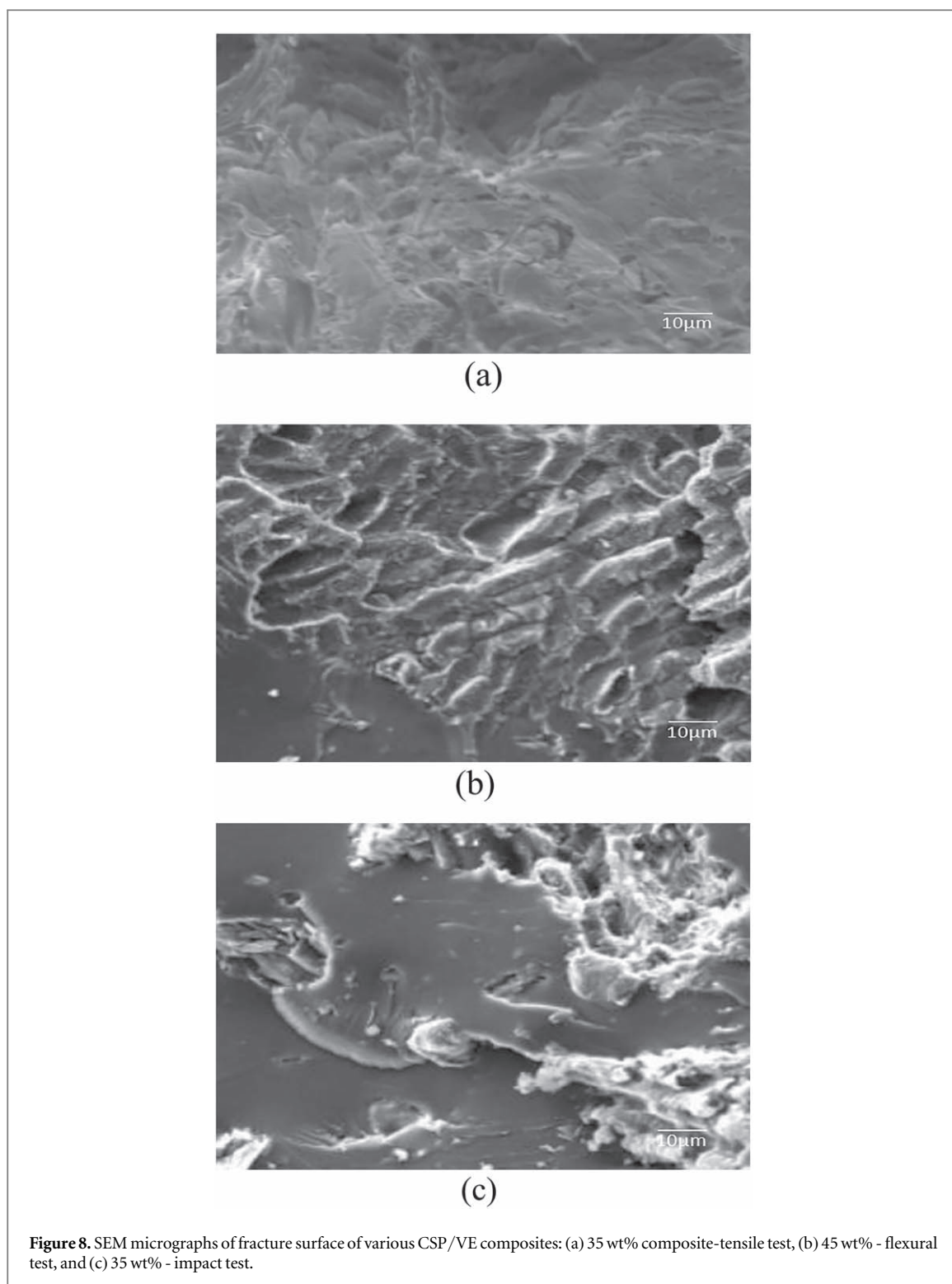


Figure 7. Impact strength of CSP/VE composites for different weight percentage of the treated CSPs.

when reinforced with the VE resin matrix. It is also observed in the treated particle composites that the flexural properties (flexural strength and modulus) increased with increasing the treated particle content. Moreover, the treated CSP/VE composites exhibit higher flexural property values compared to the untreated CSP/VE composites. It may be due to the better enhancement of interfacial bonding formed by the surface modification of the CSPs with the NaOH. The surface modified particles create the better wetting with the polymer resin matrix which improves the level of interfacial adhesion. The maximum flexural strength value (44.6 MPa) in the treated particle composite was obtained at 35 wt%, which is 15.3% higher than the untreated particle composite (38.7 MPa) at 35 wt%. The flexural strength and modulus values of the treated particle composite were also increased up to 35 wt% and then dropped. The maximum flexural modulus value (1299 MPa) was obtained at 35 wt% treated composite, which is 4.93% higher than the 35 wt% untreated composite (1238 MPa).

3.2.3. Impact strength

The impact strength results of the CSP/VE composites prepared at untreated and treated condition for different weight percentage of the CSPs were given in figure 7. Impact strength of the CSP/VE composite was found to be



increased with increasing untreated CSP's weight percentage up to 35 wt%, in VE resin matrix and then decreased. The high impact strength value was obtained at 35 wt% of the untreated CSPs. However, the impact strength values of the composite prepared with the treated CSPs were increased after alkali treatment. The impact strength of the treated composite (31.7 KJ m^{-2}) was increased by 11.62% when compared with the untreated composite at 35 wt% (2.84 KJ m^{-2}). It may be due to the removal of impurities and several soluble greasy contents to the surface of the particles, which develops a strong mechanical interlocking between the particle and the polymer matrix. However, the particle capacity like pull-out was reduced because of the significant part of the energy absorption, take place based on the ability of the particles. Therefore, the impact strength value of the treated particle composites increases [28, 29].

3.3. Fractographic studies

Figure 8 shows the SEM micrograph of the fracture surface of the treated CSP/VE composite (35 wt%) after the tensile test. Commonly, inclusion of fillers in the form of fibers or particles led to the fragment of the polymer matrix. From figure 8(a), it can be seen that the bonding of particles is better, especially for the 35 wt% reinforcement which may be the reason for the creation of good interfacial adhesion between the particles and the matrix. In the case of good interfacial adhesion, the local stresses (applied load) attain the fracture strength of the reinforced particles, which may crack. Generally, the failures of the reinforced particles may be due to geometry and orientation of the particles and also applied load. The incorporation of CSPs beyond 35 wt%, i.e., 45 wt% and 50 wt% creates poor wettability which leads to increased inability of the VE resin matrix to transfer the applied load (stress) for the particles. Here, the crack is initiated which may be due to the stress distribution at lower levels of stress. Moreover, at the higher weight percentage of particles, the deformation of the polymer matrix is blocked because the plastic strain is formed around the particles. Therefore, there is decreasing trend at tensile, flexural and impact properties for addition of 45 and 55 wt% of CSPs. The size of the particles plays a vital role on the crack initiation within the particulate reinforced polymer composites. The particles with small dimensions do not initiate the crack because these are creates plastic strain between the particles and the matrix. On the other hand, the particles with large dimensions facilitate greatly transfer of the applied load (stress) to the particles from the matrix. Here, the de-bonding of the particle-matrix interface is culminated. Several pulled out and failure of CSPs can be identified on the fracture surface of 45 wt% composite after the flexural test, as shown in figure 8(b). The reinforced particles are failed when the induced stress go beyond the strength of the particles. Few deprivation and clusters of the particles were also observed in the SEM micrograph (figure 8(c)) of fracture surface of 35 wt% composites obtained after impact test. This may be due to the improper distribution of CSPs in the certain region within the composites. It can be observed from all SEM micrograph that there is a reasonably uniform particle distribution and dispersion within the CSP/VE composites and it may be the reason for the enhancement of mechanical properties.

4. Conclusion

The effects of incorporation of CSPs on the mechanical properties of CSP/VE composites were studied in the present study. Mechanical properties of composite specimens were determined based on the weight percentages of the untreated and the treated CSPs. An increased mechanical property values was found in CSP/VE composites with the incorporation of both the untreated and treated CSPs; moreover, marked improvements were observed in the treated particle composites compared to the untreated particle composites. The best combination of mechanical properties was obtained at 35 wt% of the CSPs in both the untreated and treated particle composites. Mechanical properties of the treated CSP/VE composites were higher than the untreated CSP/VE composites. It may be due to the treatment of CSPs using an alkali solution which leads to the better enhancement of interfacial adhesion between the particles and the polymer resin matrix, resulting in improved mechanical properties. The Pukanszky's model was also proved the improvement of interfacial bonding between the particles and the matrix due to alkali treatment of particles. It is suggested that if the surface modified coconut shell particles can effectively be used as reinforcing filler for the preparation of polymer composites with the improved property levels.

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