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Experimental characterization of coconut shell particle reinforced epoxy composites

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- ✓ Load-Deflection Curve;

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Abstract

In the present investigation, composites are developed using coconut shell particles as fillers in epoxy resin. Effect of particle size and filler volume fraction on mechanical properties like tensile. flexural, impact and hardness has been evaluated experimentally. Specimens preparation and testing was carried out as per ASTM standards. The results of the investigation showed that the tensile, flexural and impact properties are found to decrease with the increase in the filler particle size and filler volume fraction. Coconut shell particle filled epoxy composites indicated hardness, greater than that of neat epoxy. The observations from fractographic investigation carried out to determine the mode of fracture under different types of loading is also reported.

1. Introduction

Fillers are used in polymer matrices for various purposes such as to reduce cost, to reduce resin curing shrinkage, to control resin viscosity and to improve the stiffness and other properties of matrix. Most commonly used filler materials in polymer matrix composites are calcium carbonate [1], alumina [2] and silicon carbide [3]. However, in the recent years, composites reinforced with natural filler materials are inconsiderable demand because of their low cost, renewable and biodegradable nature. This has reduced the use of high cost, non biodegradable traditional reinforcement materials like ceramic fillers and synthetic fibers in various secondary load bearing applications. Natural fillers such as groundnut shell particle, wood flour, rice husk, coconut husk, wheat husk, etc. are bio based materials (agricultural resources) and are available in plenty in countries like Malaysia, Indonesia, Thailand, Sri Lanka and India. It is reported that about 600 metric ton of wastes have been generated in India alone from agricultural sources [4]. Attempts have been made by several researchers to develop and characterize the composites using natural (bio) fillers as reinforcement in particle or powder form [5-11]. Flores et al [5] discussed the effect of particle size and filler concentrations on the tensile, flexural and impact properties of polystyrene filled with white oak wood flour. They found both filler particle size and content have significant effect on these properties. Brent et al [6] evaluated the tensile, flexural, impact properties of composites made of Paulownia wood (PW) particles blended with polypropylene (PP).The test results show that particle size significantly affects the mechanical properties of composites. PW composites containing particles below 250 µm size exhibited the lower ultimate strength, bending modulus, and impact strength properties compared to composites with larger particle size. Raju et al [7] evaluated the mechanical properties of groundnut shell particle / epoxy (GSPE) composites. The composites were prepared with randomly distributed groundnut shell particles of different sizes reinforced in epoxy resin with different volume fractions i.e., 70:30, 65:35, and 60:40 (filler - resin proportion).The peak values of tensile properties, flexural properties, and impact strength were observed in a composite with 60:40 volume fraction and 0.5mm particle size. They suggested that the GSPE composites can be considered as a material alternative to wood. Salmah et al [8] developed bio composites using coconut shell powder (CSP) as filler in polylactic acid (PLA). The effects of filler content and acrylic acid modification to filler, on tensile properties were investigated. The results revealed decreasing trend in tensile strength and elongation at break with the addition of CSP to PLA. Further, addition

of 3% acrylic acid (as chemical modifier) to CSP/PLA composite showed higher tensile properties with lower strain to failure. Coconut Shell particles can also be used as filler material. The shell is organic in nature and also has good durability characteristics, high toughness and abrasion resistant properties. The shell is similar to hard woods in chemical composition though lignin content is higher and cellulose content is lower. Husseinsyah et al., [9] used coconut shell (CS) as filler at different content in polyester composites. A catalyst, butanox M-60 was used to initiate the polymerization reaction. The effect of coconut shell content on the mechanical, water absorption and morphological properties were studied. The results revealed that increased in coconut shell content have increased the tensile strength, Young's modulus and the water absorption but reduced the elongation at break. Sarki et al [10] appraised the possibility of using coconut shell particle as filler in epoxy resin with different weight fractions (10%, 20% and 30%). The results showed that there is an increase in the tensile properties and marginal decrease in impact strength with the increase in coconut shell particles content. Scanning electron microscopy (SEM) observation of the composite showed that there is a good interfacial bonding between coconut shell particles and epoxy. Bhaskar and Singh [11] investigated the mechanical properties of coconut shell particle reinforced epoxy composites in different filler weight percentages of 20, 25, 30 and 35%. Their results showed that ultimate strength, modulus of elasticity and % elongation decreases with increase in the wt% of shell particle.It can be concluded from the review of the literature that bio fillers like coconut shell powder, wood flour, groundnut shell powder etc., can be effectively used as reinforcement materials to develop composites with different matrix materials for applications like furnitures, package boxes, interior decorations etc. In the present study, the coconut shell particles with different sizes and compositions are used as reinforcement in epoxy resin and the resulting particulate composites is experimentally characterized for mechanical properties.

2. Materials

The shells of fully matured coconuts were first cleaned and crushed into smaller grains. These smaller grains were then subjected to repeated grinding in a pulverizing machine, after passing through cyclones and vibratory sieves fitted with phosphor-bronze mesh. The grains are finally drawn out in different sizes of 0.25, 0.5, 1 and 2mm.The bulk density of coconut shell particle was determined to be 0.745g/cc which closely matches with the value given in articles [12] and [13]. Epoxy resin LY 556 and hardener HY 951 in the ratio 10:1 was used as matrix material. Melamine resin or melamine formaldehyde (also shortened to melamine) was also used in the epoxy to melamine ratio of 20:1, to increase the rate of curing, bonding strength and to improve the surface finish of the developed composites. The epoxy resin, hardener, and melamine were procured from M/s Insulation house, Bangalore, India.

3. Fabrication of Composite

Pre determined quantity of coconut shell particles and epoxy resin were taken in a plastic container and stirred thoroughly to get a homogeneous mixture. After adding 10% of hardener and 5% of melamine, the mixture was again stirred for 10 minutes and mixture was poured into (180×140×10) mm^3 mould and is allowed to cure for 24 hrs at room temperature. After curing, composite board was taken out from the mold and sun dried for 3 hours. Composites were prepared with filler (coconut shell particles) volume fractions of 40%, 50% and 60% with different sizes of the particles. The densities of the composites were determined by water displacement method and filler weight fractions were calculated using equation (1). The details of fabricated coconut shell particle reinforced epoxy (CSPE) composites are presented in Table 1. The test samples have been prepared as per ASTM standards. Samples were cut from the composite boards using diamond point cutter. ASTM standards D638, D790, D256 and D2240 were followed for conducting tensile, flexural, impact and hardness tests respectively.

$$W_f = \frac{\rho_f}{\rho_c} V_f \quad \text{.....(1)}$$

4. Experimental

4.1 *Tensile Test*

Tensile test was conducted using universal testing machine, Instron 3382 of 100 kN capacity using data acquisition software Instron's series IX™/s. The rate of loading adopted was 5mm/min. The tensile properties such as tensile strength, tensile modulus and % strain at break were determined from the stress-strain plot. For each filler volume percentage, five identical specimens were tested and average results are taken.

Table 1: CSPE Composites Designation

Series Name	Samples Designation	Filler Size (mm)	Filler Volume Fraction (%)	Filler Weight Fraction (%)
Series A	A1	0.25	40	39.28
	A2	0.25	50	54.45
	A3	0.25	60	68.04
Series B	B1	0.5	40	39.28
	B2	0.5	50	54.45
	B3	0.5	60	68.04
Series C	C1	1.0	40	39.28
	C2	1.0	50	54.45
	C3	1.0	60	68.04
Series D	D1	2.0	40	39.28
	D2	2.0	50	54.45
	D3	2.0	60	68.04

4.2. *Flexural test*

Three point bending test was conducted on the same machine. The ratio of span length to depth was adopted as 16. The rate of loading was 3mm/min. For each filler volume percentages, five identical specimens were tested and average results are taken. The flexural strength and flexural modulus were calculated using equations (2) and (3) respectively.

$$\text{Flexural strength (MPa)} = \frac{3PL}{2bh^2} \quad \ldots\ldots\ldots \quad (2)$$

$$\text{Flexural modulus(MPa)} = \frac{mL^3}{4bh^3} \quad \ldots\ldots\ldots \quad (3)$$

Where P is the maximum load applied on test specimen in Newton, L is the span length in mm, b is the width of the specimen in mm, d is the thickness of specimen in mm, and m is the slope of tangent to the initial straight line portion of load- deflection curve measured in N/mm.

4.3. *Impact test*

Izod impact test was conducted using pendulum type impact tester. Specimens of 63.5 mm length, 10mm width and 10mm thickness with the depth of V notch equal to 2mm and notch angle equal to 45^0 were used for the testing. The energy absorbed while breaking the specimen was recorded. For each composition, five identical specimens were tested and average results are reported. The impact strength of the specimen was computed using equation (4).

$$\text{Izod Impact Strength} = \left[\frac{J}{A}\right] \text{ in N/m} \quad \ldots\ldots\ldots \quad (4)$$

Where, J is the Energy absorbed in Joule, A is the area of cross section of the specimen below the notch in m^2

4.4. *Hardness test*

Shore hardness is a measure of the resistance of a material to penetration of a spring loaded needle-like indenter. Shore hardness test was conducted using an instrument called Durometer. The two most common indenters are ASTM D2240 type A and type D. The type A is for soft plastics, while type D is for harder materials. As CSPE composites are found harder, type D indenter was used in the present investigation. The geometry of the test sample for shore D hardness test was 50 mm × 50 mm × 12 mm.

5. Results and Discussion

5.1. *Tensile Testing*

The stress-strain curves for CSPE composite samples with filler volume fractions of 40%, 50% and 60% are analyzed. For all types of samples, the stress-strain curves are almost linear until failure at peak load. The ultimate tensile stress, tensile modulus and the %strain at break are plotted as a function of filler particle size and filler volume fraction in Figures 1, 2 and 3 respectively. It can be observed from figures, that the aforementioned tensile properties decrease with the increase in the filler particle size and filler volume fraction.

Similar kinds of results were observed for groundnut shell particles reinforced epoxy composites [7] and for periwinkle shell particles reinforced polyester composites [14]. Higher tensile stress at lower particles size and filler volume fraction may be due to the increased packing factor, lower void content [7], improved interfacial bonding and better stress transfer mechanism between resin and fillers [14]. A lower value of % strain at break is an indication of brittleness of the material [15]. CSPE composite with 40% filler volume fraction and 0.25 mm particle size depicted maximum tensile stress (32.84 MPa), whereas it is minimum for 60% filler volume fraction and 2 mm particle size(11.27 MPa).

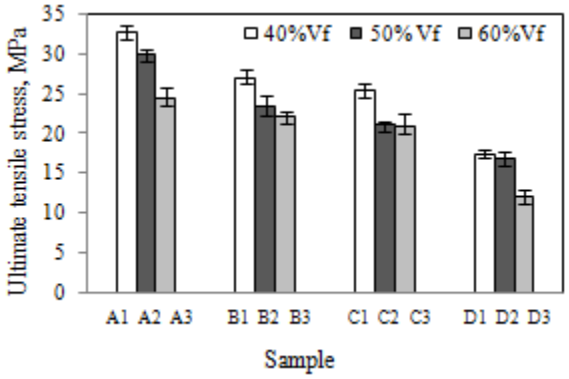


Figure 1: Ultimate tensile stress of CSPE samples

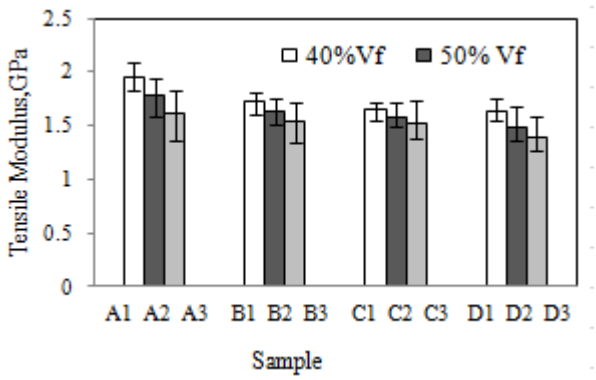


Figure 2: Tensile modulus of CSPE samples

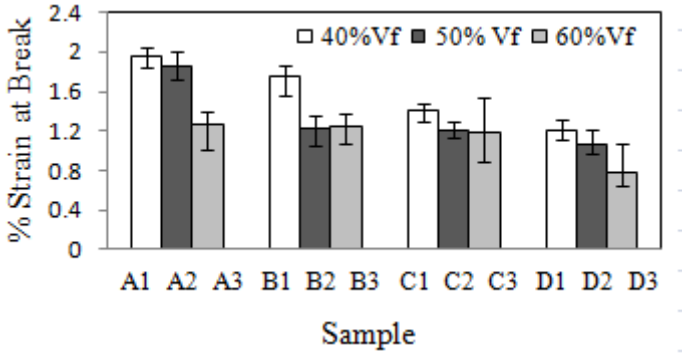


Figure 3: %Strain at Break in Tensile Fractured CSPE samples

5.2. *Flexural Testing*

The load–deflection curves obtained under flexural tests for CSPE composites with different filler volume fractions are scrutinized. It is observed that the behavior of the composite is found to be linear until fracture. Under flexural loading, the surfaces of the specimen are subjected to greater strains than the sample centre. The failure initiates with the development of crack on the tension side. The load-deflection plots for different samples are compared in Figure 4 (a-c). It can be seen from load-deflection plots that the samples with 0.25 mm particle size (A series) exhibited greater load carrying ability and deflection. This is an indication of less brittleness of the material with slow rate of crack propagation that has led to higher load carrying ability. However, when the particle size increases, the material becomes more and more brittle. This reduces the load carrying ability due to faster rate of crack propagation leading to early failure of the material as indicated by low deflection values at failure. The decrease in the load carrying ability results in the decrease of flexural properties (equations 2 and 3) with the increase in the particle size and filler volume fraction as shown in Figures 5 and 6. Further, for smaller size of the particles, improvement in bonding at the interface may also result in improvement of flexural properties [16]. The maximum value of flexural strength is 52 MPa for sample A1 (0.25 mm particle size and 40% filler volume fraction) whereas it is just 18 MPa for sample D3 (2 mm particle size and 60% filler volume fraction). Similarly, the flexural modulus is maximum for sample A1 (4.76 GPa) and minimum for sample D3 (2.83 GPa).

The tensile and flexural properties of CSPE composite with 0.25mm particle size and 40% filler volume fraction from the present investigation are compared with the tensile and flexural properties of other bio composite materials based on literature in Table 2. It can be observed from Table 2 that both tensile and flexural properties of CSPE composite are comparable to many wood flour and ground shell reinforced polymer composites.

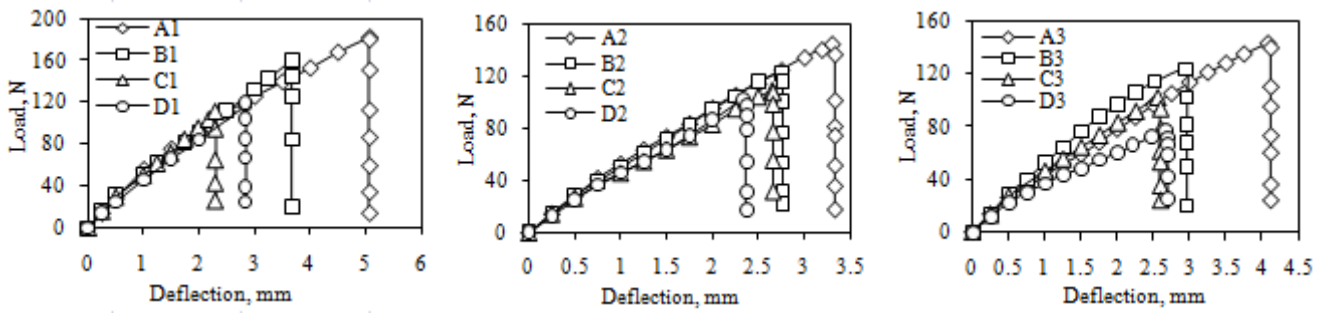


Figure 4: Load-deflection curves for (a) 40% (b) 50% and (c) 60% filler volume fraction CSPE samples

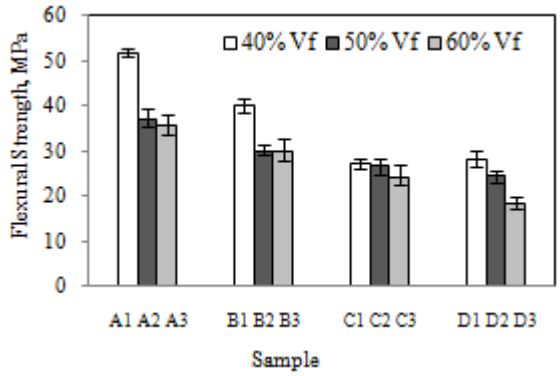


Figure 5: Flexural strength of CSPE samples

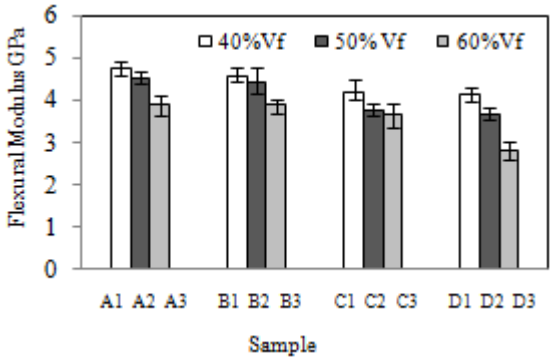


Figure 6: Flexural modulus of CSPE samples

Table 2: Mechanical properties of different bio filler based composites

Composite Samples	Composition Filler wt % and size	Tensile Strength (MPa)	Tensile Modulus (MPa)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Reference
Coconut shell/ Epoxy	39.28 wt%, 0.25mm	32.84	1951.7	52	4765.1	Present Study
Polystyrene/WF	10wt%, 100 mesh	31.5	25200	40.3	3600	[5]
PW/PP	25wt%, 425-600 µm, 5% MAPP	29	1400	--	3850	[6]
GNS/Epoxy	60wt% , 0.5mm	9.6842	243.79	22.612	2010	[7]
CSP/Epoxy	20wt% , <100 µm	37.31	688.14	--	--	[10]
WF/HDPE	35 wt%, 50 mesh, 2% MAPE	27.43	693.67	--	--	[18]
Wood/PP	60wt% , 70-150µm	39	5400	--	--	[19]
Polystyrene/WF	20 wt% , 40-150 µm, 2% SMA	--	--	67.43	4806	[20]
WF/ PP	40wt%, 40-60 µm, 5%MAPP, 3% Clay	18.8	5998	35	3200	[21]

5.3. *Impact Testing*

The Izod impact strengths for various samples are compared in Figure 7. The Figure reveals that the impact strength of the composites is considerably affected by filler particle size as well as filler volume fraction. Composites with higher filler particle size and volume fraction exhibited poor impact strength. The decrease in the impact strength at higher filler size and content may be due to the weak interfacial interaction between the filler and matrix materials [7]. Further, at higher filler content, the energy required to initiate the crack decreases, thereby reducing the impact bending strength [5]. The maximum value of impact strength is 65 kJ/m^2 for sample A1 (0.25 mm particle size and 40% filler volume fraction) whereas it is 45 kJ/m^2 for sample D3 (2 mm particle size and 60% filler volume fraction).

5.4. *Hardness Testing*

The hardness trials were taken at three different regions on the surface of the sample and the average values are shown in Figure 8. It is clear from the Figure that the composites with filler particle size of 0.25mm have the highest value of hardness for all the filler volume fractions. The hardness of CSPE composites was found to

decrease with the increase in the filler particle size up to 1mm [22]. With the change in the volume fraction, composites with 0.5 mm and 1 mm particle size exhibited marginal variation in the hardness number.

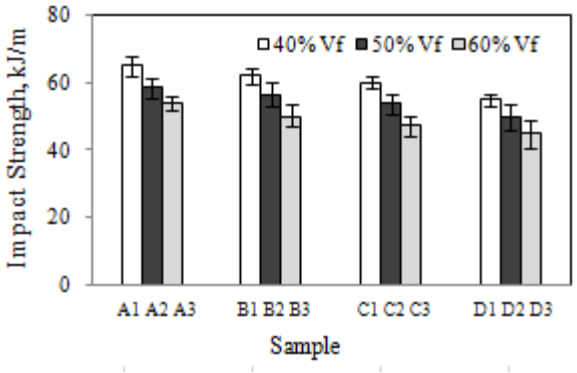


Figure 7: Izod Impact strength of CSPE samples

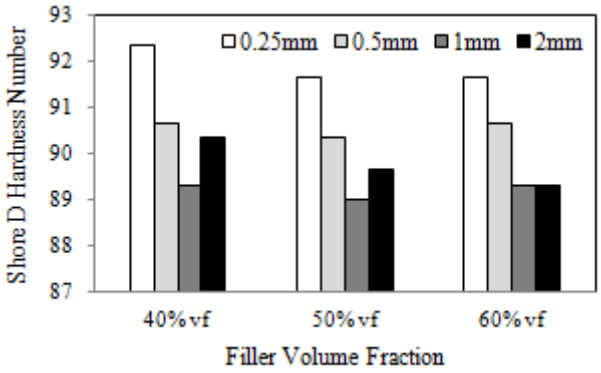


Figure 8: Hardness number of CSPE samples

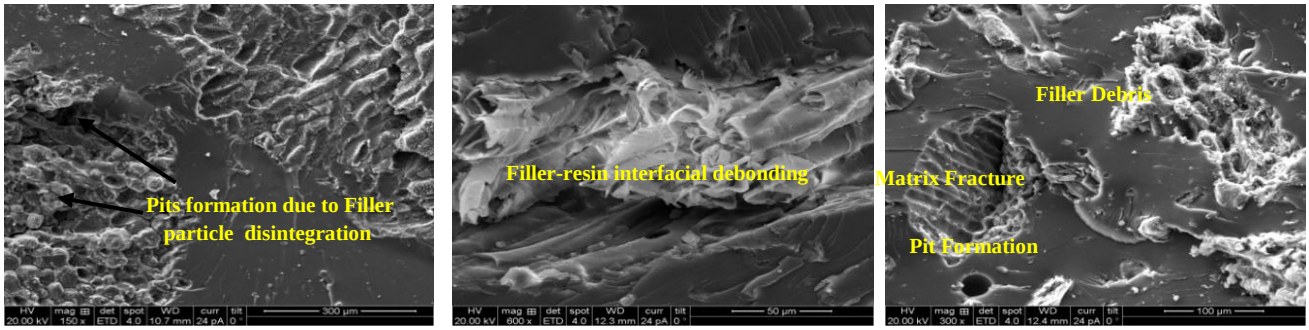
The highest value of hardness for 0.25 mm CSPE composites was found to be 92.33 for 40 % filler volume fraction and the minimum value of hardness number is 89 for 1mm filler size composites with filler volume fraction of 50%. However, this value of hardness is still greater the hardness of neat epoxy LY556 which is 82 [17]. Hardness numbers have also been evaluated for commercially available and most commonly used varieties of wood and a comparison was made with that of CSPE composite (A1 sample) in Table 3. The results presented in table reveal that CSPE composite is harder than the wood samples considered and hence, it can be a better substitute for wood in various applications.

Table 3: Comparison of Hardness number of CSPE composite sample A1 with different wood materials

Material Designation	Commercial Name	Binomial Name	Hardness Number
A1	------	**CSPE composite (40%Vf / 0.25mm)**	**92.33**
W1	Teak	Tectona Grandis	70.0
W2	Rosewood	Dalbergia Latifolia	77.3
W3	Honne	Pterocarpus Marsupium	71.7
W4	Acacia	Acacia Auriculiformis	68.7
W5	Todsal	Grewia Titifolia	73.3
W6	Neem	Azadirachta Indica	73.0
W7	Jack Tree	Artocarpus Intergrifolia	67.7
W8	Matthi	Terminalia Arjuna	77.7
W9	Bilvara	Albizia Odoratissima	77.7
W10	Subabul	Leucaena Leucocephala	62.0

6. Fractography

Scanning electron microscopic study was carried out to investigate the mode of fracture under different tests. The fractured surfaces of tensile, flexural, and impact tested specimens with 0.25 mm particle size and 40% filler volume fraction (sample A1) that exhibited highest properties, have been examined using computer interfaced scanning electron microscope JEOL 6360. The instrument was operated at 20 kV. The samples for examination were obtained by cutting sections of about 5mm in length from just below the fractured zone. The fractured surfaces of the samples were sputter coated with a thin layer of gold to minimize the charging problem and then kept under microscope for scanning. Figure 9(a) shows the micrograph of tensile fractured surface of the sample A1. In this case, the failure of the sample was mainly due to filler particle disintegration with the matrix forming pits on the surface of the sample and interfacial debonding between the filler particles and the resin. Some of the pulled out filler particles can also be seen in the figure 9. Figure 9(b) shows the micrograph of flexural fractured surface of CSPE sample A1 at a higher magnification factor of 800 X. It can be seen from the figure that, the failure of the sample was mainly due to interfacial debonding between the filler particles and the resin. The micrograph of Izod impact fractured surface of CSPE sample A1 is shown in Figure 9(c). In this case the failure was observed to be due to multiple modes of fracture like interfacial debonding, matrix fracture, pit and debris formation.



(a) **(b)** **(c)**

Figure 9: SEM Images of (a) Tensile fractured (b) Flexural fractured (c) Izod impact fractured surface of typical CSPE sample A1

Conclusion

The objective of this study was to experimentally investigate the effect of particles size and its volume fraction on mechanical properties of CSPE composites. It has been observed that the mechanical properties of developed composites are much affected by both the size as well as the content of filler particles. Tensile, flexural and impact properties are found to decrease with the increase in the filler particle size and filler volume fraction. CSPE composite with 40% filler volume fraction and 0.25 mm particle size exhibited highest tensile stress of 32.84 MPa, flexural strength of 52 MPa, and impact strength of 65 kJ/m^2. Coconut shell particle filled epoxy composites indicated hardness, greater than that of neat epoxy. The highest value of hardness for 0.25 mm CSPE composites was found to be 92.33 for 40 % filler volume fraction. Hence this composite can be a promising material for low load bearing applications in automotive and aircraft industry, furniture, packaging industry, partition panels etc.

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