

The Mechanical Performance of Sugar Palm Fibres (Ijuk) Reinforced Phenolic Composites

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Sugar palm fibres are one of the natural fibres which have many features and need further study to understand their properties. The aim of this work is to investigate the flexural, compressive and impact properties of sugar palm fibres reinforced phenolic composites. Sugar palm fibres were used as a filler (particle size 150 μm) and with loading of 0, 10, 20, 30, and 40 vol.%. The fibres were treated by sea water and then fabricated into composites by hot press technique. Flexural, compressive, and impact tests were carried out as per ASTM D790, ASTM D695-08a, and ASTM D256 standards, respectively. Scanning electron microscopy (SEM) was used to investigate the morphology and the interfacial bonding of the fibres-matrix in composites. The results show that the mechanical properties of the composites improve with the incorporation of fibres. The composite of 30 vol.% particle loading exhibit optimum values which are 32.23 MPa, 61.66 MPa, and 4.12 kJ/m² for flexural, compressive, and impact strength, respectively. This was because good compatibility of fibre-matrix bonding. Consequently, sugar palm fibre is one of the prospective fibres and could be used as a potential resource to reinforcement polymer composite.

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1. Introduction

Global warming and environmental threats have been attracting a significant attention in recent years.^{1,2} These issues pushed researchers to seeking for root solutions, and looking out for alternative materials and green technologies in many applications such as transportation, construction, and furniture.³⁻⁵ Natural fibres have several benefits such as readily available, cheap, biodegradable, non toxic, low energy consumption, and have good mechanical properties.⁶ Thus, polymer composites based on natural fibres as reinforced materials have shown positive properties not only in mechanical behaviour, but also in physical and thermal properties compared to the pure polymers. Nevertheless, there are some limitations on using natural fibres in composites such as their lack adequate of adhesion between the fibre and the matrix, hydrophilic nature (moisture absorbing), and poor thermal stability.^{6,7}

There are a number of studies that have been done on sugar palm fibre reinforced polymer composites.¹⁰⁻¹³ For instance, Leman, Sastra¹⁴ carried out a study of the effect of long and chopped fibres on the impact strength of sugar palm reinforced epoxy composites. The results

show that the impact strength of long fibres reinforced composites was higher compared with both of chopped fibre composites and pure epoxy composites. The effect of sea water and fresh water treatment for sugar palm fibre reinforced epoxy composites for different period of treatment time was investigated.⁹ Morphology and tensile test results show that the treatment with sea water for 30 days proved to be the best. Bachtiar, Sapuan¹⁵ determined the impact properties of sugar palm fibre reinforced epoxy polymer composites after treating with alkaline. The higher concentration of alkali and the higher soaking time show the dominant impact strength. Moreover, the effect of the same condition of alkline treatment of sugar palm fibre reinforced epoxy composite was employed to study flexural properties.¹⁰ The result reported that maximum flexural strength occurred with 1 hour of soaking time of 0.25 M. It would improve by 24.4% from untreated fibre composite. On the other hand, 0.5 M (NaOH) solution with 4 hours soaking time gave maximum flexural modulus with improvement of 148% from untreated composite.

Selecting the best composite based on bending strength and stiffness for sugar palm fibre reinforced unsaturated polyester composites was

conducted by Sahari et al.¹⁶ The composite reinforced with sugar palm trunk had higher flexural strength and flexural modulus which is 41.906 MPa and 3.363 GPa respectively. In another study, investigation the tensile and impact behaviour of same composites of the previous study was carried out.¹⁷ Not like the first study, sugar palm frond reinforced unsaturated polyester composite shows higher properties compared with sugar palm fibre, bunch, and trunk composites. In addition, the effect of fibre content on mechanical and thermal properties and water absorption behaviour of sugar palm fibre based plasticized sugar palm starch biocomposites were studied.¹⁸ The results indicated an increasing trend with increasing fibre loading. This was due to the positive compatibility of fibre-matrix bonding. A composite of banana peels / phenolic composites were investigation as a new brake pad formulation.¹⁹ The outcoming shown that the less banana filler content the higher compressive strength.

However, up to the knowledge of authors' none of the earlier studies had been done on the mechanical properties of sugar palm fibre reinforced phenolic composites. Since, phenolic polymer has good thermal stability and could be withstand at high temperature, thus it is commonly used in the friction materials. This paper studies the mechanical behaviour of sugar palm particles as a filler in phenolic matrix composites. Its aimed to investigate the effect of fibre content on compressive, flexural, and impact properties and provide background information which will help us to develop a novel friction composites for brake lining and friction applications.

2. Materials and Methods

2.1 Materials

The materials used in this work included sugar palm fibres, phenolic resin modified powder (PH-3507), methanol, and sea water. Sugar palm fibres were obtained from the area of Kampung Kuala Jempol, Negeri Sembilan, Malaysia. The fibres were taken manually from the tree trunk. Phenolic resin modified powder (PH-3507) was supplied by Polcomposite Sdn. Bhd, Kajang, Malaysia and material properties shown in Table 1 (manufacturing data sheet). Methanol was supplied by Al Hassan Jaya Enterprise, Selangor Darul Ehsan, Malaysia and used as a solvent for phenolic resin modified powder (PH-3507) and it was evaporated during hot press process. The percentage of methanol depended on the percentage of the resin and fibre intake and it is about (30-50)% of phenolic polymer. Sea water was obtained from Port Dickson, Negeri Sembilan in Peninsular, Malaysia. It was taken 200 meters from the shore.²⁰ The measured pH of sea water is 8.32 and the salinity is 3.05%.

2.1.1 Physical properties

The fibres used in this study are sugar palm fibres. This fibre is stiff and durable and it is brown or black in colour.⁹ Individual fibre diameters were measured using optical microscope (type Leica MS 4). Twenty five samples of single fibres were measured. It was found the diameter of sugar fibre in the range of (166-461) μm and this was within the range that reported by Ishak et al., 2011.²¹ Fig. 1 shows the pictures of raw materials and the optical microscope of sugar palm fibres.

The densities of sugar palm particles and phenolic resin were

Table 1 Phenolic resin modified powder (PH-3507) properties

Material properties	Units	Value observed
Appearance	White/pale yellow	Pale yellow
Melting point (by capillary)	0°C	90±2
Hexamine content	%	10
Ash content	%	0.08
Viscosity in sprit (FC B-4 method)	secs.	24
Sieve analysis #200	%	100

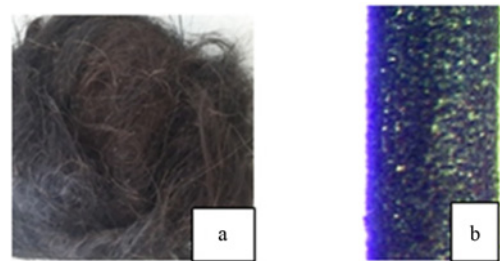


Fig. 1 (a) Sugar palm fibres (b) Optical microscope of sugar palm fibre

measured using gas pycnometer tester (AccuPyc II 1340) using helium gas at pressure 19.5 psig and temperature 29.24°C. By using an analytical balance with the capability of reading up to 0.0001 g, an empty chamber of size 3.5 cm³ was weighed before and after filling with the materials separately. The variance of both weights was recorded as the mass of the material. Then, the filled chamber placed into the tester and the density of the material was recorded. The average of five reading was taken as the final result. Sugar palm fibre and phenolic resin modified powder (PH-3507) densities were found to be 1.4799 g/cm³ and 1.2991 g/cm³.

2.2 Methods

2.2.1 Process on the sugar palm fibres

The raw materials, sugar palm fibres were extracted, collected and cleaned completely to remove impurities, waste, and small stones that may come with the fibres when collected. The fibres were washed with tap water for several times to get rid of any debris and then dried for three days in a well ventilated room at a temperature (27.2-32.5°C) and relative humidity of (69.4-72.3)%.

It is well known that natural fibres reinforced polymer composites have many advantages as well as some drawbacks.^{22,23} One of the main disadvantages is the lack of sufficient interfacial bonding between the fibre and the matrix.²⁰ A biological, low cost, and easy treatment using sea water was employed to treat sugar palm fibres in this study. The scanning electron microscope (SEM, model Hitachi S-3400N) was used to investigate the microstructure of the fibres before and after the treatment.

The fibres were treated with sea water to remove the outer layer and to get positive compatibility between the fibre and the matrix.⁹ The fibres soaked in sea water for 6 days and then left to dry in a room with a temperature range of (28.7-31.2)°C and relative humidity of (68.3-72.1)%. The dry fibres grounded to particles using a pulverizer and thereafter sieved for 15 min using auto sieve shaker (BS410-1). Particles

fibre (150 μm) were used for this research and particles of size above 150 μm reprocessed and passed through the sieves again.¹⁹ Finally, to eliminate the moisture effect, sugar palm powder oven dried at 80°C for 24 hours.²⁴

2.2.2 Fabrication of composites

The composites were prepared using compression moulding technique. Sugar palm particles were priorly dried in an electric oven (Mettler modle TV 30 U) at 80°C for 24 hours. Five set of composites of (0, 10, 20, 30, and 40) vol.% of sugar palm particles reinforced phenolic resin were fabricated. First, phenolic powder mixed with methanol liquid which was used as a solvent and the hardener is hexamine which is within phenolic resin content as shown in Table 1. After that, the mixture is then placed in an oven at 105°C for (5-10) min until the phenolic powder solved completely into methanol. Then, sugar palm particles were added gradually to the mixture and stirred using a glass rod and a mechanical stirrer at (250-450) RPM for 15 min to achieve uniform distribution.²⁵

It is very important that the matrix must have a good viscosity in order to protect the mixture from precipitation that affects the heterogeneity of the fibre and the matrix that could result in conglomeration after hardening.²⁶ The liquid composite was poured in an open steel mould and Mylar sheet was placed under and above the fabricated sample so as to extract it easily. The mould pressed at 150°C and 20 ton for 4 min. The fabricated samples were post cured in an oven at 130°C for 4 hours.¹⁹ The process of sample preparation was shown in Fig. 2. Finally, the specimens were cut from composite plates using vertical saw.

2.2.3 Flexural test

Flexural test was performed according to ASTM D790-10 standard using universal testing machine (model: Instron 3365) at room temperature. The samples of dimensions of (3.2×12.7×127) mm³ were used to conduct the test with a crosshead speed of 5 mm/min. Five samples of each fibre load composites were tested and the average amount of them was recorded as a final value. The test is carried out according to 3-point bending test and the flexural stress and flexural modulus were calculated according to the following equations:

$$\sigma = \frac{3pl}{2bh^2} \quad (1)$$

$$E = \frac{L^2 m}{4bh^2} \quad (2)$$

Where: p, l, b, and h are applied load, span length, width, and height of specimen and m represents the slope of the stress- strain curves for flexural test. Fig. 3 shows the flexural samples before and after test and during the test.

2.2.4 Compressive test

The test employed using universal testing machine (UTM: model Instron 3366) according to ASTM D 695-02a. A specimen was placed between the compression plates tools. Load was then applied gradually with cross head 1.5 mm/min. The average of five specimen of each fibre loading was considered as final reading. Maximum compressive

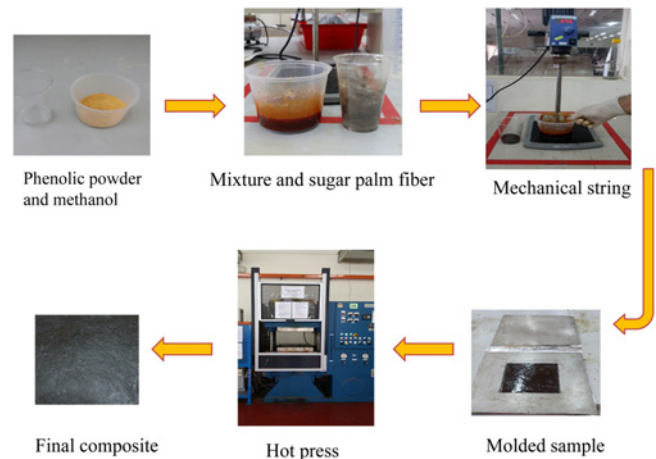


Fig. 2 Process of composite preparation

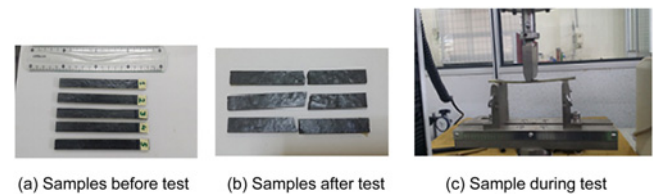


Fig. 3 Flexural test of sugar palm particles reinforced phenolic composite

strength calculated by dividing the maximum compressive load by minimum cross sectional of the specimen. Compressive strength and strain can be studied from the test.²⁷

2.2.5 Impact test

The un-notch Izod impact test was conducted following ASTM D256 standard using INSTRON CEAST 9050. The test sample is supported as a vertical cantilever beam and is impacted by swing pendulum. A rectangular specimen of dimension (3.2×12.7×64) mm³ were used to conduct the test with a hammer type 0.5 J. The average of five specimens of every fibre load composites was calculated. The impact strength (toughness) in kJ/m² and the impact energy in J can be analysed.

2.2.6 Morphology of fracture surface

Morphological analysis was conducted using scanning electronic microscope (SEM), modle Hitachi S-3400N to examine the qualitative distribution of fibre-matrix and the interfacial adhesion between the fibre and the matrix after impact test. All the fractured specimens were coated with a thin layer of gold, to provide electrical conductivity and to eliminate charging effects, and subjected to a voltage of 15 kV.

3. Results and Discussion

3.1 Flexural test

Flexural test was conducted on sugar palm particle reinforced phenolic resin composites with fibre loads of (0, 10, 20, 30, and 40)

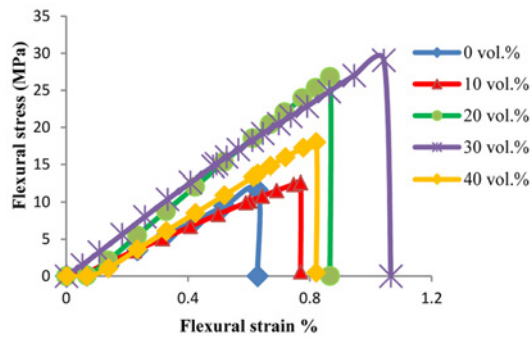


Fig. 4 Flexural stress-strain behaviour of sugar palm particles reinforced phenolic resin composites

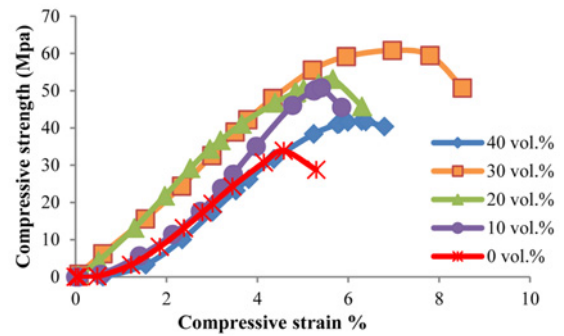


Fig. 6 Compressive stress-strain graph of sugar palm particles based phenolic composites

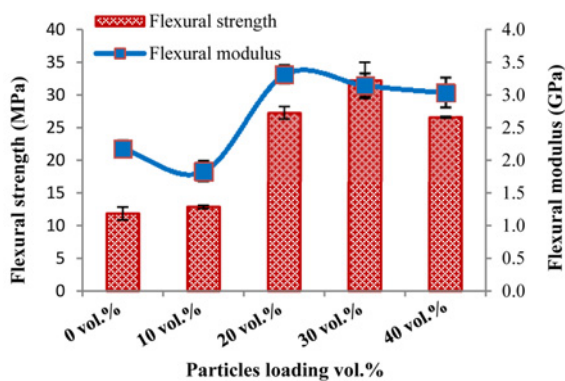


Fig. 5 Flexural stress and flexural modulus of sugar palm particle reinforced phenolic resin

vol.% The relation between the flexural stress-strain of the composites was shown in Fig. 4. From the Fig., it was found that the flexural stress increases linearly with strain up to the fracture point for all types of particle loading composites. The stress-strain shows the typical pattern of polymer composites that illustrates a significant strain for small stress.²⁸⁻³⁰ Moreover, the curve shows that all types of the composites are suddenly broken after first crack due its brittle behaviour. It is found that 30 vol.% sugar palm particles reinforced phenolic composites gave the higher flexural strength, which is 32.23 MPa. Beyond this optimum concentration, the flexural strength for 40 vol.% was dropped to 26.58 MPa.

A significant increase of flexural strength with the increase of fibre load indicated that the natural fibre hold the stress. Adding more fibres make the composite ductility become higher. Consequently, the addition of fibres allows the composite to withstand the fracture under flexure test. Moreover, this also was due to the adequate adhesion of fibre- matrix interface caused by sea water treatment.²⁰

Fig. 5 shows the flexural strength and flexural modulus of sugar palm fibre reinforced phenolic resin modified powder. In terms of flexural modulus, net phenolic composite shows higher value than 10 vol.% sugar palm composite. Then flexural modulus increased drastically with the addition of sugar palm content. The high flexural modulus values which are 3.32 GPa and 3.15 GPa for 20 vol.% and 30 vol.% of sugar palm reinforced composites respectively. The results agreed

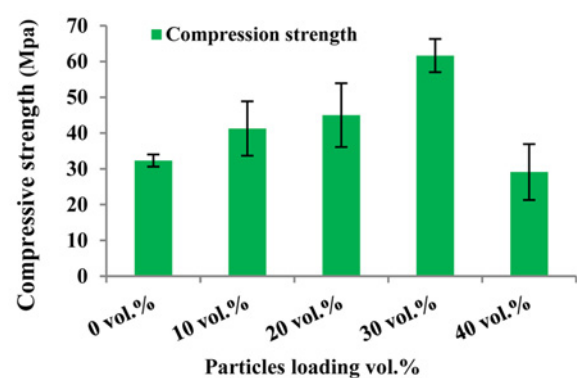


Fig. 7 Maximum compressive strength via particles loading for sugar palm fibres/phenolic composites

with that found in sugar palm fibre reinforced epoxy composites and unsaturated polyester composites.^{9,16}

3.2 Compressive test

Fig. 6 illustrated the variation of stress-strain diagram of sugar palm fibres reinforced phenolic composites for different fibre loading at compressive test. It is clearly shown that compressive stress increased linearly with compressive strain and followed similar trend with that of flexural test. Compressive strength increase with the increase of sugar palm filler content. This may be attributed to the adequate dispersion of sugar palm particles with matrix which results in positive fibre-matrix adhesion. The small size of particles (150 μm) allow them to penetrate and spread in the resin and less pores leads to increasing of compressive strength. Consequently, The capability to resist the load increase for the composites as explained by Ademoh and Olabisi, 2015.³¹ Also, sea water treatment plays significant role to improve the bonding of fibre-resin which result in good properties for the composites.⁹

The maximum compressive strength of the composites presented in Fig. 7 which are (32.33, 41.27, 45.02, 61.66, 29.12) MPa for (0, 10, 20, 30, 40) vol.% particle loading respectively. The compressive strength increases with the increase of particles loading. This may be account to increasing surface area and pore packaging capability of the sugar palm particles in the resin. The present finding also support Idris et al., 2015¹⁹

Table 2 Comparison of the obtained result with existing findings

Configuration of composite		Flexural strength (MPa)	Flexural modulus (GPa)	Impact strength (MPa)	References
SPF* (short fibres) / epoxy	Untreated	50.18	-	18.46	20
	Treated with sea water	53.87	-	17.57	
SPF (long fibres) / epoxy (fibres treated with alkali)		96.71 ^a	6.95 ^a	60.0 ^b	^a 10, ^b 15
SPF (short fibres) / high impact polystyrene	Untreated	34.82	3.58	3.96	28
	Alkali treated	38.99	4.27	5.31	
SPF / unsaturated polyester		33.74 ^c	2.42 ^c	4.57 ^d	^c 16, ^d 17
SPF / plasticized sugar palm starch		2.50	0.40	10.5	18
SPF (particles) / phenolic		32.23	3.32	4.12	Current study

*SPF: Sugar palm fibres.

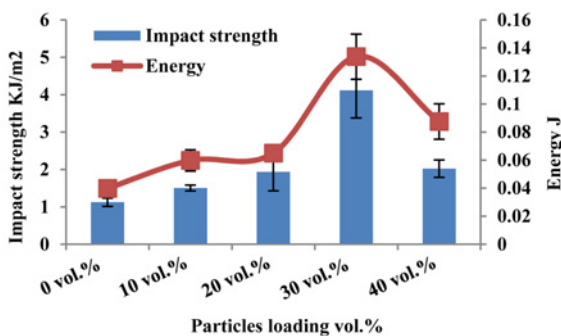


Fig. 8 Impact strength and impact energy of sugar palm particles reinforced phenolic composite

and Ruzaidi et al., 2011²⁷ studies which concluded that the higher filler the higher properties. However, compressive strength decreased for 40 vol.% composite beyond this could be the poor adhesion of fibre-matrix of the composite because of the insufficient resin to bond the fibre particles.

3.3 Impact test

The impact properties of sugar palm particle reinforce phenolic composite of different particles content of (0, 10, 20, 30, and 40) vol.% are shown in Fig. 8. The absorbed impact energy (J) is the total energy required to break the specimen. It is determined from the difference in potential energy before and after the test. The impact strength of composite (kJ/m^2) was obtained by dividing the recorded absorbed impact energy with the cross-section area of the samples.³⁰ The impact strength and the absorbed energy were improved for all sugar palm particle loading. Fig. 8 clearly shows that impact strength of the composites increases with the increasing of particle loading up to 30 vol.% which is 4.12 kJ/m^2 . Meanwhile, the absorbed energy increased considerably with increasing content of sugar palm particles for the composites which are (7.97, 11.94, 13.00, 26.75, and 17.49) % respectively. This may be attributed to the good adhesion between the fibre and the matrix. Similar outcome for lignocellulosic fibres and/ or cellulose nanofibre based thermosetting polymers were reported by other works.^{9,20,21,29}

Table 2 detect comparison of the recent work of sugar palm fibres reinforced polymer composites for flexural and impact tests.

However, there have been no controlled studies to compare

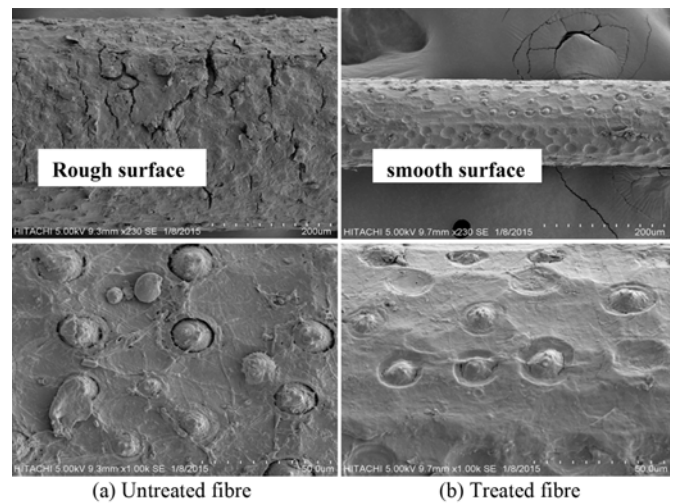


Fig. 9 Morphology structure of sugar palm fibre (a) Untreated (b) Treated

differences in compressive properties. According to the table, sugar palm fibres based phenolic composites show lower maximum flexural stress among all sugar palm fibres reinforced polymers composites. This may be belongs to the matrix behaviour.

3.4 Morphological analysis

Fig. 9 shows micrographs of sugar palm fibres before and after treated with sea water with X 230 and X 1.00 K magnifications.

From (SEM) analysis, it was clearly shown, that untreated fibres covered by the outer layer of hemicelluloses and pectin. This layer plays a big role to protect the fibre from environmental conditions. On the other hand, it has weak interfacial adhesion with the interior layer which comprises of lignin and crystal celluloses. Moreover, sea water treatment would help to remove this layer due to its salinity properties.

Fibre-matrix adhesion plays a big role in the interface microstructure bonding which determines significant mechanical properties of the bio-composites. Good interfacial bonding between the fibre and the matrix is important to transfer the stress which leads to higher mechanical properties of the composite.¹⁸ Moreover, the positive compatibility of fibre-resin makes the matrix hold stress not only the fibres. To understand the failure mechanism of the composite, fibre-matrix bonding is usually investigated.

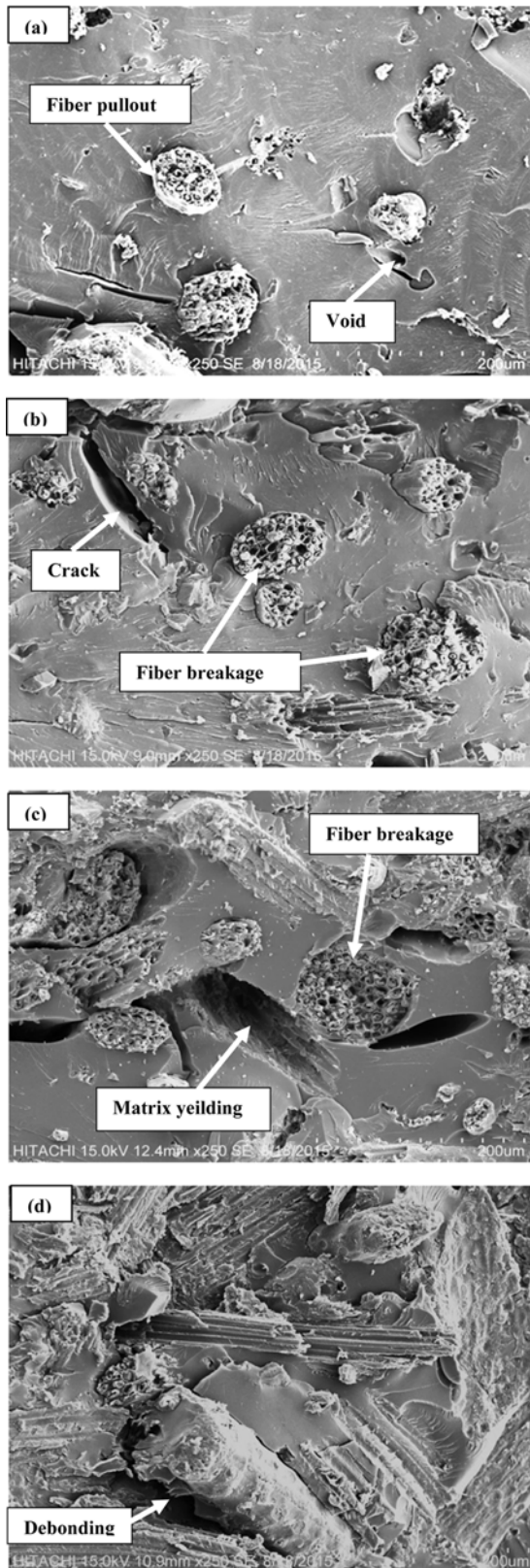


Fig. 10 Scanning electron microscope images of an impact fracture sample of sugar palm particles reinforced sugar palm fibres (a) 10 vol.% (b) 20 vol.% (c) 30 vol.% (d) 40 vol.%

Fig. 10 illustrates a different (SEM) micrographs of the fracture surface of of sugar palm particles reinforced phenolic composites after

the impact the test for different particle content. The microstructure reveals that there are small discontinuities and a reasonably uniform fibre which embedded in the resin. Smooth surface of sugar palm fibre was noticed due to sea water treatment because it removes the thick and rough outer layer from the fibre.²⁰ As a result, adequate adhesion between the fibre and the matrix as well as homogeneous matrix and few voids were found in the composites. Therefore, it is clearly observed more breakage of the fibres due to applied load and less fibre pullout. Moreover, the fibre breakage is more dominant in the composites compared to fibre pull out which indicate positive adhesion between the matrix and the fibres. The (SEM) images of 30 vol.% and 20 vol.% sugar palm particles reinforced phenolic resin exhibited more homogeneous distribution and less fibre pullout. However, the fibre breakage due to the applied load and there is a less gap between the fibre and the matrix of the fracture samples. This indicates good compatibility between the fibre and the matrix. Sugar palm particles with 40 vol.% reinforced phenolic composite reveals debonding between the fibre and the matrix. This could be related to the weak linkage of fibre-matrix because the matrix not enough to spread around the particles.

Besides, the resin is not sufficient to move through particles and bonding them completely. This results in poor adhesion between the fibre and the matrix, that is the reason for properties to decline for this composite.

4. Conclusions

The effect of particle loading on flexural, compressive, and impact properties of sugar palm fibres based phenolic composites were analyzed. The results showed an increasing trend with increasing particle fibre loading for all properties composites were analyzed. The results showed an increasing trend with increasing particle fibre loading for all properties. However, the impact strength of neat phenolic show higher impact strength that of the 10 vol.% sugar palm particle reinforced composite. Morphological studies via (SEM) indicated uniform distribution of fibres and matrix and showed good compatibility of fibre-matrix adhesion. Overall, the highest values of flexural, compressive and impact strength demonstrate by 30 vol.% sugar palm particles reinforced phenolic polymer composite. Consequently, a significant improvement of mechanical properties of the novel green composite of sugar palm particle based phenolic polymer is achieved.

5. Conflict of Interests

The authors declare that there is no conflict interests regarding the publication of this study.

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