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Experimental Investigation into Mechanical Properties of Coconut Shell Powder Modified Epoxy/3D E-Glass Composites

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Abstract

The aim of this research work was to investigate the influence of resin modification with Coconut Shell Powder (CSP) on the tensile, flexural and impact properties of composites reinforced with 3D E-glass orthogonally woven fabric. The composites were fabricated using a combination of hand lay-up and press moulding techniques. Three different proportion of CSP namely, 0.5%, 1.5% and 3% by weight of resin were considered for modifying the epoxy resin. The properties of these composites were determined and compared with composites fabricated without coconut shell powder. Additionally, to ascertain the effect of dispersion technique on the mechanical properties of the composites, their tensile strengths were compared with composites fabricated with epoxy in which CSP was added to the resin and mixed mechanically. Improved mechanical properties were obtained for composites fabricated with modified resin and an increasing trend was observed with increase in proportion of CSP. The highest properties were obtained for composites with 3% CSP content showing an increase of about 117%, 87% and 39% in tensile, flexural and impact strengths respectively over the composites without CSP. Tensile strengths of composites prepared by mechanical dispersion of CSP were lower than the resin modified composites having the same CSP content, showing a drop of about 53% and 25% thereby proving the efficacy of resin modification process. Scanning Electron Microscopy (SEM) was employed to analyse the characteristics of the CSP and to investigate the various failure modes.

Keywords: 3D orthogonally woven E-glass; Coconut Shell Powder; Resin modification; Mechanical properties; Scanning Electron Microscopy.

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1. Introduction

Over the last few decades, Polymer Matrix Composites (PMCs) have emerged as one of the widely used structurally advanced materials and have shown great potential for future growth. Composites are a combination of two or more materials that possess significantly different properties, which are separated by an interface that results in a material that has properties intermediate to those of its constituents. Polymer composites possess the advantage of having high strength to weight ratio, resistance to chemical attacks, corrosion and wear, and higher stiffness.[1] Furthermore, they allow flexibility of design and composition, maintain dimensional accuracy and yield efficiency in production cost.[2] Due to the numerous benefits that polymer composites offer, such materials have been adopted in various industries. Polymer composites are widely used to manufacture lightweight airframes for aerospace vehicles, the framework of automobiles, marine or submarines, locomotives, wind turbine blades, personal protective armors, sporting goods, furniture, and is also used in biomedical applications such as prosthesis.[3–9]

Glass fibers are popular due to their low cost and are available in the form of chopped strands, long continuous fibers, woven mats, and 3D fabrics. Orthogonal 3D glass fabrics consist of fibers woven bi-directionally and stacked without interlacing of the yarns. The fibers are oriented in two mutually perpendicular directions, namely the warp and the weft. A third yarn which is called the binder yarn runs in a plane that is perpendicular to the plane of the warp and the

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weft, adding a third out-of-the plane element to the fabric. This provides strength for the out-of-the-plane loads.[10] The advantages of 3D glass fabric include increased permeability of the resin through the thickness of the fabric, an increased resistance to delamination due to the existence of a binder yarn along the z-direction, reduced infusion time and high formability.[11] Researchers also concluded that 3D fabric had more fatigue resistance in fill (warp) direction than the same in plain weave fabric.[12] The composites were fabricated using 3D E-Glass non-crimp orthogonal fabric which demonstrated superior mechanical properties in the fill direction, as the Z-binder yarn had resisted crack propagation and delamination.[13]

Synthetic fibres such as glass, carbon, aramid, have proven their superiority as far as reinforcements are concerned but, bring along with them some critical drawbacks such as increased production costs, increased carbon emissions, health hazards and difficulty in recycling at the end of its useful life. To reduce the ill-effects of use of synthetic fibres, researchers are exploring means to reduce the amount of synthetic reinforcement by following the path of hybridization, either by using natural fibres or natural fillers.[14-16] Researchers are also exploring use of nature friendly matrices like biopolymers that include Polylactic Acid, Thermoplastic starch, Polyvinyl Alcohol, Chitosan, *etc*.[14] But like natural fibres, have some inherent limitations like sensitive to moisture, low mechanical strength and easily degradable; thereby making them unsuitable for engineering applications where strength and longer durability are the key requirements.[7,17]

Addition of fillers improves the properties of the composites from the structural perspective. Use of micro and nano fillers have shown to improve the mechanical, thermal, tribological, impact and electromagnetic shielding properties of composites.[7,18-23] Fillers can be either natural or synthetic, based on their origin. Commercially available synthetic fillers such as carbon nanotubes, aluminium oxide (Al_2O_3), graphene have been employed in industries and have shown promising results. Vinay *et al*.[24] in their study involving Al_2O_3 as nano fillers in Basalt epoxy composites reported improvement in flexural and Inter Laminar Shear Strength properties but drop in tensile properties. They also observed improvement in hardness and hence improved wear properties. Graphene as nano fillers were explored by Ganapathy *et al*.[25] wherein they reported improvement in mechanical properties such as tensile, and flexural strengths till a certain proportion. Though, commercially available fillers have proven to useful in improving the properties of composites, the growing ecological aspect provides an incentive to look for natural alternatives. Natural fillers are typically found in the form of ash or powder. Fillers such as rice husk ash, sawdust, fishbone powder, laterite, crushed Acacia Nilotica, eggshell powder, tamarind powder, coconut coir powder, coconut shell powder have been explored.[26-34] They have gained prominence due to their biodegradability, low cost, and ease of availability. The methods that are used to process them are traditional and basic, such as fragmentation and crushing them into finer particles without the use of heavy machinery, yielding productivity in manufacturing.[35] Abhishek *et al*[36] explored the potential of nano fillers obtained from fish bone in date palm epoxy composites, where they observed improvement in the mechanical properties of the composites. Ganesan *et al*.[37] used nano clay and egg shell fillers in combination and obtained positive results for jut fibre polyester composites.

Agro-fillers are natural fillers derived from agricultural products. One of the common agricultural sources are coconuts. Several families in India are dependent, either directly or indirectly, on coconut production for their livelihood. India has made tremendous growth in coconut cultivation and has become one of the leading countries in coconut production along with Indonesia and The Philippines. The four southern states of Kerala, Tamil Nadu, Karnataka, and Andhra Pradesh dominate in Coconut production by producing more than 90% of overall production in India. Focusing on the anatomy of the coconut, there exists several layers. The interiors consist of a cavity that is filled with water. Encapsuling this water is the tender, edible (kernel) of the fruit. Surrounding the kernel is the hard shell or endocarp. This shell of the coconut fruit is extremely durable as it has high toughness and abrasion resistance.[38] They are often decorated as ornaments and handicrafts or used as household utensils. The charcoal obtained from burnt coconut shell is also used as a source of activated carbon. The powdered shell is also commonly employed in making insect repellent in the form of incense sticks and mosquito coils.[39] Despite having such vast application, a large remnant of coconut shells is disposed of as agricultural waste by most industries. Coconut particles being wear and chemically resistant and having high strength modulus, can be crushed into micro-sized particles to be utilized as filler in polymer composites. The production of natural fillers, once commercialized, will also serve as an employment opportunity in rural areas and improve the economic status of the community. It will also help to reduce the harmful and toxic waste disposal techniques and improve environmental conditions.

Somashekar *et al*.[32] used a combination of coconut shell powder and tamarind shell powder as fillers. They studied the change in the mechanical properties of the epoxy resin with individual fillers and a mix of both. They reported increase in tensile strength from about 20 MPa to 30 MPa while flexural strength increased from about 40 MPa to 100 MPa with addition of fillers. Sarki *et al*.[40] and Kumar *et al*.[41] used

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coconut shell powder as a filler to modify the epoxy resin and compared its properties to that of neat epoxy resin. While Sarki *et al*.[40] reported increase in tensile strength from about 18 MPa to 37 MPa, Kumar *et al*.[41] reported an improvement in mechanical properties, namely the tensile and flexural strength which reached to an optimum filler percentage beyond which the properties deteriorated.

While coconut shell powder does show tremendous benefits, there are significant drawbacks that are brought about by natural fillers. The lignocellulosic nature of the fillers, increases the hygroscopicity of the composite, hindering the interfacial adhesion between the phases.[42] Considering their microscopic size, particles often agglomerate and results in the formation of stress concentration zones making the material prone to early failure.[43]

The dispersion of the particles at this scale can become non uniform easily, either by agglomeration or gravity segregation.[44] This is due to the very low particle size and the density differences in that of the particle and the dispersion medium, in this case, resin. Thus, there is usage of appropriate mixing methods to ensure uniform dispersion of the filler particles. This ensures consistency in the properties of the resultant composite. Some methods of dispersion of fillers that have been used in the research works so far include ultrasonic mixing, shear mixing, micro-fluidizing. direct mixing, magnetic stirring.[45] The commonality of the usage can vary with particle sizes, *i.e.*, the scale of particles. Nano particles such as carbon nanotubes have been dispersed using mixing methods such as ultrasonic mixing, shear mixing, microfluidizer, *etc*. Particles such as coconut shell powder, rice husk powder have been dispersed mainly either through direct mixing,[46] or magnetic stirring.[47]

However, the effect of the dispersion methods such as ultrasonic mixing or magnetic stirring has not been extensively explored in the area of micro scale particles such as coconut shell powder. Now, conventional methods such as mechanical stirring have been overwhelmingly tried for micro scale particles. This work aims to investigate the effect of the nano particle dispersion methods, mainly ultrasonication and magnetic stirring in the epoxy resin, on the mechanical properties of Coconut Shell Powder filled 3D - E Glass Orthogonal Woven fabric epoxy composites. Furthermore, Fourier Transform InfraRed Spectroscopy and Scanning Electron Microscopy has been done to support the findings.

2. Experimental details

2.1 Materials

3D orthogonally woven E-glass fabric with an areal density of 1830 GSM was procured from Fibermax Composites, Greece. The fabric had a thickness of around 1.7 mm with 8 threads per cm in the warp direction and 11.4 threads per cm in the weft direction. Epoxamite A103 slow curing epoxy resin was procured from Smooth-On Inc, USA. This resin was chosen as it has a longer pot life of around 45 min which makes fabrication easy. Coconut Shell Powder (CSP) was prepared from shells of locally bought coconuts. The shell was thoroughly cleaned with emery cloth to remove any coir from its outer surface and residual coconut flesh from the inside. The shells were washed, and later, sun-dried for a day in order to remove any moisture from it. The sun-dried coconut shells were broken down to small chips using a pulverizing equipment and then grounded to a fine powder using a ball mill. CSP was then sieved using a 45 μm diameter opening sieve, as shown in Fig. S1.

2.2 Fabrication of composites

Composite panels were fabricated using a combination of hand lay-up and press moulding techniques. Each laminate made use of two plies of 3D fabric, and a fiber weight fraction of about 72% was maintained during the hand lay-up process, which was done on an open mould. The laid-up composite panel was later compressed under a press mould where it was allowed to cure for 24h. The panels were compressed to a thickness of 2.5 mm using a compression factor of 1.4.[11] In order to achieve the required thickness, precisely machined shims were used. Four different panels of composites were fabricated with varying proportions of CSP. Post curing was in the form of leaving the panels at room temperature for 7 days. Before the lay-up process, the resin was modified by dispersing CSP into it using a magnetic stirrer and sonicator. CSP was added to acetone using a ratio of 1:25 by weight and was stirred using a magnetic stirrer. The stirred mix was sonicated and later added to the resin. The magnetic stirring and sonication were repeated with the resin-acetone-CSP mix. Both magnetic stirring and sonication were done for 15 min each in the first phase for CSP – acetone. The CSP-acetone-resin mix was then stirred, sonicated for 20 min and 15 min respectively followed by heating at 70 °C for 15 min to remove the acetone that was dispersed in it.[48] This process is generally used to mix nanoparticles into epoxy, but to the best of the authors' knowledge, this type of dispersion process has never been used to disperse CSP into epoxy resin. Fig. 1 shows the stages involved in resin modification.

To evaluate the influence of CSP dispersion technique on the mechanical properties of the composites, two additional composites were fabricated wherein the above said procedure was not adopted. The first among the two additional panels were fabricated by adding the CSP using mechanical stirring. The test results of this composite would allow us to compare and ascertain the influence of the magnetic stirring and sonication against mechanical stirring alone. The second panel was also prepared in the same way as the first but, had an additional heating step where the resin-CSP mix was heated at a temperature of 70 ℃ for 30 min. This allowed us to determine whether heating the resin had any effect on the mechanical properties of the composite. CSP proportions were chosen as 0.5%, 1.5% and 3.0% of the resin based on findings from published literature wherein deterioration of properties was reported with higher filler proportions.[49] The detail designation of all the composites are presented in Table 1.

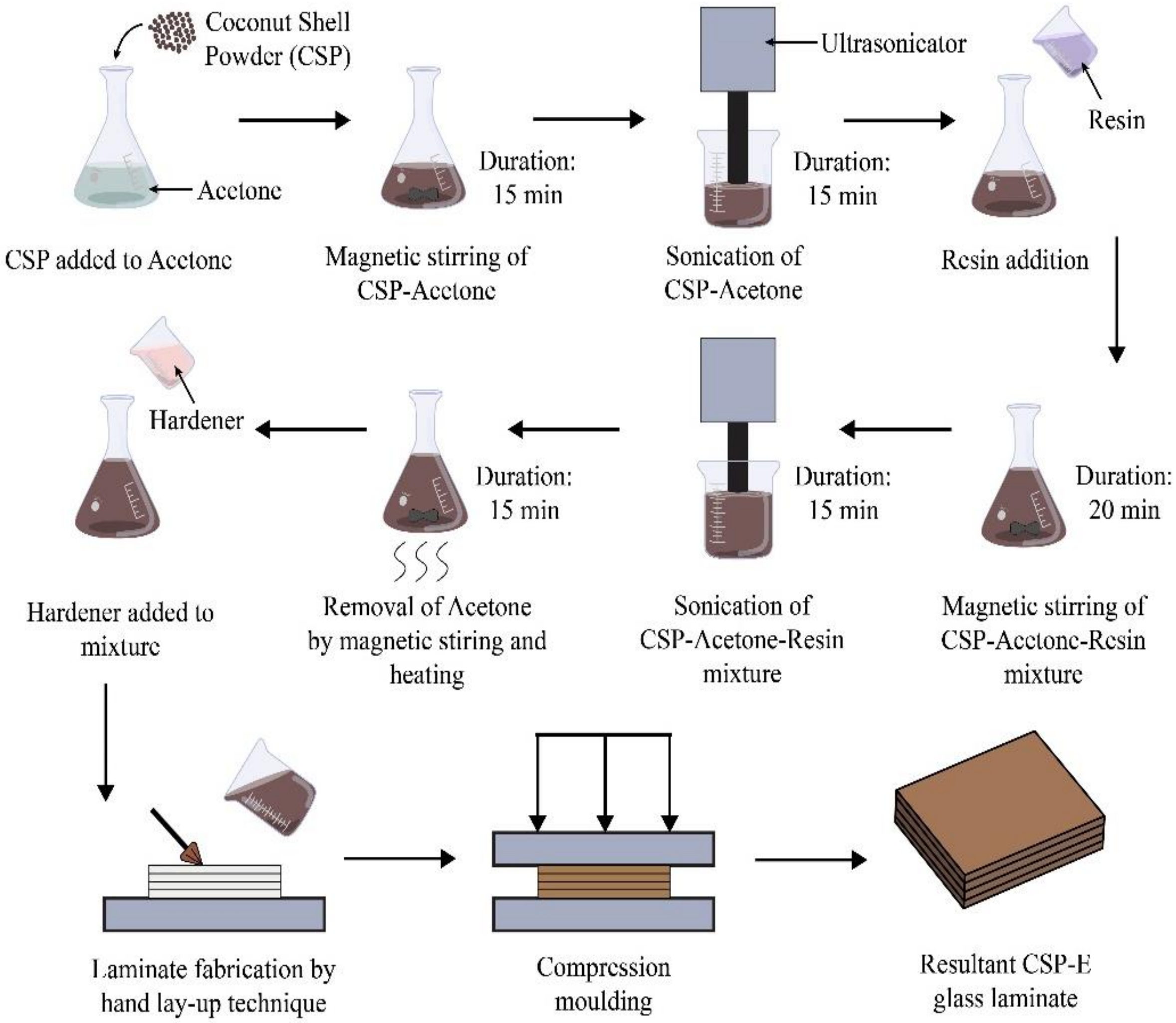

Fig. 1 Graphical representation of methodology followed to modify the resin with CSP.

Table 1. Designation and compositional details of each composite laminate.

Designation	Proportion of CSP	CSP Dispersion Technique
AG	0	Not Applicable
CS05	0.5	Magnetic Stirring + Sonication
CS15	1.5	Magnetic Stirring + Sonication
CS30	3.0	Magnetic Stirring + Sonication
CM15	1.5	Mechanical Stirring
CH15	1.5	Mechanical Stirring + Heating

2.3 Mechanical testing of composites

The composites that were fabricated were tested for their mechanical strength which included tensile, flexural and impact strengths. Tensile and flexural tests were conducted on a Universal Testing Machine (UTM) (Make -Zwick Roell; Model: Z050). Tensile tests were conducted as per ASTM D3039 with the specimen dimensions being 250 mm x 25 mm. The rate of loading was set to a constant of 2 mm/min. Flexural tests were carried out as per ASTM D7264 at a constant rate of loading of 1 mm/min. The specimen length was 96 mm; span length was 80 mm while the width was 13 mm. Impact testing (Izod) was done on notched specimen using a pendulum type impact testing machine (Make -Zwick Roell; Model –HIT 50P). The dimensions of the impact test specimen were 63.5 mm x 12.7 mm. The specimens were subjected to an initial velocity of 5.5 m/s. For evaluation purpose, only tensile tests were conducted on the additional composites- CM15 and CH15. Five specimens were tested for each of the above-mentioned tests. The specimens were cut from the cured composite panels using jig saw cutting.

2.4 Physical, chemical and thermal properties of CSP

Density of the CSP was determined using a digital density balance, Contech Make (Model: CAS 234). Determination of density using density balance involves weighing the CSP pellet in air (W_a) and water (W_l). Knowing the density of water (ρ_l), density (ρ) is determined using Equation 1.

$$\rho = \frac{W_a}{W_a - w_l}\rho_l \tag{1}$$

To determine the chemical constituents of CSP, gravimetric analysis was carried out. Constituents like Cellulose, hemicellulose and lignin were estimated by Neutral Detergent Fiber (NDF), Acid Detergent Fiber (ADF) and Acid Detergent Lignin (ADL). NDF = Hemicellulose + Cellulose + Lignin + minerals. ADF = Cellulose + Lignin + Minerals. Hemicellulose = NDF - ADF; Cellulose = ADF - ADL; Lignin = ADL[50,51]

A Fourier Transform Infra-Red Spectroscopy (FTIR) study was done on the coconut shell powder to understand the presence of functional groups. This characterization technique helps in identifying the presence of specific functional groups in the compounds, which correspond to the constituents of the CSP. The identification of these functional groups can then be extended to check for the presence of phenol, cellulose, hemicellulose, lignin, *etc*. FTIR spectra were recorded in the transmission mode in 4000 –4500 cm-1 regions. Pellets of CSP were made by pressing the powder at a pressure of 5 psi for 15 min. 25 scans of resolution 4 cm-1 were conducted each time on a Fourier Transform Infrared Spectrophotometer (Make –SHIMADZU; Model –FTIR 8300).

Thermogravimetric analysis (TGA) was conducted on the CSP to determine its thermal characteristics. Such an analysis helps in understanding the thermal stability of the particles when subjected to high temperatures. TGA of the CSP involved heating of the powdered sample, in a temperature range of 30 – 900 °C at a ramping rate of 10 °C/min under nitrogen atmosphere. Sample weight of around 10 mg was considered and was performed on a Shimadzu make DTG – 60H model machine as per ASTM E1131.

2.5 Scanning electron microscopy (SEM)

To analyze the shape of CSP and also to determine the various modes of failures in a composite specimen, Scanning Electron Microscope (SEM) (Make –Zeiss; Model –EVO 18 Special Edition) was used. CSP was consolidated into a pellet form and was given a coating of gold-palladium by ion sputtering. Pelletization of CSP was required since powdered samples are not allowed on such SEM and can be seen as an operational limitation. Such coating makes the specimen conductive and also avoids charging of the specimen due to its prolonged exposure to the electron beam. Failed samples of tensile and impact tests were also given the same coating before they were analyzed on the SEM. An accelerating voltage of 15 kV was used.

3. Results and discussion

3.1 Mechanical properties of the composites

3.1.1 Tensile strength

The tensile strength of composites that are prepared with varying proportions of CSP are presented in Fig. 2. It is evident that the addition of CSP to the composite had a positive influence over the tensile strength of the composites.

With the addition of 0.5% CSP, the tensile strength of the composite (CS05) increased to 300 MPa which is about 67% higher than the composite without any CSP in it (AG composite). With 1.5% CSP, the tensile strength increased by another 27% when compared to CS05 composite and by about 111 % when compared to AG composite. The highest tensile strength was obtained for CS30 composites which was 390 MPa. When compared with CS15 composite, though there is an increase in the tensile strength, the increase is insignificant suggesting that the peak performance of the composite is somewhere around CSP content of 3%. Insignificant improvement in strength at higher proportion of CSP can be considered to be an indication of matrix saturation. Hence, any further increase in content of CSP would have an insignificant influence on the strength of the composite and hence was not studied. The standard deviations in the range of 16.55 – 25.3 MPa were observed in the values of tensile strengths of the composite panels. Addition of CSP resulted in increase in the modulus of the composites. It can be seen that with increase in proportion of CSP, the modulus improved progressively. The highest modulus (21.05 GPa) was obtained for composites with 3% CSP in it while the lowest (17.73 GPa) was obtained for composites without CSP. The increase in modulus though not significant, with increase in CSP proportion was observed to be of the order 9% with addition of 0.5% CSP; 6% with addition of 1.5% CSP while with addition of 3% CSP the modulus further increased by about 2%. Increase in modulus is mainly due to the stiff nature of the CSP which results in increased stiffness of the composites. Similar results were obtained by researchers in the past.[52-55] The standard deviations in the modulus was observed to be in the range of 0.65 to 0.83 GPa.

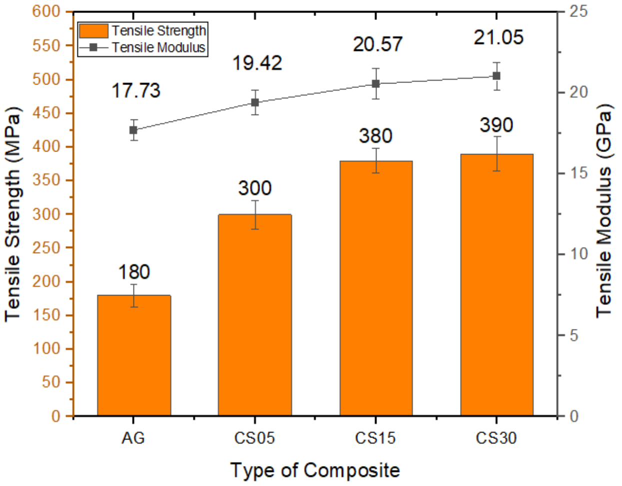

Fig. 2 Variation in average tensile strength of composites.

3.1.2 Flexural strength

The flexural strength of the composites is shown in Fig. 3. The results follow the same trend as seen in the tensile strength results wherein flexural strength improved with the addition of CSP. An increase of 50% in flexural strength over AG composites was observed with the addition of 0.5% CSP, whereas at 1.5% CSP content, the increase over AG composite was of the order of 77%. The absolute increase in flexural strength for CS30 composite over AG composite was about 87% while when compared with CS15 composite the increase was a mere 5%. Standard deviations obtained in the values of the flexural strengths varied from 27.33 – 32.12 MPa. As is the case with tensile modulus, flexural modulus also increased with addition of CSP in the composites. The modulus increased by about 6% with addition of 0.5% CSP; while with addition of 1.5% CSP, the modulus increased by further 6% approximately. The highest flexural modulus was observed to be for the composites with 3% CSP (18.74 GPa), approximately 9% over the 1.5% CSP composites.

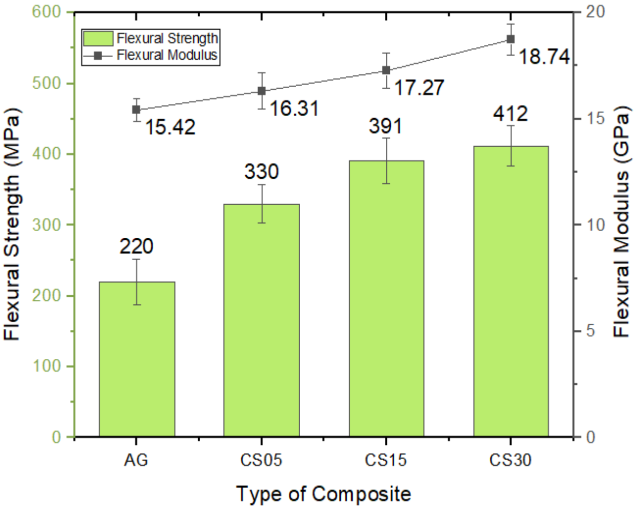

Fig. 3 Variation in average flexural strength of composites

3.1.3 Impact strength

The highest impact strength was obtained for composites with 3% CSP (CS30 composite) which could absorb about 177.39 kJ/m^2 energy. When compared with AG composite which absorbed about 127.17 kJ/m^2, the increase in energy absorbed was about 40%. CS05 composite performed a shade better than AG composite indicating that 0.5% CSP in the composite did not contribute much in terms of increasing its energy absorption capability. The impact test results are presented in Fig. 4. The standard deviations obtained in the values of impact strength varied from 6.14 kJ/m^2 – 10.12 kJ/m^2. The process of impact testing involves two kinds of energies. The first is the initial energy, which entails the energy upon impact. This energy gets transmitted and conceived further in various ways culminating into either total failure of the composite, delamination and/or fiber breakage, *etc*. Alternatively, the energy may be consumed by the resin, specifically it gets distributed over the CSP particles. The measured strengths of the composites exhibit an increasing trend with increment in the CSP concentration. Since, the fabric employed for this study allows the utilization of a third dimension, the total volume of available for embedding increases. This consequently results in an increase in the fiber – to – fiber friction. Therefore, a larger fraction of the impact energy is dissipated in the form of overcoming fiber to fiber friction than total failure of the panel, in the case of CS30.

The results prove the capability of CSP in enhancing the mechanical properties of 3D E-glass composites. It should be noted that, the amount of CSP that has been added is not very significant, but the improvement seen is quite substantial since CSP based composites will definitely have a higher specific strength. The largest granule used in this work had the gauge consistency of 45μm. Particles of smaller size present greater surface area for the matrix spread and can be seen as one of the primary reasons for improvement in the strength of composites.[56,57] Shape of the particles also plays an imperative role in deciding the mechanical properties of the composite. Rounded particles are passed over for angular particles due to their poor gripping capabilities.[58] This study uses angular particles and hence is a deterministic factor in the enhanced properties that are displayed by the composites fabricated. When compared to the tensile strength (405 MPa) reported by Nayak *et al*.[11] for composites fabricated with 4 plies of 3D E-glass fabric, tensile strength (390 MPa) exhibited by CSP composites studied here (2 plies of 3D E-glass fabric) possess tensile strength almost at par with the same fiber weight fraction of 72%. Hence, a higher strength to weight ratio was realized with respect to the composite laminates studied by Nayak *et al*.[11]

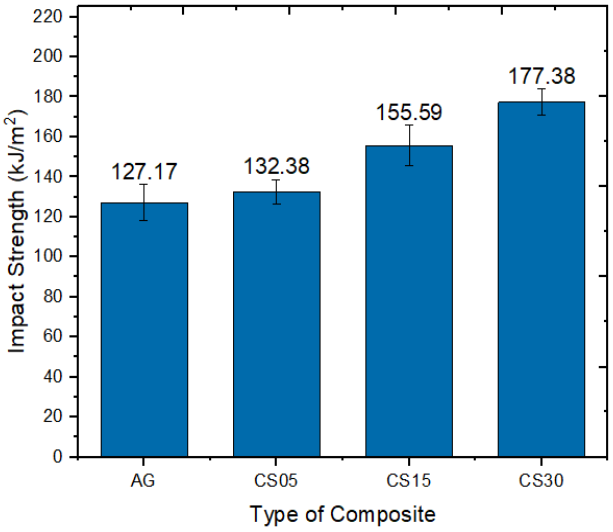

Fig. 4 Variation in average impact strength of composites.

Improvement in the mechanical properties can be largely attributed to the dispersion method that was followed. As had been explained earlier, the CSP was dispersed in the resin using a combination of magnetic stirrer and sonication. The process involved heating as well in order to remove the acetone from the mix. Heating of the resin resulted in the extraction of oil from the CSP which remained trapped in it. Oil extracted from the CSP has about 65% phenol in it.[59] Phenols when used with epoxy resins results in cross-linked polymers. This polymerization process strengthens the material. Also, phenols react with amine, which is the most common hardening agent, resulting in a polymer chain. The curing time at room temperature for the resin that is used in this work is 24h. This additional time facilitates the reaction of phenol with the epoxy and amine to proceed further, thereby improving the properties of the matrix. Moreover, the use of magnetic stirrer and sonication have resulted in better dispersion of the powder in the matrix with reduced agglomeration. An improved dispersion and reduced agglomeration have always been proven to be beneficial for the strength of the composites.[60] The increase in the mechanical properties of the composites with an increase in the proportion of CSP can also be attributed to increased reinforcement. More CSP would in turn mean higher quantities of oil that is extracted for the same amount of resin.

3.1.4 Tensile strength of additional composites

In order to ascertain the influence of heating and dispersion technique on the mechanical properties of the composites, their tensile strengths were compared with those of additional composites namely, CM15 and CH15.The tensile strength of these two additional composites are presented in Fig. 5.

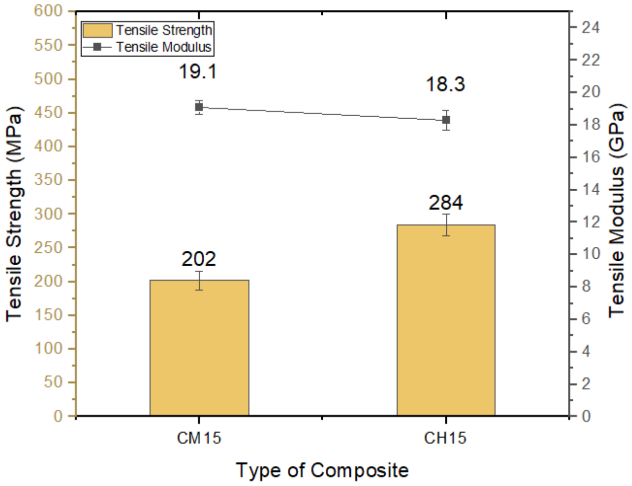

Fig. 5 Variation in average tensile strength of additional composites.

It can be seen that the tensile strength of CM15 is very close to AG composite and is far below the strength of CS15 composite. Though CM15 and CS15 composite have the same proportion of CSP in them, a huge difference in strength signifies that the mixing method has a strong influence on the performance of the composites. When the tensile strengths of CH15, CM15 and CS15 composites are compared, it is evident that heating also contributed to enhancing the strength. CH15 composite performed better than CM15 composite even though they were both prepared purely by mechanical stirring but in the former case, the heating the resin – CSP mix for 30 min resulted in extraction of oil which as discussed earlier helped in improving the properties of the polymer. The variation in the tensile strength in CM15 and CH15 was seen to be 14 MPa and 16 MPa respectively. Tensile modulus of CM15 composites was observed to be around 19.1 GPa with a standard deviation of 0.454 GPa while that for the CH15 composites, the modulus was observed to be around 18.3 GPa with a standard deviation of about 0.612 GPa. When compared with the modulus values obtained for the resin modified composites (CS15), the modulus obtained for the additional composites are higher, further signifying the role of the resin modification process. This can be again attributed to the heating step involved in the resin modification process which improves the property of the matrix by reducing its brittleness and making it tough. For CM15 composites, there was no heating involved while CH15 was prepared by mechanical mixing followed by heating and hence modulus of CH15 was observed to be lower than CM15.

3.2 Physical, chemical and thermal properties of CSP

The density of the CSP was estimated to be around 1.138 g/cc which is close to the density of the Epoxy resin which is around 1.1 g/cc. With CSP being heavier than the resin, the chances of gravity segregation exist though not very pronounced. Large difference in the density values of the resin and CSP would have resulted in pronounced gravity segregation making the composite unsound. With CSP having density close to that of the epoxy matrix, maintaining a uniform dispersion was not very difficult.

From the gravimetric analysis it was observed that the cellulose content was in the range of 50 to 52%; hemicellulose in the range of 36 to 38% and lignin in the range of 8 to 10%. Cellulose helps in better binding between the constituents. With CSP having high cellulose content, it not only helped in getting a good bonding between the constituents but also contributed in bearing some proportion of the load coming over the composites thereby acting as secondary reinforcements.

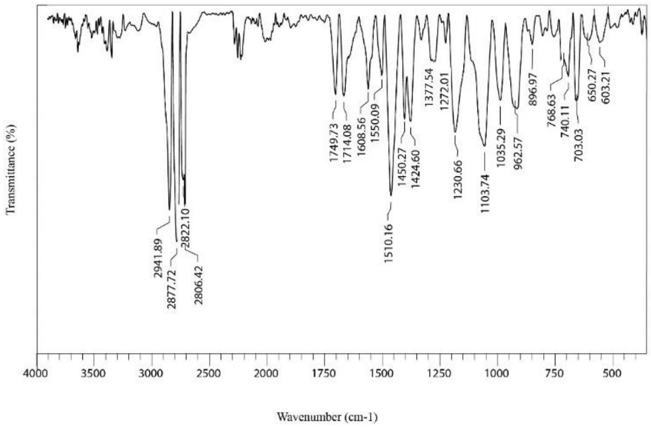

Fig. 6 The FTIR spectroscopy of CSP.

The absorbance and their relation to various functional groups for CSP is presented in Table 2. The FTIR spectroscopy (Fig. 6) indicates strong bands in the region 2941.89 cm^{-1} and 1035.29cm^{-1}. This region is attributed to cellulose. Cellulose adds to the tensile strength and improves the structural integrity of the material. The presence of phenol at 1749 cm^{-1} in the FTIR spectrograph is a positive indication since it is known to undergo polymerization with epoxy and amine while fabricating the composite to form a mix that is known for its high strength characteristics. This region is attributed to cellulose. Cellulose adds to the tensile strength and improves indication since it is known to undergo polymerization with epoxy and amine while fabricating the composite to form a mix that is known for its high strength characteristics. The presence of pyroligneous acid is ascertained by the carbonyl stretching in the bands between 1100 and 1230 cm^{-1}. The presence of lignin and hemi-cellulose is characterized by recurrence of peaks repeatedly over the span of the FTIR spectroscopy. It is an imperative component of every naturally sourced, plant–based reinforcements.

The thermogravimetric results (Fig. S2) show that the CSP is thermally stable till 230 °C since the first significant drop in mass was observed at around that temperature. The associated mass loss at that temperature was of the order of 2.3%. Since there was heating involved during the resin modification process, chances of any decomposition of CSP is ruled out since the heating temperature (70 °C) is way below 230 °C.

Table 2. FTIR bands for analysis of coconut shell powder.[61]

System	Symbol	Wave Number (cm^{-1})
Cellulose	C-H stretching	2941.89
	C-O stretching	1035.29
Hemicellulose	C=O stretching (carbonyl ester)	1726
	C-O stretching of acetyl	1252
Phenol	Aromatic ring	1749.73
Lignin	-C=C- stretching (aromatic)	1608.56 1550.09 1510.16
Pyroligneous Acid	C=O carbonyl stretching	1230.66 1103.74

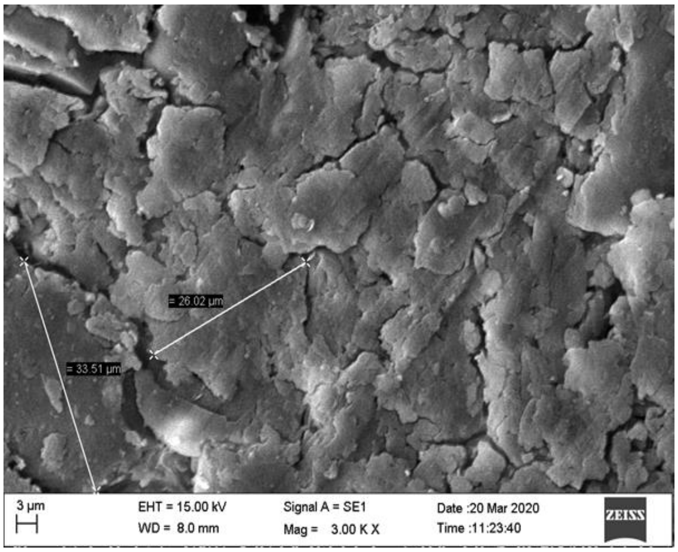

Fig. 7 SEM micrograph of CSP pellet.

3.3 Scanning electron microscopy (SEM)

SEM micrograph of CSP is shown in Fig. 7. The powder was consolidated in the form of a pellet. From the micrograph, it can be seen that the shape of the particle is random and irregular. The particles are solid, non-porous and non-fibrous and have a size that is less than 45 µm. Irregular shapes of particles help in better interfacial bonding and in better transfer of stress from the matrix.[62] Particles being solid and non-porous provide them with the required rigidity to act as reinforcing member along with glass fibers. As far as the distribution of particle and their size is concerned, it appears to be a mix of particles of different sizes and a large proportion of them, having a size that is less than 45 µm.

Figure 8a shows the SEM micrograph of the CS05 impact specimen. The cross section as well as the outer curved surface of a single glass fiber is clearly visible. On the curved surface, deposition of CSP is visible in the form of arbitrarily shaped, flat particles. This conforms with the findings obtained through the SEM micrograph of the pelletized coconut shell powder as discussed hitherto. Fig. 8b shows the SEM micrograph of the CS15 impact specimen. In this image, a collection of glass fibers can be seen, as well as a strand of binder yarn, detached and entangled amongst the fiber strands. The exposed surfaces of theses fibers are coated with CSP-modified resin, indicated by the slight protrusions seen along their length. The SEM micrograph of the CS30 tensile specimen is displayed in the Fig. 8c. Where the distribution of CSP particles is sparse and moderately sparse for specimens CS05 and CS15 respectively, the distribution appears to be conspicuously denser for specimens with CS30. When a comparison is drawn with respect to the presence of CSP particles on the lateral surface of the fiber, it can be seen that the CS30 shows an increased amount of filler particles. This shows that with increasing filler percentage, the coating of the filler on the fiber surface increases. This thorough coating of the fibers can be construed as a reason for the improved mechanical strength of CSP based composite as irregular shaped particles interlock with each other as well as with other constitutes that make up the composite namely, matrix and the fibre. Higher forces are required to separate the locked particles and can also be seen as one among the various reasons for improved performance of CSP dispersed composites.[62] With irregular CSP coating on the fibres, higher pull-out forces are required to separate the fibres form the matrix and this transforms not only higher impact resistance but also in higher resistance to tensile and bending forces.[63]

Micrograph shown in Fig. 9a demonstrates brittle failure of the AG specimen, indicated by the broken fragments of matrix. This is not visible in the case of CSP filled composites (Figs. 9b to 9d). Conclusively, it can be said that the addition of CSP into the matric results in the reduction of the brittle character of the epoxy resin.

Different modes of failure were observed in the various composite panels subjected to tensile testing. These comprised of delamination, fiber pull – out and snapping of fibers. Fiber pull – out can be identified through minute holes in the cross-sectional view of the panel amidst a bunch of exposed, disoriented fibers. The SEM micrograph in Fig. 10a displays the fractured surface of CS05 impact specimen. The

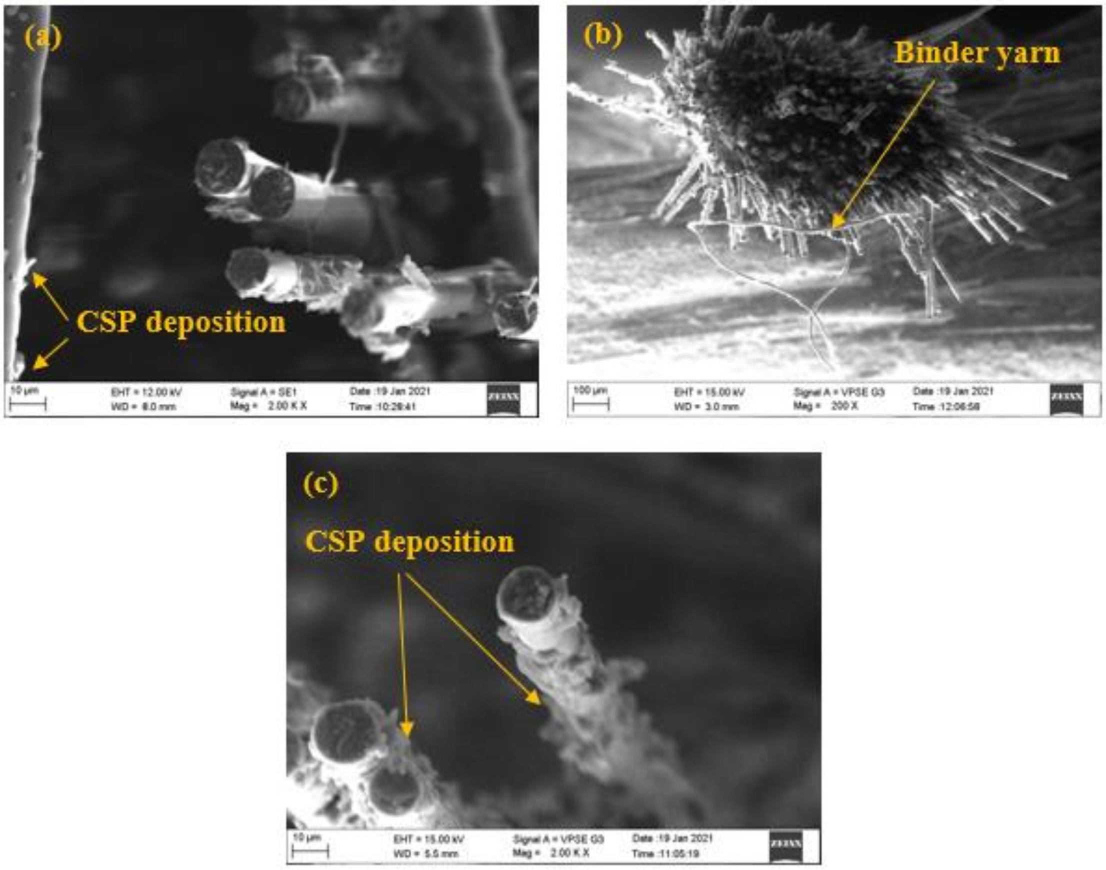

Fig. 8 SEM micrographs of a) CS05 - Impact b) CS15 - Impact c) CS30 – Tensile.

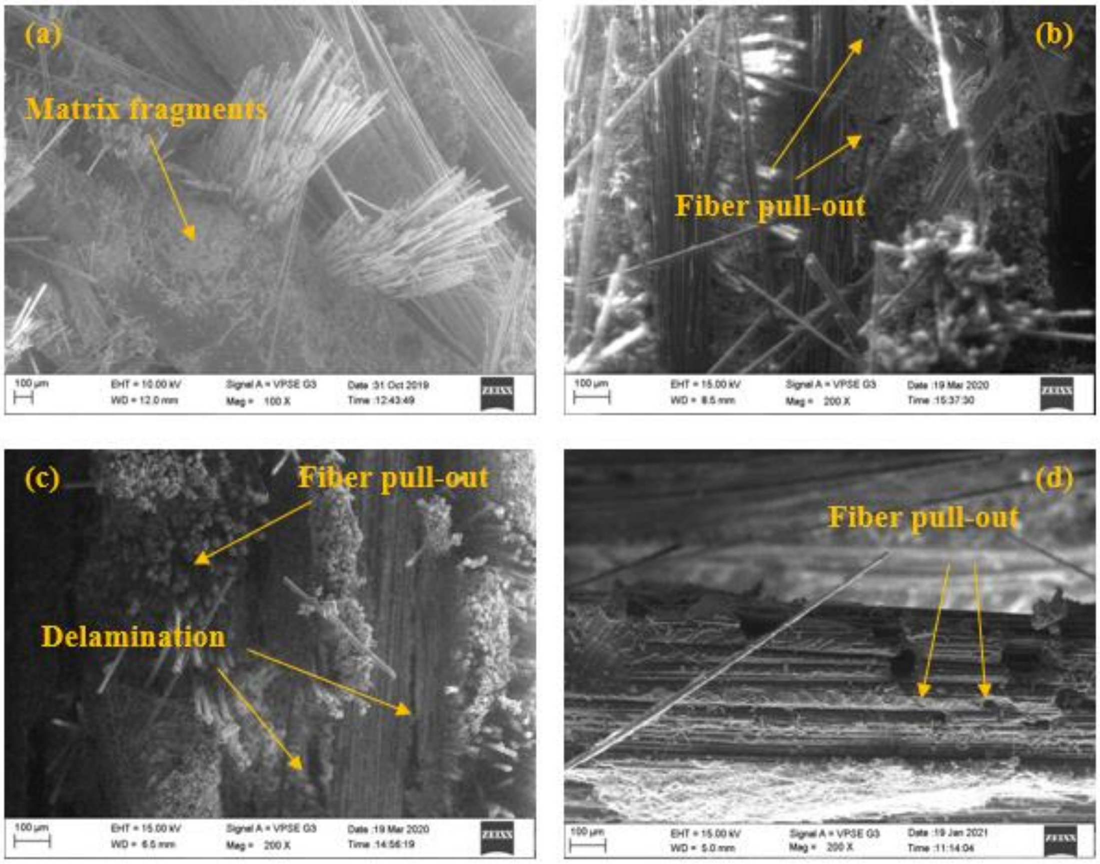

Fig. 9 SEM micrographs of tensile a) AG b) CS05 c) CS15 d) CS30.

disoriented and broken fibers can be clearly categorized into warp and weft directional fibers. When the test specimens undergo impact testing, the composite is to absorb the impact energy, the unique characteristic of E-glass fibers is that binder yarn is responsible to hold the warp and weft fibers. Additional energy is required to overcome the binder yarn, therefore increasing the impact strength of the composite. This phenomenon validates the 17.1% increase in impact strength from 132.38 kJ/m^2 to 155.59 kJ/m^2. Similarly, 14% increase from 155.59 kJ/m^2 to 177.39 kJ/m^2, as the percentage of filler increases from 1.5% to 3%. The binder yarn, traditionally oriented in the z direction, can be seen to be displaced across the fractured surface in Fig. 10a.

The breakage of the binder yarn leads to separation of the layers that is known as delamination. Unlike the impact specimens for CS30 (Fig. 10b), where little to no delamination is observed on the fractured surface, delamination contributes as one of the major failure mechanisms for the impact specimens for CS05 and CS15. When the interfacial bond between the fibers and matrix is lacking, small holes appear across the cross-sectional surface as seen in the micrograph (Fig. 10c), that correspond to the voids left by fibers that were pulled out. Fragments of broken matrix are scattered on the fractured surface of the CS15 impact specimen as seen in Fig. 10b which can be attributed to brittle nature of the epoxy. The sudden shock from the impact test caused the fibers to snap into fragments. The failure of this specimen is credited to snapping of binder yarn, delamination, matrix cracking and fiber breakage.

As mentioned earlier, the CS30 specimens appear be more thoroughly coated than the other specimens that resulted in better interfacial adhesion and bonding between the layers. This signifies good infusion of the particles and resin through the gaps between the tightly packed fiber yarns, during the fabrication stage. In the tensile tests since the force is exerted in the longitudinal direction, the failures seen are mainly fiber pull-out and fiber breakage.

The good binding of coconut shell particles on the surface of the fiber clearly shows a positive impact on the mechanical properties of the composite with respect to all glass panels. The percentage increase in the tensile strength as the filler percentage increases tends to be reducing.

From CS05 to CS15 there is an increment of about 27% in the tensile strength from 300 MPa to 380 MPa. However, from CS15 to CS30 there is only 2.6% increment *i.e.*, 380 MPa to 390 MPa. When compared against the SEM images, this might indicate an approaching optimum percentage of the coconut shell powder. Additionally, it can also be observed that the excessive coating of the filler on the fiber surface is not malignant to the properties of the fiber.

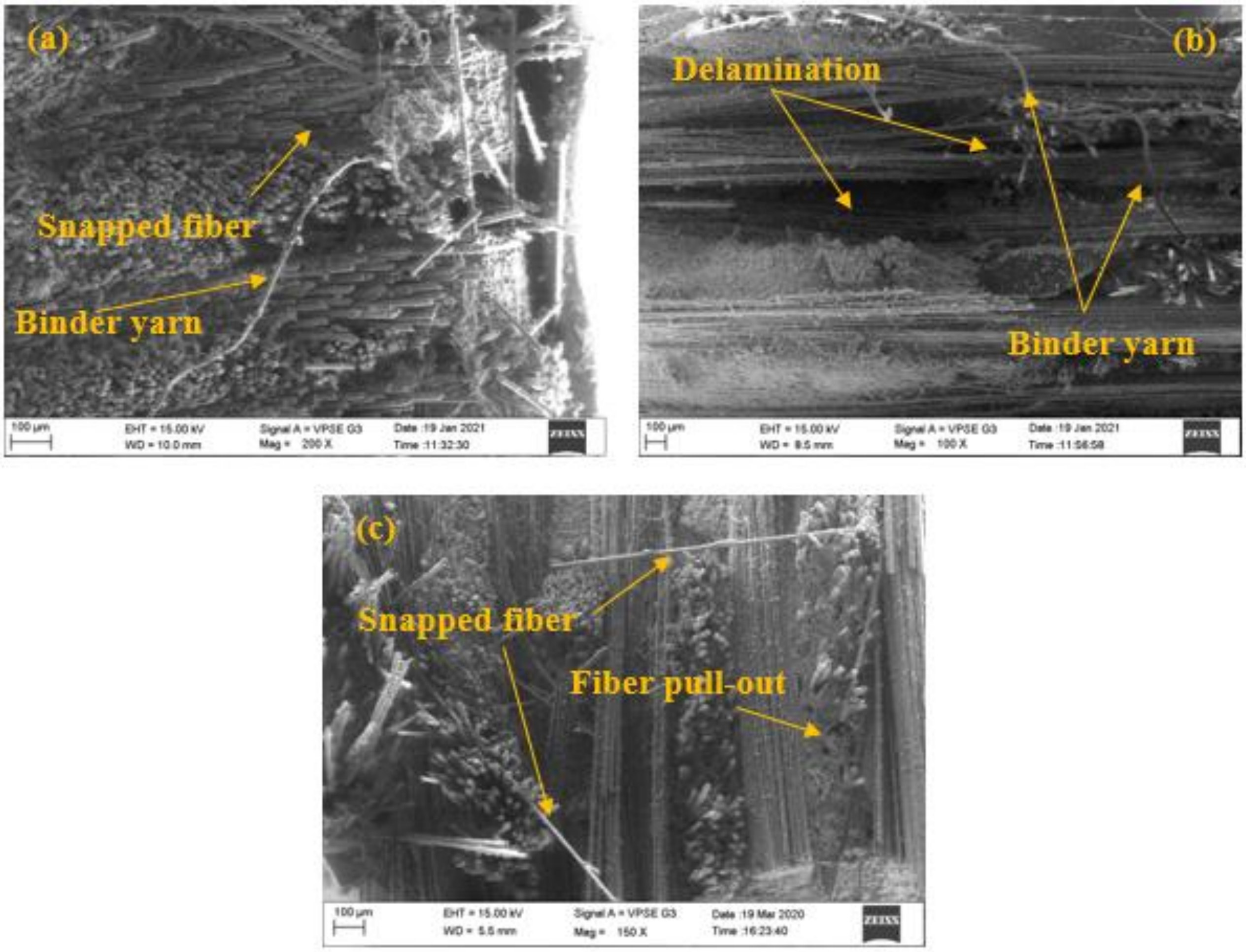

Fig. 10 SEM micrographs of impact a) CS05 b) CS15 c) CS30 test specimens.

4. Conclusion

Composites with 3D orthogonally woven E-glass fabric were fabricated with different varying proportions of Coconut Shell Powder (CSP). Before fabrication, the resin was modified with the acetone-CSP mix using magnetic stirring and sonication. Additional composites to check for the influence of the dispersion technique were also fabricated. The results indicated a significant improvement in the mechanical properties of the composites. The highest tensile strength of 390 MPa, flexural strength of 412 MPa and impact strength of 177.39 kJ/m2 was obtained for composites with 3% CSP. The lowest tensile strength of 180 MPa, flexural strength of 220 MPa and impact strength of 127.17 kJ/m2 was reported for composites without any CSP. The phenol that was present in the CSP was primarily responsible for the enhancement of the composite's strength which was confirmed using the FTIR spectrograph. SEM revealed that the CSP was non-porous and solid with an irregular shape. Delamination, fiber breakage, fiber pull-out, shearing of the fabric and matrix cracking were the major modes of failure as observed from the SEM micrographs of the failed specimen.

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Conflict of Interest

The authors declare no conflict of interest.

Supporting information

Applicable.

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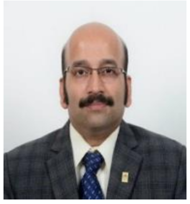

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