

DOI: <https://doi.org/10.24425/amm.2022.137768>A. SUJIN JOSE¹, A. ATHIJAYAMANI², S.P. JANI^{3*}, V. MAGO STALANY⁴, M. ADAM KHAN⁵

INVESTIGATIONS ON MECHANICAL PROPERTIES OF BIO-WASTE MICRO PARTICLES REINFORCED PHENOL FORMALDEHYDE COMPOSITES

A characteristic study on the phenol formaldehyde (PF) composite is carried out based on the micro level bio waste particles such as wood sawdust (WSD) and coir pith (CP). Composite is characterized by mechanical properties such as tensile, flexural and impact at different percentages of particles (0-50% by weight) to find out the optimum percentage of particle loading to get the maximum properties. The WSD/CP/PF hybrid composite is also prepared by two different methods i.e., one: one (1:1) ratio and rule of mixture. The first method (1:1) is used to find out the optimum level of hybrid particles loading to get the maximum properties. But, the second method is followed to find out the weight percentages of particles influencing the properties of resulting composite. The results show that the mechanical properties of WSD/PF are higher than CP/PF composite in the entire particle loading. The optimum particle loading to get the maximum properties is 40 wt.% in CP/PF composite, whereas for WSD/PF composite are at 30 wt.%. The hybrid composite (1:1) also gives the maximum properties at 30 wt.%. Moreover, the hybrid composite (20WSD/10CP) prepared by rule of mixture showed the highest mechanical properties compared to the other particle loading. It is identified that the WSD particles are most influencing the properties of PF composites than the CP particles. Fractographic study was performed using scanning electron microscope to examine the failure mechanism of the composite specimens.

Keywords: Wood Sawdust Particles; Coir Pith Particles; Hybrid Composites; Mechanical Properties; The rule of Mixture Method

1. Introduction

Currently, the bio-based natural cellulosic fiber and particle Reinforced Polymer composites are becoming a popular material and the substitute of synthetic fibers for a wide range of industrial and structural applications due to their high specific properties, low cost and low density, etc. [1-4]. In India, the bio-waste cellulosic particles as bio residues like rice straw, rice husk, wheat straw, cotton gin waste, corn stalks and cop, groundnut shells, coconut shells, wood sawdust, sugar cane trash, coir pith, etc. are creating a problem to their industries due to the no way for the disposal, i.e. increasing transportation and storage costs [5-7]. Among these, the waste coir pith particles, a by-product spongy material obtained after extraction of fibers from the husk, are considered as a bio waste, creating serious environmental pollution in the East and Southern region of India [8-10]. The WSD particles are also considered as bio-waste materials, creating an

environment and health problems. The fertile and useful lands are occupied due to the increasing accumulation and dumping of these saw dust every year. They spoil the quality and health of useful lands by contaminated water due to leaching and microbial growths [11-13]. The natural bio waste particulates are used as a potent reinforcing agent in polymer composites [14]. The tensile strength and other mechanical properties of polymer composites have been increased by the addition of coir and wood dust particles [15]. Mechanical properties with fiber were increased linearly with fiber loadings [16]. The bad odor created by the waste WSD particles is creating serious health problems for human living and the nearest village people. To overcome these problems created by these bio waste particles, they should convert as a useful material or they should use for making other materials like as a reinforcement for polymer composite materials.

This study is started to create awareness for utilization of bio-agricultural waste as reinforcing agents for polymer

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composite because of their abundant availability. Moreover, the purchase cost of these materials is very low compared to others. Due to lack of sufficient literatures about the utilization of bio-agricultural waste particulates as reinforcing agent in resole type phenol formaldehyde matrix, this attempt is carried out on the mechanical properties of WSD/CP particles reinforced phenol formaldehyde hybrid composites. The effects of particle loading on the mechanical properties of CP/PF, WSD/PF, WSD/CP/PF hybrid composite prepared by 1:1 ratio and WSD/CP/PF hybrid composite prepared by the rule of mixture method are studied in a detailed manner. Finally, a comparison is carried out among the mechanical properties of these four composites.

2. Materials and methods

2.1. Materials

The WSD particles of the variety of woods are taken from the waste Godown of the Kumar Timber and Sawmill, Karai-kudi, Tamilnadu, India. After collection, the wood dust particles are cleaned by an air blower and then, separated based on their size using a sieving machine. The raw coir pith particles with non-uniform is collected from the Coir Industry, Sozhavanthan, Tamilnadu, India. Then, they are also cleaned by an air blower and separated by sieving machine based on their size. Before crushing and sieving both particles (with non-uniform size), the raw particles are washed with 2% soap or detergent solution at 50°C for 1 h, then washed with distilled water and finally dried in an oven at 70°C. The cleaning and separating process are carried out in our composite laboratory.

The resole type Phenol formaldehyde liquid resin (trade name: Axotherm Industry, Bengaluru) was used with the divinylbenzene as cross-linking agent and hydrochloric acid as acidic catalyst. All the chemicals were procured from the POOJA Chemicals, Madurai, Tamil Nadu, India.

2.2. Fabrication of composites

Composites were fabricated by casting using mould with the size of 150 × 150 × 3 mm dimension at room temperature in the form of lamina. Before applying resin, the mould was sprayed with mould releasing agent to ensure easy removal of cured composite plate without damage. A mixture of resin and particles (ratio of 100:2.5:1.5) was prepared by a mechanical stirrer for 30 minutes and poured into the mould. The power of the mechanical stirrer is a few hundred to a few thousand watts with the speed of 100 to 1000 revolution per minute. Then, the mould was closed and allowed to cure at room temperature for 48 hours. After curing, composites were removed from the mould and cut into composite specimens (for tensile and flexural test: 150 × 20 × 3 mm), for impact test (20 × 10 × 3 mm) specimens as per ASTM standard for mechanical characterization.

2.3. Testing of composite specimens

The tensile property values of composite specimens were measured according to ASTM D638-10 (ASTM D638, 2010) on an FIE universal testing machine (UTE 40 HGFL). The flexural property values were measured according to ASTM D790-10 (ASTM D790, 2010) on the same testing machine. According to ISO 180 (ISO 180, 2000), the impact strength values of composite specimens were measured. Five specimens were tested for each combination to get the average value.

2.4. Scanning Electron Microscope (SEM) study

The surface of the fracture surface of composite specimens was observed under the Scanning Electron Microscope (Model Hitachi S-3000N). Prior to process, composite specimens were gold coated with a sputter coater using ultrathin film.

3. Results and discussion

Mechanical properties of WSD/PF, CP/PF, WSD/CP/PF (1:1) and WSD/CP/PF (rule of mixture) based on the particle loading is presented here. The optimum particle loading to obtain the best combination mechanical properties was identified for these four categories of PF composites. The variation of mechanical properties of composites for different particle loading was studied in a detailed manner.

3.1. Effects of CP particles addition on the mechanical properties of PF composite

The tensile property values of the CP particles reinforced and neat resin sample recorded during the tests are presented in Fig. 1a. Fig. 1a shows that initially, the incorporation of coir pith particles reduces the tensile properties of PF composite and then, enhances the properties of PF composite on further addition of CP particles. It is due to the reason that in lower particle loading, the interface zone of the resulting composites is unable to play major role in transferring the applied load between the particle and the matrix. Another reason is available of the ineffective surface area of the particle to contact with the matrix. The tensile strength of composite was increased up to 40 wt.% of particle loading and then decreased. It can be observed from this that the addition of 40 wt.% of particles enhances the strength and modulus of PF composites in higher range due to the effective surface area of particles and the effective interface zone between the particles and the matrix. Similarly, the modulus of composite also showed an increasing trend up to 40 wt.% of particles. Generally, the toughness of polymer composites may increase with the addition of particle reinforcement. The plasticizing effect increases in particle-reinforced polymer composites due to the presence of cellulosic particles.

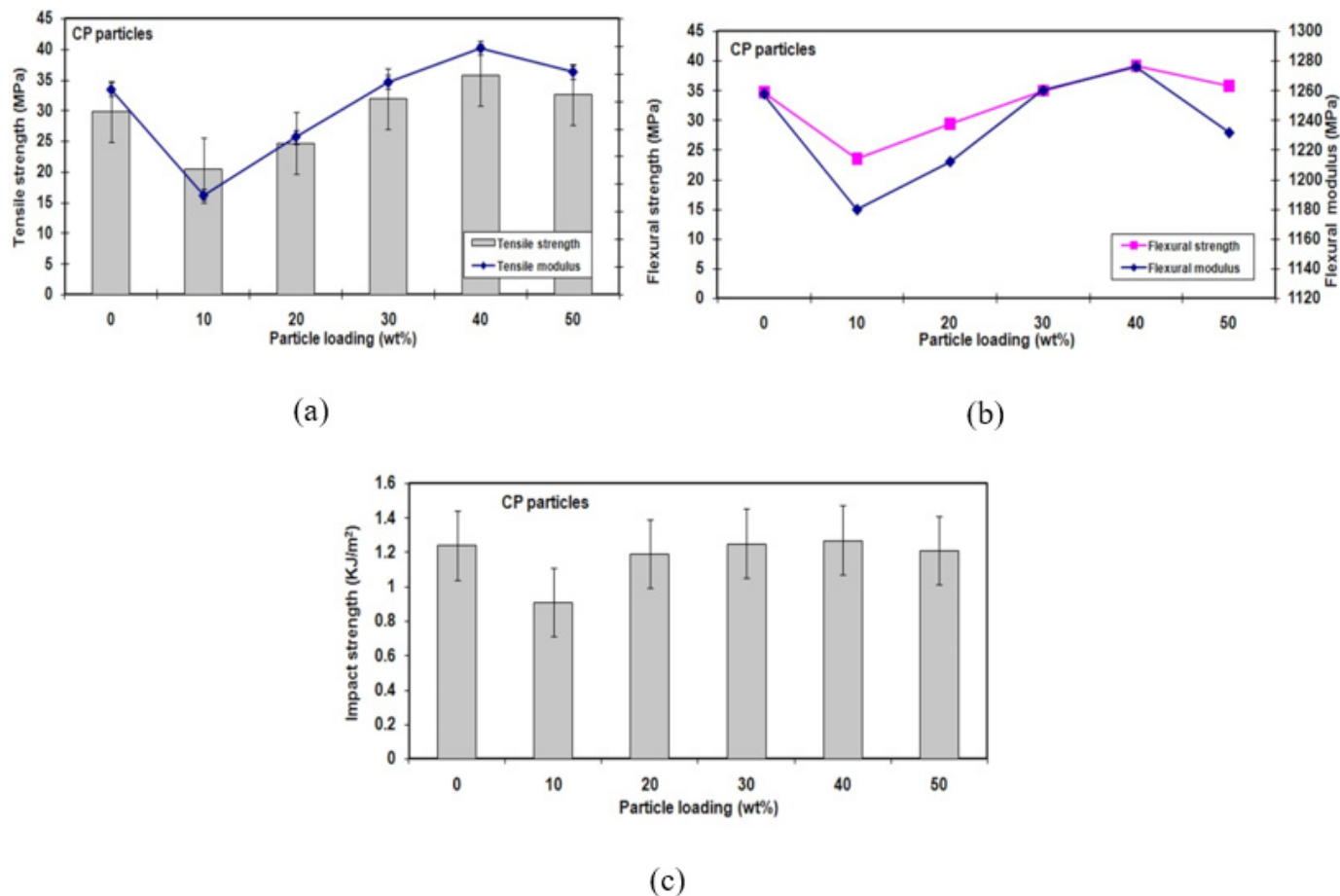


Fig. 1. Variation of (a) tensile properties, (b) flexural properties, and (c) impact strength of CP/PF composites

Fig. 1b shows flexural properties of CP/PF composites at different particle loading. It can be seen that the flexural properties of PF composite also decreased with the initial addition of CP particles and then, shown an increasing trend with the further addition of CP particles. It was also observed that the flexural strength got increased with particle loading, and the maximum value was also for 40 wt.% of particle loading and thereafter decreased. In the case of lower particle loading, the particles could not have the efficient surface area for contact with the polymer matrix, which results in failure of composites under low strain. Similarly, at higher particle loading (50 wt.%), the effective stress transfer between the particle and the matrix is not possible due to dumping of particles in a particular location. Therefore, the optimum range (40 wt.%) of particle loading was necessary for effective reinforcement in polymer matrix. The flexural modulus was also increased up to 40 wt.% of particles. It may also be due to the plasticizing effect of composites due to the addition of CP particles.

Fig. 1c shows the impact strength of the CP/PF composite for varying particle loading. The impact strength of CP/PF composite reached the strength of the neat resin sample at 30 wt.% and gets the maximum value at 40 wt.%. The impact strength of 50 wt% composite was slightly lower than 30 wt.% of composites. It can be observed that enhanced particle-matrix compatibility was created in optimum particle content

(40 wt.%), which leads to improved transfer of stress from the polymer matrix to the particles and thus, the impact strength was found to increase. In general, the properties of the polymer composite reinforced with the particles are strongly influenced by the interfacial adhesion between the particles and the matrix. The strength of the interfacial zone plays an important role in the fracture of particle reinforced polymer composites. For effective stress transfer through the particles, the effective surface area to contact with the polymer matrix is necessary in particle reinforced polymer composites. But, at the particular range of particle loading, the brittleness of composites is increased due to the insufficient polymer matrix to wet the particles, results in very quick failure of composite.

3.2. Effects of WSD particles addition on the mechanical properties of PF composite

The effect of WSD particle loading on the tensile properties of PF composites is presented in TABLE 1. It was observed that the tensile strength initially decreased with the incorporation of WSD particles, i.e. lower particle loading and then increased with the further incorporation of WSD particles.

Composite reached the tensile strength of the neat resin sample at 20 wt.%. From 10 to 50 wt.% particle loading, the

TABLE 1

The mean value with the standard deviation for the mechanical properties of WSD/PF composite based on particle loading

Particle loading [wt%]	Tensile strength [MPa]	Tensile modulus [MPa]	Flexural strength [MPa]	Flexural modulus [MPa]	Impact strength [MPa]
0	29.8 (±3.3)	1168.4 (±48.2)	34.7 (±4.1)	1257.4 (±50.3)	1.24 (±0.08)
10	26.6 (±2.9)	1131.2 (±40.3)	29.2 (±3.4)	1208.6 (±46.4)	1.18 (±0.07)
20	32.5 (±4.2)	1189.9 (±50.4)	35.7 (±4.8)	1259.4 (±48.6)	1.25 (±0.14)
30	39.8 (±5.1)	1213.5 (±47.9)	44.8 (±5.3)	1311.3 (±39.2)	1.29 (±0.19)
40	36.7 (±4.6)	1182.1 (±49.1)	38.5 (±4.2)	1294.5 (±41.5)	1.24 (±0.13)
50	32.1 (±3.8)	1143.6 (±44.8)	34.9 (±3.9)	1262.8 (±42.9)	1.20 (±0.10)

tensile strength reached the maximum value at 30 wt.%, which is 39.8 MPa. Therefore, it is proved that composite having the particle loading of 30 wt.% are stiffer and tougher than the composites with lower and higher particle loading and also, with the neat resin sample. The effective surface area (better interaction between the particle and the matrix) is essential in the case of particle reinforced polymer composites because, in theory, the strength of the polymer composite reinforced with the particles is lower than that of the unreinforced neat resin sample. It is indicated by the inability of particles to transfer the applied load during tests. TABLE 1 shows the opposite results for the theory of the tensile strength of polymer composites reinforced with the particles. It may be due to the interaction between the particle and the matrix, resulting in a better stress transfer between the particles and the matrix. In further addition of WSD particle loading (beyond 30 wt. %), the tensile strength of composite decreased. This may be due to the poor interaction between the particle and the matrix and the particle agglomeration.

The effect of particle loading on the tensile modulus of WSD/PF composites is given in TABLE 1. From the table, it is clear that the tensile modulus values decreased by the initial addition of the particles (10 wt.%) and then, increased with the further addition (20 wt.%). It reaches the maximum value at 30 wt.%, which is 3.86% higher than the neat resin sample. It can be observed that the mechanical properties of polymer composites reinforced with the particles mainly depend on the strong and fully bonded interface between the particle and the matrix i.e. the strength of the interface. Due to this, the stress from the matrix can be easily transferred to the particles.

TABLE 1 also shows the variation of flexural properties of WSD/PF composites for different particle loading. Initially, the flexural properties decreased with the addition of WSD particles and then increased with the further addition of particles. The flexural strength of composite also reaches maximum value when the particle loading was about 30 wt.% (44.8 MPa). This maximum value of flexural strength may be due to the existing of the better interaction (static adhesion strength and interfacial stiffness) between the particle and the matrix. Beyond 30 wt.% particle loading, the flexural strength of the composite decreased. This may be due to the aggregation of particles with a lower surface area by poor dispersion of particles, which reduces the interfacial adhesion between the particles and the matrix. Finally,

it initiates the composite failure due to the reduced interaction between the particles and the matrix.

TABLE 1 shows the effect of different particle loading on the flexural modulus of WSD/PF composites. From the table, it is clear that the results of flexural modulus of WSD/PF composite show a similar trend with the flexural strength which initially decreased with the particle loading and then increased with increasing particle loading. The maximum flexural modulus was identified at 30 wt% of particles, after which it decreased. The reason for this trend is strong tendency of particles to join each other i.e. particle-to-particle interaction (particle agglomeration), which reduce the particle and the matrix interaction. Due to this, the strength and modulus of composite are reduced.

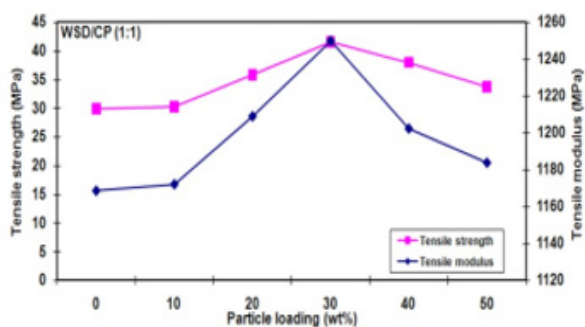
The impact strength of WSD/PF composite for five different particle loading is also presented in TABLE 1. It can be seen that the impact strength of WSD/PF composite shows an identical trend of tensile and flexural strength which initially decreases with the addition of particles and then increases and also gets the maximum value at 30 wt% of particles. The impact strength of 50 wt% composite was 1.20 KJ/m², which was 6.98% lower than the 30 wt.% composite.

3.3. Effects of hybrid WSD/CP particle loading (in 1:1 ratio) on the mechanical properties of PF composites

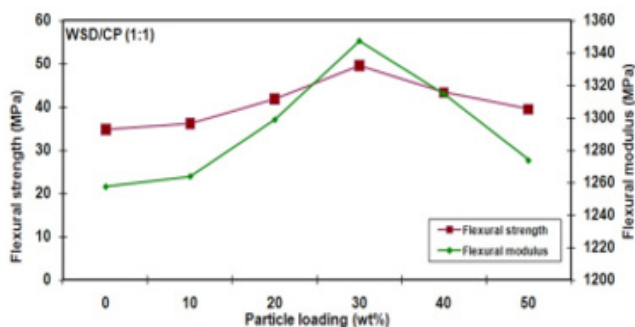
To study the hybridization effects of particles on the mechanical properties of PF composite, the WSD/CP/PF hybrid composites were prepared in 1:1 (50:50) ratio for five different particle loading (10, 20, 30, 40 and 50 wt.%) and their mechanical properties were evaluated based on the particle loading.

Fig. 2a shows the variation of tensile properties of WSD/CP/PF hybrid composites for different particle loading. The figure shows that the tensile strength of hybrid composite reached the tensile strength of the neat resin sample at 10 wt.%. The addition of hybrid particles to the PF matrix increases the tensile strength of the composite up to 30 wt.% of particles and then decreased with the further addition of particles. An improvement of 39.26% was obtained at 30 wt.% composite when compared with the neat resin sample. This may be due to the better interaction of the particles and the matrix, as it is shown in Fig. 3a. The addition of 40 and 50 wt.% of particles reduced the tensile strength of the

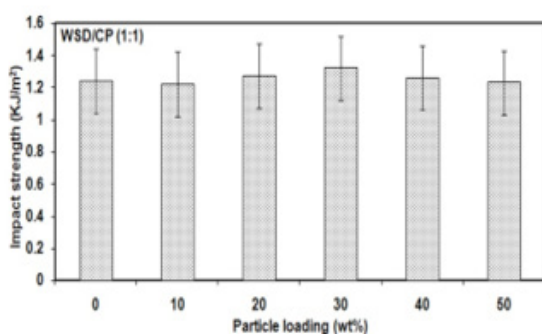
composites. The tensile strength of the 50 wt.% composite was 19.04% lower than the 30 wt.% composites. It may be due to the aggregation of particles by particle-to-particle interaction, what is clearly shown in Fig. 3b,c (formation of agglomeration). The tensile modulus of hybrid composites was also increased up to 30 wt.% of particles and then decreased. The maximum tensile modulus of 1249.6 MPa was obtained at 30 wt.% of particles which is 6.95% and 5.58% higher than neat resin sample and 50 wt.% composite, respectively.



(a)



(b)



(c)

Fig. 2. Variation of (a) tensile properties, (b) flexural properties, and (c) impact strength of WSD/CP/PF composites prepared by 1:1 ratio

The variation of flexural properties of hybrid composites for five different particle loading is given in Fig. 2b. It was clear from the figure that the results of flexural tests also show the identical trend with the tensile properties. The flexural strength increased with the increase of particle loading up to 30 wt.% and then dropped. 30 wt.% composite showed 42.94% incre-

ment compared to the neat resin sample. Composite having the particle loading of 50 wt.% shows the lower flexural strength than the 20 wt.% composite. The reason is that poor wettability of particles by the matrix is created due to higher possibility for the particle-to-particle interaction. The flexural modulus values also increase with increase of particle loading up to 30 wt.% and then decreased. The flexural modulus of 30 wt.% composite is 6.59% higher than the 10 wt.% composite and 7.13% higher than the neat resin sample.

The impact strength of hybrid composites at five different particle loading is shown in Fig. 2c. The impact strength of hybrid composites increased initially and decreased for 40 wt.% of hybrid particles, as shown in Fig. 2c. The maximum impact strength was identified at 30 wt.% of hybrid particles which was 6.45% higher than the neat resin sample. The increase in impact strength of hybrid composite may be due to the better interaction between the particles and the matrix. On the other hand, the decrease of impact strength at higher particle loading may be due to the weak interfacial adhesion between the particles and the matrix.

3.4. Effects of hybrid WSD/CP particle loading (the rule of hybrid mixture) on the mechanical properties of PF composites

Mechanical properties of WSD/CP/PF hybrid composite prepared by rule of mixture method were measured based on the relative weight fraction of particles and presented in Fig. 3. The total reinforcement loading of hybrid composite was maintained at 30 wt.% of particles because WSD/PF composite showed the maximum property values at 30 wt.%. On the other hand, in the case of CP/PF composite, the maximum property values were obtained at 40 wt.% of particles. But, the property values of WSD/PF composite at 30 wt.% were higher than the CP/PF composite at 40 wt.%.

Fig. 3a shows the tensile property values of hybrid composite for four different particle loading. The maximum tensile strength was observed at 20WSD/10CP/PF hybrid composite, which is 9.79% and 36.99% higher than the virgin WSD/PF and CP/PF composites respectively. It may be due to the plasticizing effect of CP particles to the effective surface area of WSD particles. The interaction between the particles may be high in the 20WSD/10CP/PF hybrid composite. The tensile strength of 10WSD/20CP/PF hybrid composite was lower than the virgin WSD/PF composite. This is due to the ineffective interaction between the particles and the matrix. The effective surface area for contact with the matrix is reduced by lower loading of WSD particles, which reduce the interfacial adhesion between the particles and the matrix. Therefore, the 10WSD/20CP/PF hybrid composite shows lower property values than virgin WSD/PF and 20WSD/10CP/PF hybrid composites. The tensile modulus value of 20WSD/10CP/PF hybrid composite was also high compared to the other composites. It was 5.22% and 8.78% higher than virgin WSD/PF and CP/PF composites.

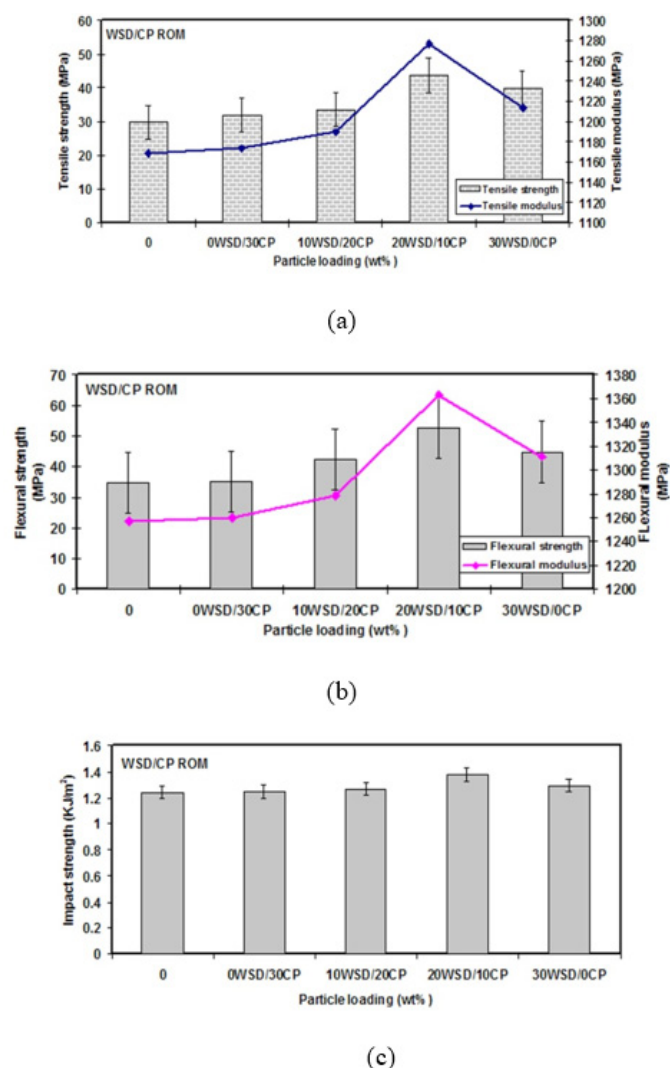


Fig. 3. SEM view of fracture surfaces of WSD/CP/PF composite (1:1) (a) WSD 30 wt. %, (b) WSD 40 wt. % (c), WSD 50 wt. %

The flexural strength and modulus of the WSD/CP/PF hybrid composites are shown in Fig. 3b. The figure indicates that the flexural strength and modulus of 20WSD/PF composite was improved as 10 wt.% of CP particles were added to form a hybrid composite. However, further addition of CP particles (20 wt.%) decreased the flexural strength and modulus of 10WSD/20CP/PF hybrid composite. The flexural strength of 20WSD/10CP/PF hybrid composite was 17.63% and 51% higher than the virgin WSD/PF and CP/PF composites respectively. This may be due to effective interaction of particles which helps to effective transfer of applied load from the matrix to the particles.

The impact strength of WSD/CP/PF hybrid composite was measured in various weight fractions of particles such as 0WSD/30CP, 10WSD/20CP, 20WSD/10CP, 30WSD/0CP respectively and presented in Fig. 3c. It shows that the impact strength increases with the addition of 10 wt.% of CP particles, after increasing the CP particle (20 wt.%) the impact strength was reduced. 20WSD/10CP/PF hybrid composite shows the maximum value of impact strength when compared to the virgin WSD/PF and CP/PF composites respectively.

From the above results of WSD/CP/PF hybrid composites prepared by the rule of mixture, it can be observed that the optimum hybrid effect occurred at 20 wt.% of WSD particles and 10 wt.% of CP particles, i.e. at 20WSD/10CP. Moreover, the contribution of WSD and CP particles for the mechanical properties of the PF hybrid composite can also be observed from the above results. Based on the weight fraction, the contribution of WSD and CP particles was different for the properties of PF composite. To know the effective contribution of the particles, the hybrid composite was fabricated with the rule of mixture method and found that the contribution of WSD particles was higher than the CP particles on the mechanical properties of PF hybrid composites.

3.5. Comparison of results of WSD/PF, CP/PF, WSD/CP/PF hybrid composite (1:1) and WSD/CP/PF hybrid composite (ROM)

In the case of virgin WSD/PF and CP/PF composites, the CP/PF composites showed maximum property levels at 40 wt.%, whereas the WSD/PF composites showed the maximum property levels at 30 wt.%. Besides, the properties of WSD/PF composites were higher than the CP/PF composites. However, the WSD/CP/PF hybrid composites prepared by 1:1 ratio also showed the maximum property values at 30 wt.% (15WSD:15CP). The maximum property values of the hybrid composites prepared by rule of mixture method can be observed at 30 wt.% of hybrid particles (20WSD/10CP).

It may be concluded that the optimum particle loading required to get the maximum mechanical properties is varied depending upon the effective interfacial adhesion due to the effective surface area of the particles for contact with the polymer matrix. When considering the virgin composites, the optimum particle loading for CP/PF composites was 40 wt.%, whereas the optimum particle loading for WSD/PF composite were 30 wt.%. The maximum properties of hybrid composites prepared by 1:1 ratio were identified at the optimum particle loading of about 30 wt.% (15WSD:15CP). The WSD particles have most influenced on the properties of PF composites than the CP particles.

3.6. SEM observations

With the help of scanning electron microscopy, the fractured surfaces of the composite specimens after mechanical tests were examined to understand the WSD/CP particles and phenol formaldehyde matrix interfaces. The scanning electron microscopy studies can help us to understand the interfacial adhesion between the various components of reinforcements and matrices. The surfaces of the fractured composite specimen in this study were examined by SEM, using a Hitachi S-3000N. From SEM images, Figures 4a-c, it is observed that many voids and porosity are identified on the surface of the composite specimens. These voids and porosity can act as

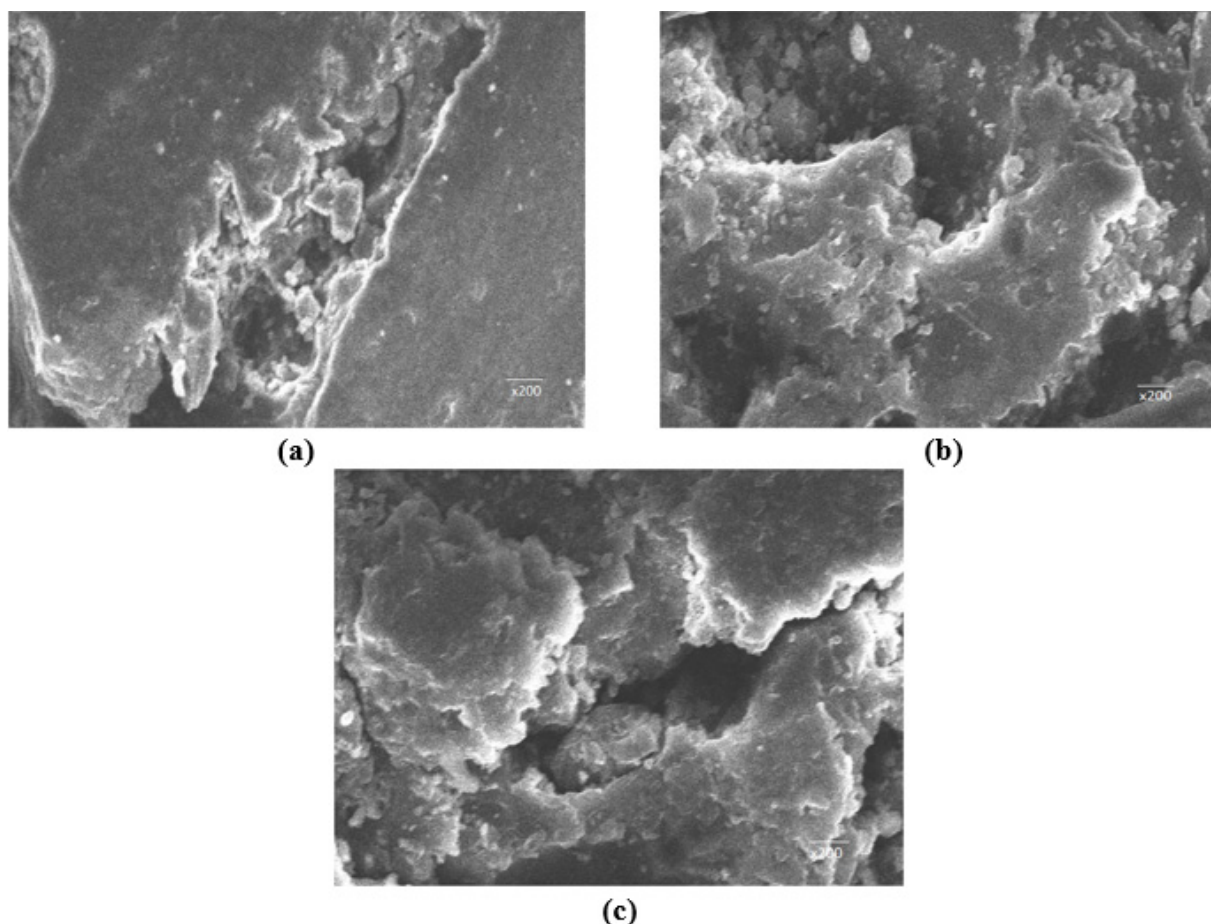


Fig. 4. Variation of (a) tensile properties, (b) flexural properties, and (c) impact strength of WSD/CP/PF composites prepared by ROM

stress concentration points, which leads to the early failure of the composite specimens during mechanical testing. The SEM images are also shown the matrix failure, crack formation and pull out particles with the matrix. The failure identified by the SEM examination was brittle nature. When the irregularly shaped particles were placed in composites they did not ordered properly, leading to a disordered agglomeration position. The order of particles played a crucial role in the final properties of composite specimens because of the disordered agglomeration of particles reduces the interfacial adhesion or bonding between the particle and resin matrix. There was a particle agglomeration in particle-reinforced polymer composites, resulted in the quick failure of the composites. Generally, the nature and geometry of particles, particle content and particle-matrix interfacial adhesion plays the major role on the mechanical properties of particle reinforced polymer composites.

4. Conclusion

A detailed mechanical characteristic study between mechanical properties of fabricated PF composite with the micro level WSD and CP particles has been qualitatively and quantitatively evaluated. Mechanical properties of PF composites were found to increase with increase in particle loading. In CP/PF

composites, 40 wt.% were identified as an optimum loading to obtain the maximum mechanical properties, whereas in WSD/PF composites, 30 wt.% were identified as an optimum loading to obtain the maximum mechanical properties. WSD/PF composites give higher property levels than the CP/PF composites. The test results of WSD/CP/PF (1:1) composites clearly reveal that the optimum particle loading to obtain the maximum properties was also 30 wt.% (15WSD/15CP). When compared with the virgin WSD/PF and CP/PF composites, the WSD/CP/PF (1:1) composite gives superior property levels. Moreover, the WSD/CP/PF composites prepared with the rule of mixture method show the maximum property levels at the combination of 20WSD/10CP. It is proved that WSD particles are most influencing on mechanical properties of PF composite. Therefore, it is designated as most contributing parameter for the mechanical properties of PF composite.

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