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The effect of OPWF filler on impact strength of glass-fiber reinforced epoxy composite

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Abstract

In the present study, oil palm wood flour (OPWF) particles with less than 250 µm sizes have been used as filler materials in the woven-glass-fiber reinforced epoxy composite. The hybrid composites were fabricated using a hand lay-up method and cured at room temperature under a compressive load of 196 N (20 kg). The OPWF of 2.5 to 10 parts per hundred (pph) by weight was used to evaluate its effect on impact strength of the hybrid composites at a range of temperature from -50 to 50 °C. The impact strength, evaluated using V-notch Charpy method, showed reduction with increasing filler content up to 5 pph and then the strength increment in those composites containing more than 5 pph OPWF. More severe damages were found in specimens with higher filler contents resulting higher energy absorption during impact. The composites with a large amount of OPWF particles deflected crack propagation paths or created obstacles at the crack tips and increased toughness of the composites. The impact strength was found to decrease when the samples fractured at subzero temperatures and this happened because of the reduction of the matrix ductility at lower temperatures.

Keywords: Hybrid composite; Oil palm wood fiber; Epoxy resin; Impact strength

1. Introduction

Composite materials offer many desired properties for engineering design over conventional materials such as metals. The significant advantages of composites include high specific strength, low density, and excellent resistance to corrosion and temperature. This has lead to a great interest in the research and development of composite materials in the past decades especially for aircraft and automobile applications where weight reduction is of major concern. The use of composite materials in replacing the conventional materials has shown to be more cost effective both in production and fuel efficiency of vehicles. One of the examples is the use of a single piece composite material made from sheet molding compound (SMC) to replace the front-end metal panel of a vehicle. The weight savings for the replacement is estimated to be about 25 % of a steel front-end panel or equivalent to about 2 to 3 kg in weight reduction [1].

In an effort to reduce the cost further, natural fillers are getting more attention from researchers nowadays. Natural fillers have specific gravities in the range of 1.25-1.50 gcm⁻³ versus 2.50-2.60 gcm⁻³ for glass fiber [2]. This has contributed to high specific strength for

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naturally filled composites. In the present study, the natural filler used was derived from oil palm empty fruit bunch. This material, also called oil palm wood flour (OPWF), consists of about 65 % of hollow cellulose cells and 25 % lignin [3].

The use of OPWF on epoxidized natural rubber composite was reported by Ismail et al. [4]. They found that at any filler content, the larger OPWF particle shows a shorter cure and scorch time; a lower tensile strength, tensile modulus and tear strength. Beside this, their study also found that by increasing the filler content, the tensile strength and elongation at break were reduced but the tensile modulus, tear strength and hardness of the composite increased.

Zaini et al. [5] reported that polypropylene/OPWF composite filled with larger particle has higher modulus, tensile and notched impact strength. Investigation with wood flour filled polypropylene composite using different species (hardwood and softwood) and particle sizes, Stark et al. [6] found that in general the hardwood exhibited slightly better tensile and flexural properties than the softwoods. On the other hand, the notched impact energy, flexural modulus and tensile modulus increased with increasing the particle size.

Till today not much investigation has been carried out on OPWF filled hybrid composite and little information is available on the impact performance of this type of hybrid composite. The paper describes the influence of OPWF filler addition on the impact strength of hybrid composite under a temperature range from -50°C to 50°C.

2. Experimental work

2.1 Sample preparation

The OPWF supplied by Sabutek Sdn Bhd was sieved to various sizes and particles of below 250 µm sizes were used for this study. Mixing of the OPWF with the epoxy matrix was manually done in a bowl before the hardener was added in a 4:1 (volume) ratio. The blended epoxy-OPWF matrix was then applied on a woven glass fiber (150 mm x 150 mm) using paint brush. Hand lay-up method was used to fabricate the sample containing 17 plies of woven glass fiber, as shown in Fig. 1. The sample was then cured at room temperature under 196 N (20 kg) load for 14 h (see Fig. 2). Samples containing 0, 2.5, 5, 7.5 and 10 pph OPWF were fabricated. Impact test specimens of 55 mm long and 10 mm across were cut



Fig. 1. Layers of glass fiber and blended epoxy-OPWF paste between the layers.



Fig. 2. Load application at curing process.

from these cured samples using water jet machine (model Excel WJ4080); milling machine is used to cut 45° V-notch of 2 mm deep at the middle of a face.

2.2 Measurement of impact strength

The impact test was conducted using Tinius & Olsen Charpy impact tester (Model 84) with a pendulum of 700 kg and an impact velocity of 5.47m/s. The recorded value, U_t is the Specific Energy Absorption (SEA) which is given by:

$$SEA = \frac{Total \ energy \ absorbed \ by \ the \ test \ specimen(U_i)}{Net \ cross \ sectional \ area[W(D-d)]}$$
(1)
$$U_i = \frac{m}{2} \left(U_1^2 - U_2^2 \right)$$

where W is specimen width, D specimen thickness, d depth of the V-notch, m mass of the pendulum, U_1 velocity of the pendulum before impact and U_2 velocity of the pendulum after impact.

The specimen temperature, and the heating and cooling methods used to achieve the temperature are shown in Table 1. To ensure uniform temperature throughout the cross section, specimens were placed in the required temperature environment for at least 1 h before testing.

Temperature, °C	Environment	Measurement gauge
+50	Oven	Oven indicator
+30	Oven	Oven indicator
+10	Refrigerator	Thermocouple
0	Refrigerator	Thermocouple
-10	Refrigerator	Thermocouple
-30	Liquid nitrogen + alcohol	Thermocouple
-50	Liquid nitrogen + alcohol	Thermocouple

Table 1. Processes used to condition specimens at right temperatures.

3. Results and discussion

3.1 Specific density of the composite with OPWF

The specific density for OPWF, epoxy resin and glass fiber were determined and they are 1.10 g/cm³, 1.21 g/cm3 and 2.399 g/cm3, respectively [7]. The densities of two types of composites fabricated with different amounts of filler material, one with OPWF in epoxy and the other one with OPWF/17 ply glass fiber in epoxy, were assessed and the results are plotted against OPWF content in Fig. 3. The results clearly demonstrate that the density decreases linearly with increasing filler addition in both composites. The density of the hybrid composite (OPWF/glass/epoxy) is higher than that of the OPWF/epoxy composite. This is because of high density of glass fiber (2.399 g/cm^{3}). The reduction of specific density from 1.693 g/cm³ to 1.526 g/cm³ for the epoxy composite reinforced with 10 pph OPWF is equivalent to 9.86 % weight loss. For the hybrid composite, the specific density decreased from 1.210 g/cm³ for the specimen without OPWF to 1.082 g/cm³ with 10 pph OPWF. This reduction is equivalent to 10.6 % weight loss which is very significant for aerospace and automotive applications.

The reduction in specific density is also reported by Sreekala et al. [8] who used oil palm fiber in glass/PF (phenol-formaldehyde) hybrid composite. In automotive industry, composite materials with lower specific density are usually designed for weight reduction purpose which means better fuel efficiency of vehicles. According to Tucker and Lindsey [9], 10 % reduction in weight can give approximately 5 % reduction in fuel consumption (depending upon size of vehicle).



Fig. 3. Reduction of density with increasing filler content, (top) with glass fiber and (bottom) without glass fiber.



Fig. 4. Glass fiber volume fraction against OPWF filler content.

3.2 Volume fraction of glass fiber

The glass fiber volume fraction of a composite can be calculated using the following expression:

$$V_{f} = \frac{V_{f}}{\{V_{f} + V_{matrix}\}} = \frac{\frac{W_{f}}{\rho_{f}}}{\left(\frac{W_{f}}{\rho_{f}} + \frac{W_{matrix}}{\rho_{matrix}}\right)}$$
(2)

where ρ_f is density of glass fiber, ρ_{matrix} density of matrix, V_f volume of glass fiber, V_{matrix} volume of matrix, W_f weight of glass fiber, W_{matrix} weight of matrix. In order to determine the glass fiber volume fraction in the hybrid composite which contains three constituents, Eq. (2) is modified to accommodate the filler material. The weight of matrix is replaced with the weight of epoxy/OPWF and the density of matrix is replaced with the density of epoxy/OPWF filler. With these conditions Eq. (2) can be written as

$$V_{f} = \frac{\frac{W_{f}}{\rho_{f}}}{\left(\frac{W_{f}}{\rho_{f}} + \frac{W_{epoxy+OPWF}}{\rho_{epoxy+OPWF}}\right)}$$
(3)

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where ρ_f is density of glass fiber, $\rho_{epoxy+OPWF}$ density of epoxy/OPWF, W_f weight of glass fiber, $W_{epoxy+OPWF}$ weight of epoxy/OPWF. The fiber volume fractions of hybrid composite specimens containing different filler contents are determined using density in Fig. 3 and measuring weights of glass fiber (W_f) and epoxy/OPWF. The results are presented as glass volume fractions against OPWF content in Fig. 4, which demonstrates that the glass fiber volume fraction diminishes with the addition of filler material. The figure shows that the composite without OPWF has 46.51 % glass fiber and it reduces to 36.05 % with 10 pph OPWF, which is about 10 % reduction of glass fiber fraction. The microstructure analysis shows more matrix impregnation in the specimen with higher filler content and this may be the reason for reducing the fiber volume fraction in the composite with increasing filler addition.

3.3 Structure of the hybrid composite

Pores were commonly found in the fabricated hybrid composites and they are less at the surface and more down to the surface (in the cross section). Air bubbles are entrapped in the epoxy during fabrication and they are considered to be responsible for pore/ void formation. The short gel time of the epoxy is also an important factor for void formation. Polymerization of the epoxy may be very fast and compressive load applied during curing could not remove the air bubbles. The voids are seen more in 10 pph specimen and this is because of higher viscosity of the OPWF blended matrix which gives greater resistance to air bubbles to be squeezed out during curing under load. The matrix was observed to impregnate more in the inter ply regions than in the intra ply; pores are larger in the matrix-rich areas. Micrograph of the cross section of the 10 pph specimen in Fig. 5 shows larger voids in the matrix rich region (inter-ply) and smaller voids in the weft and warp regions. Hagstrand et al. [10] also observed voids in glass fiber-reinforced polypropylene composites. They found that porosity is mainly in the form of macro voids and located in polymer rich regions.



Fig. 5. SEM micrograph of the cross section of 10pph OPWF filled specimen showing larger voids in the matrix rich area.



Fig. 6. OPWF particle in epoxy resin showing its internal structure (500x).

The OPWF particles are seen well distributed across the cross section but they are more in the matrix rich areas, especially the larger particles of the 10 pph specimen. These large particles are likely to play a vital role in influencing the impact energy of the composite because of its microstructure which consists of hollow-cellulose cells and thick lignin walls, see Fig. 6. Husin et al. [3] reported that the composition of oil palm fiber consists of 65 % hollow-cellulose cells have empty cores like a "donut" shape which leads to lighter weight in OPWF.

3.4 Effect of filler content on impact strength

The impact test was conducted on specimens fabricated with different amounts of OPWF materials and 17 plies of woven glass fiber. The composite specimens containing 2.5, 5, 7.5 and 10 pph OPWF were tested at temperatures of 0, \pm 10, \pm 30, \pm 50, \pm 10, \pm 30 and \pm 50 °C. One set of composite specimens containing no OPWF (0 pph) was also tested at

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different temperatures in order to compare the strength with that of hybrid composites. The impact strength is recorded as specific energy absorption (SEA) and the results for each temperature are plotted against the filler materials in Fig. 7.

Results in Fig. 7 show that the variation of filler content do not give any definite correlation with impact strength but it fluctuates between 150 to 250 KJm⁻² with different temperatures and filler contents. The results also show that majority of the specimens decreases in SEA with the filler content up to 5 pph and then increases with increasing filler content except for those specimens tested at 10 °C and -50 °C. This behavior of reduction of impact strength with 5 pph filler is not known. The composite without filler (0 pph) gives lower strength with decreasing test temperature, which has also been reported earlier [11]. The impact strength of composite depends on the glass fiber volume fraction which carries most of the impact load. This has been reported by Sreekala et al. [8], Lee et al. [12] and Thomason et al. [13] with glass fiber reinforced polymer composite, and Nazrin [11] with glass reinforced epoxy composites.



Fig. 7. Variation of SEA with filler content and test temperature.



Fig. 8. SEM micrograph showing a distorted filler particle in the 10 pph specimen.

As OPWF increases beyond 5 pph, the SEA starts to increase. This result is similar to what Sreekala et al. [8] observed where impact strength of glass/oil palm/phenol-formal-de-hyde hybrid composite increased with increasing oil palm fiber volume fraction. The reason for higher impact strength in specimens containing over 5 pph OPWF may be that the filler material is effective above this range to influence the impact property of the hybrid composite. It is to be noted that specimens with higher OPWF content have larger particles which mostly appear at the inter-ply regions. The wood particle has hollowcellulose cell (Fig. 6) which presumed to act as 'cushion' in the epoxy matrix and that improves the matrix deformability especially at high temperatures. This is evident from the micro-graph of a fractured specimen in Fig. 8, which shows a large distorted OPWF particle in the matrix. The distortion is presumed to have occurred during impact fracture. This process of improving impact strength with higher OPWF content is true parti-cularly at high temperatures.

3.5 Effect of temperature

Results in Fig. 9 show that with increasing temperature from -50 °C to 50 °C, the energy absorption also increases from 163 KJm⁻² to 278 KJm⁻² for the 10 pph specimen, but for the 0 pph specimen this increment is small, i.e., from 218 KJm⁻² to 238 KJm⁻². The SEA increment for the 10 pph specimen is 115 KJm⁻² which is very significant compared to that specimen without OPWF which is only 20 KJm⁻². This could be related to the volume fraction of the impregnated matrix in the composite. It was found that the 10 pph OPWF specimen has more matrix impregnation than the 0 pph specimen.

It is to be noted that the epoxy matrix has thermal expansion 10 times higher than the glass fiber. The



Fig. 9. Variation of SEA for specimens tested under different temperatures.

thermal conductivity variation will generate compressive stresses in the composite. This stress is presumed to have created more interlocking between matrix and fiber interface, and hence it requires extra energy to fracture. A similar explanation has been proposed by Nazrin [11] and Mallick [14] for high energy absorption for breaking composite at elevated temperatures. However, some researchers have shown variation to this finding. Thomason et al. [13] investigated the effect of temperature using short fiber in polypropylene resin and found no variation in impact energy at temperatures ranging between -50 °C and 40 °C. Study by Kalthoff [15] found that the energy absorption does not follow any definite trend during the impact of glass/vinyl-ester composite between -40 °C and 140 °C.

3.6 Failure modes

The structure of the hybrid composite broken by impact shows cracks which propagated mostly through the inter ply matrix regions. However, the addition of filler material acts as a crack arrester. Micrograph in Fig. 10 gives the evidence how the OPWF particle at the crack tip stopped the crack propagation in the 10 pph specimen. The impact strength is found to increase with filler addition. The micrograph in Fig. 10 explains the reason how impact strength increased in the 10 pph OPWF composite. Ismail et al. [4] also found higher tear strength with filler content and suggested that the hindrance imposed by the filler to the tear path generated more resistance to crack propagation. The filler particle is also expected to absorb more energy probably because of the structure with hollow-cellulose cells and thick lignin walls (see Fig. 6). This structure is likely to possess more flexibility and deformability under impact. Study by Stark [6] on pine wood flour particle size ranging from 0.841 mm to 0.125 mm has shown that the impact energy of the composite increases with the filler particle size. This result is also confirmed by Zaini et al. [5] and Myers et al. [16].

Fig. 11 shows how a filler particle acted as an obstacle to crack propagation and deflected it from the interfacial region into the weft area. The propagation of crack along the weft and warp interface suggests the weakness of interfacial bonding. The crack deflection by the filler particle indicates that the resin is weaker and the crack did not penetrate from matrix into the filler particle. This finding is in agree-

ment with Stark and Rowlands [17] who also found the occurrence of crack propagation at the wood flour/polypropylene interface. They suggested that the poor interfacial bonding is due to the hydrophilic wood flour and the hydrophobic polypropylene. It has been reported that for a good toughening effect with filler material, the interfacial strength or bonding between the filler/matrix should not be too good or too weak [18]. If the bond strength is too high, particle rupture will occur prior to debonding and no crack bridging will develop. On the other hand, if the bonding is too weak, only little energy will be dissipated. For crack-blunting it is important to raise the toughness of the composite, and Hull et al. [19] suggested that in order to increase toughness in a composite, the crack must be deflected at fiber/matrix interfaces. In the present study, Fig. 11 shows that the large OPWF particle has deflected the crack propagation, and this may contribute to increased toughness of the composite.

A comparison to the extent of damages was made between the specimens tested at -50 °C and 50 °C.



Fig. 10. SEM micrograph showing arrest of crack by the OPWF particle at the tip in the 10 pph specimen.



Fig. 11. SEM micrograph showing crack deflection in a 2.5 pph specimen fractured by impact at 50 °C.

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When fractured, the 50 °C specimen showed high deformation with delamination, fiber buckling, breakage and fiber pull-out. On the other hand delamination failure is dominant when tested at -50 °C. This delamination behavior at subzero temperature (-50 °C) has also been reported earlier [20]. At subzero temperature the matrix becomes brittle and it promotes delamination failure by crack propagation through the matrix in between glass layers. This may be one of the causes to have relatively low specific energy absorption when tested at -50 °C (see Fig. 7). The V-notch serves as crack initiator with tensile stress which causes fiber pull out, splitting, breakage as well as matrix cracking. The location of the notch tip plays an significant role on the failure mode and hence the impact strength measurement using this Vnotch Charpy method is largly dependent on the location of the notch tip.

4. Conclusions

1. An addition of 10 pph OPWF in the glass fiber reinforced composite can reduce weight by 10 %. Pores are visible in the matrix rich areas and more with 10 pph OPWF specimens.

2. The increment in the SEA is very significant in the 10 pph specimen compared to the composite without filler material. It is presumed that higher matrix impregnation, which caused higher interlocking between the matrix and the fiber glass along with more cushion action of the OPWF particles, generating increased strength.

3. Microstructural investigation of the broken specimens fractured by impact evidently shows that the OPWF particles, especially large ones, can arrest or deflect crack propagation and the wood particle can deform in the matrix rich region. These phenomena have resulted higher impact strength in the hybrid composite with higher filler contents.

4. With increasing temperature from -50 °C to 50 °C, the specific energy absorption increased from 163 KJm^2 to 278 KJm^2 for the 10 pph specimen. At subzero temperatures the specimens fractured with severe delamination along the matrix regions and this is because of reduced ductility of the matrix at subzero temperatures.

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