

MECHANICAL PROPERTIES OF ROTATIONAL MOULDED EMPTY FRUIT BUNCH FIBER REINFORCED POLYETHYLENE COMPOSITES

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ABSTRACT

Rotational moulding is a method for producing hollow plastic articles. Nowadays the advantages which it has to offer in terms of the economic production of quite complex, stress-free articles has made it a very competitive alternative to blow moulding and injection moulding. Rotational moulding is unique amongst plastics moulding processes because the heating, shaping and cooling of the plastic all take place inside the mould with no application of pressure. In this research, oil palm empty fruit bunch fibre reinforced polyethylene (EFB-PE) composites which is using high density polyethylene (HDPE) have been produced using a rotational moulding as the compounding equipment. Three levels of EFB were employed; 5%, 10%, 15% and 20% of the total weight of the sample. Two types of coupling agent, maleic anhydride polyethylene (MaPE) and vinyl trimethoxy silane (VTMS) were used. Overall, both coupling agents imparted considerable improvement in the impact strength and tensile modulus, MaPE showed the highest enhancement. However, only MaPE treated EFB-PE was observed to improve flexural strength and tensile strength. The effect of different fiber length size (75-150, 150-250, 250-400µm) was also investigated; the result showed that all mechanical properties decreased with increasing filler loading. These have been attributed by the poor dispersion of the fiber in matrix and the presence of fiber bundles that remain intact even after several types of surface treatment were carried out. Thus, the role of EFB as reinforcing agent is not fully realized.

Key Words: Oil palm empty fruit bunch, polyethylene, rotational molding, coupling agent, composite, natural fiber

1.0 INTRODUCTION

Natural fibers can vary from wood to leaf fibers. Among these fibres, empty fruit bunch (EFB) is one of the outstanding reinforcing fiber which is usually an abundant of harvest process. Empty fruit bunch from the oil palm plant *Elaeisis guineensis* under the family *Palmecea* is one of the oil palm lignocellulosic by-products generated from oil-palm industries in Malaysia. The incorporation of EFB fiber as a reinforcing component in polymers has received much intention. Most of the research on EFB so far has dealt with polymers such as polypropylene, polyurethane, polyethylene, polystyrene and phenol-formaldehyde resin. The researchers focused on how fibre loading, fibre size distribution, fibre surface treatment affected and effect of compounding techniques on mechanical properties of the composites.

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As with order other natural fibers, many issues have been identified in the processing of the composites. According to Abu Bakar and coworkers [1], that issues include the low compatibility between EFB fibers and the polymer matrix, high moisture intake, poor dispersion of the fibers in the matrix, and effect of oil residues on the mechanical properties. Investigations based on natural fiber are still ongoing. Several studies on other lignocellulosic filled thermoplastic composites have indicated that the mixing procedures could play an important role in determining the properties of composites, reported by Rozman *et al.* [2].

No study has been conducted using rotational molding for EFB reinforced thermoplastic composite process. Thus, it is the objective of the present research to investigate the mechanical properties of EFB fiber reinforced PE composites using rotational molding process.

2.0 EXPERIMENTAL

2.1 Materials

Polyethylene that was used as matrix for EFB reinforced composite is a High Density Polyethylene (melt index of 2.6g/10 min and density of 0.96g/cc). It was obtained from Titan Polyethylene (Malaysia) Sdn. Bhd. This polymer is for general purpose of rotational moulding. Empty fruit bunch (EFB) comprising a bunch of fibres in which the palm fruits are embedded and consisting of about 83% holocellulose and 17% lignin in the form of fibre strands. It was supplied by Sabutek Sdn. Bhd., Teluk Intan, Perak, Malaysia. The properties of EFB fibres are shown in Table 1.

Table 1 Properties of EFB (Abu Bakar *et al.* [1])

Holocellulose (%)	82.5
Alpha-cellulose (%)	60.6
Lignin (%)	17.2
Extractives (%)	2.3
Ash Content (%)	5.4
Tensile Strength (MPa)	130
Tensile Modulus (GPa)	3.58
Elongation (%)	9.70

2.2 Filler Preparation

EFB fibre was obtained in the form of a long strand of fibres. The fibres were ground into small particles. A Restsch shaker was used to separate the EFB fibres into different sizes. The shaking time was 10 min. The filler size used in this study was a mesh of 75-150, 150-250, and 250-400 μ m. The fibres were dried in an oven at 105°C for about 24 hours to constant weight before chemically treated for further processing. Prior to composite preparation, the fibres were treated with coupling agents such as maleic anhydride-polyethylene (MaPE) and vinyltrimethoxysilane (VTMS) to improve the adhesion between the matrix and fibre.

2.3 Filler Treatment

Applications of the coupling agents (MaPE) and (VTMS) to the PE-EFB composites differ from one another due to the nature of the chemical themselves. VTMS was delivered in liquid form and before application; the coupling agent was mixed in water-ethanol mixture (40: 60). Then the fibres were dipped in that silane solution for about 1 hour with constant agitation. The pH of the solution was maintained at 3.5-4. After washing and ethanol has evaporated, fibres were dried for 24 hours in an oven at 65°C. The amount of the coupling agent used in this study was about 1% by weight of EFB filler. MaPE was used as delivered and was added directly to the EFB-PE mixture.

2.4 Sample Preparation in Rotomould

EFB fibers were added as reinforcement at concentrations varying from 0% to 20% by weight. Silicone was used as a demoulding agent. The composite sample was prepared as follows; mixture of High Density Polyethylene (HDPE) and EFB were loaded into the mold. Before placing materials into a rotomould, the mold was cleaned up and applied with external mold release agent to the mold surface to ensure successful release. The mold loaded with materials, and then was heated within 300 seconds. Afterwards it was cooled within 600 seconds. For forward time, it was 330 seconds and the reverse time was 370 seconds. The speed of rotation was in a range of 11-13 rpm. The part would shrink on cooling, coming away from the mold, and facilitating easy removal of the part. The specimens produced were then removed and cut into three types of test sample for flexural, tensile and impact tests according to standards discussed later.

2.5 Tensile Test

According to ASTM (D638), tensile test were carried out on samples with dimensions of 12 x 4 x 3 cm³ (length x width x thickness). The testing will done in standard laboratory atmosphere of 23°C \pm 2°C (73.4°F \pm 3.6°F) and 50 \pm 5 percent relative humidity. This condition of plastic for not less than 40 hours prior to test in accordance with Procedure A of ASTM D618. Universal Testing Machine (Instron 5567) was used at cross-head speed of 50 mm/minute. Three consistent results were chosen for study.

2.6 Flexural Test

Flexural test were conducted on the Instron Machine Model 5567 according to ASTM D790. The samples, with dimensions 125 x 13 x 3 mm³, were tested at a crosshead speed of 3 mm/min at room temperature. The support span for the flexural test was 51 mm. all the reported values for the tests were average of four specimens.

2.7 Impact Test

Samples collected from compression moulding process were shaped according to ASTM D256. The standard specimen for ASTM is 65.2 x 13.0 x 3.0 mm³. In this study, pendulum impact test - Notched Izod Impact Test was utilized. The specimen then was clamped into the pendulum impact test fixture with the notched side facing the striking edge of the pendulum. The pendulum then was released to strike through the specimen. The pendulum velocity was at an impact velocity of 3.0 m/s and 90° swing angle.

3.0 RESULTS AND DISCUSSION

3.1 Tensile Properties

Figure 1 shows that tensile modulus increased significantly with the addition of 1% coupling agent for both MaPE and VTMS. This would obviously results in an increase in the efficiency of stress transfer from matrix to the filler, which consequently gave rise to higher modulus. The higher tensile modulus value was observed for MaPE. Rozman *et al.* [2] has reported that, it is believed that a good filler-matrix interaction could be derive from the formation of an ester bond between the anhydride groups of MaPE and the hydroxyl groups at the surface of EFB fibers, and/or hydrogen bonding between hydroxyl groups and oxygen from carboxylic groups produced from the former reaction. Because MaPE is a derivative of PE, it should be very compatible with PE.

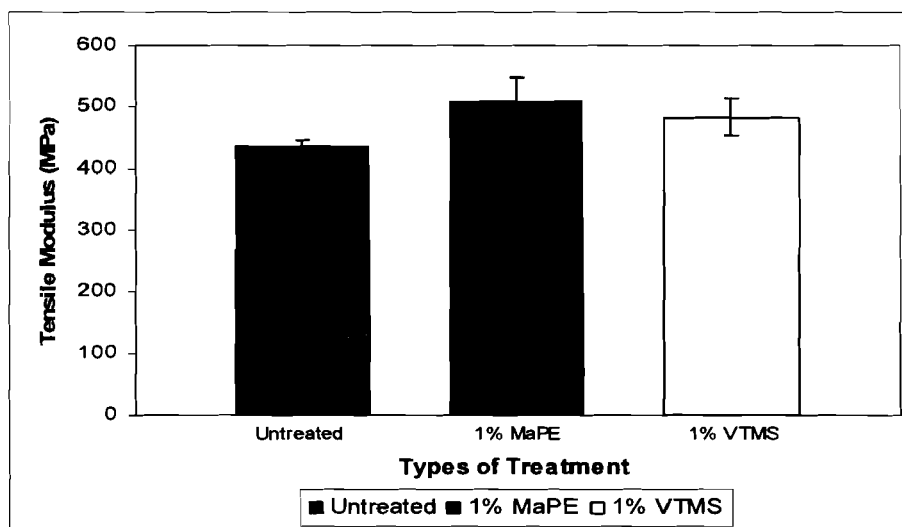


Figure 1 Effect of Various Treatments on the Tensile Modulus for EFB-PE Composites.

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On the other hand, tensile strength of the various treatments of EFB reinforced PE are shown in Figure 2.

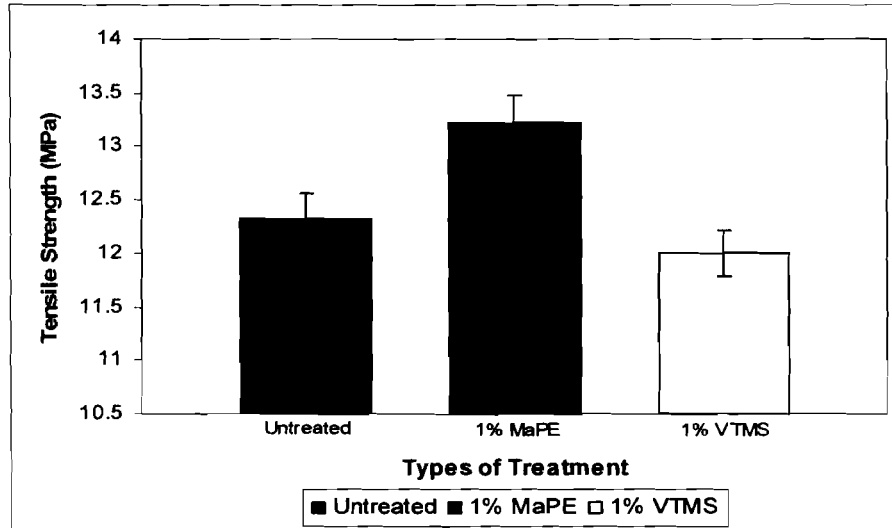


Figure 2 Effect of Various Treatments on the Tensile Strength for EFB-PE Composites.

A significantly increased in strength with 1% MaPE but a decreasing value for 1% VTMS was observed. This may due to the incompetence of tensile test piece preparation; this includes non uniform specimen specification in width, thickness and gauge length. The result was still acceptable to the untreated value because the standard deviation obtained was still in the experimental error. Tensile strength of the composites was further improved with MaPE. This observation was in agreement with the research conducted by Rozman *et al.* [4] on the effluence of maleated PP on the mechanical properties of EFB-filled PP. The greater reinforcement by the EFB in the presence of the maleated PP has been attributed to the better dispersion of the EFB in the PP and better bonding between the constituents' materials. Figure 3 illustrates the hypothetical reaction of lignocelluloses EFB filler with MaPE.

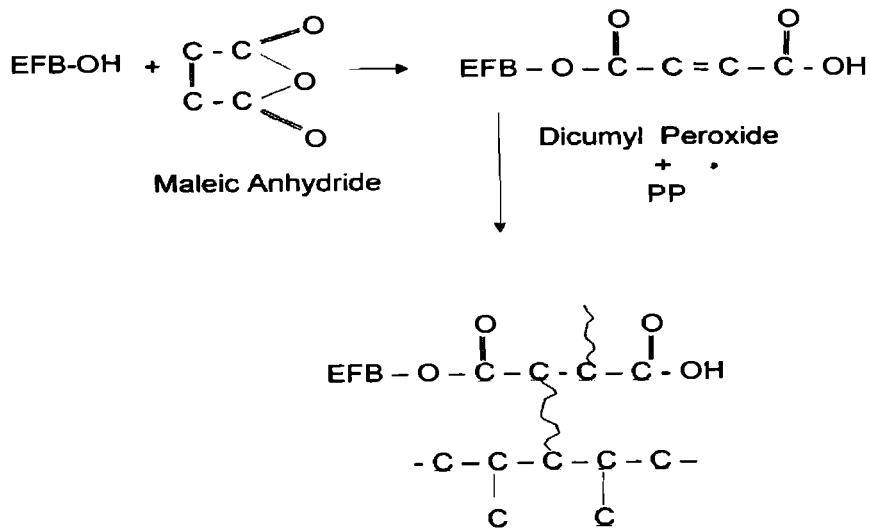


Figure 3 Schematic illustration of the reaction involved in producing the MaPE-treated EFB-PE Composites (Rozman *et al.*, [5]).

Figure 4 shows the effect of filler incorporation on the tensile modulus of treated EFB-PE composites. The increase in modulus with filler loading clearly indicates the ability of EFB fillers to impart greater stiffness to the PE composites. Generally, the composites showed maximum tensile modulus for samples with 5-10% EFB. It can be seen that those with mesh 150-250, 250-400 μm attained maximum stiffness at 5% EFB, whilst, those with mesh 75-150 μm at 10% EFB. Generally, it appeared that sample with smaller particles size produced higher tensile properties. This is in agreement with previous results reported for natural fiber reinforced composites as stated in the works of Nabi Saheb and Jog [6] and Torres and Aragon [7].

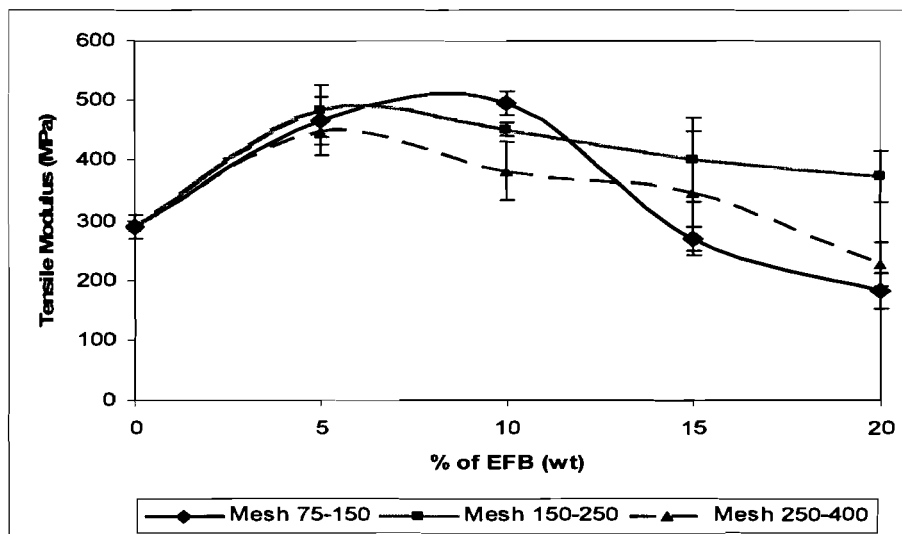


Figure 4 Tensile Modulus for Different Loading and Sizes of Filler to EFB-PE Composites.

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On the contrary, tensile strength decreased steadily for every size of fillers as the concentration of EFB increased in the composites (Figure 5). This is not surprising since others studies have also indicates that the incorporation of filler into thermoplastic matrix may not necessarily increase the tensile strength of a composite as stated by Mohd. Ishak *et al.* [8] and Torres and Aragon [7]. This seems to indicate that the improvement in the filler-matrix interaction was still not capable of overcoming the main problem associated with the filler geometry. Mohd. Ishak *et al.*, [8] has reported that the irregular shapes of the EFB coupled with its strong tendency to bundles together have suppressed the ability of the filler to support stresses transmitted by the HDPE matrix, even in the presence of coupling agent. Torres and Aragon, [7] has reported that low mechanical properties at high fiber contents are mostly associated with the presence of fibre clumps and void. Reinforcing discrete fibers increase the viscosity of the polymer matrix. An increased viscosity might also contribute to the formation of clumps during melt processing of natural fibre composites.

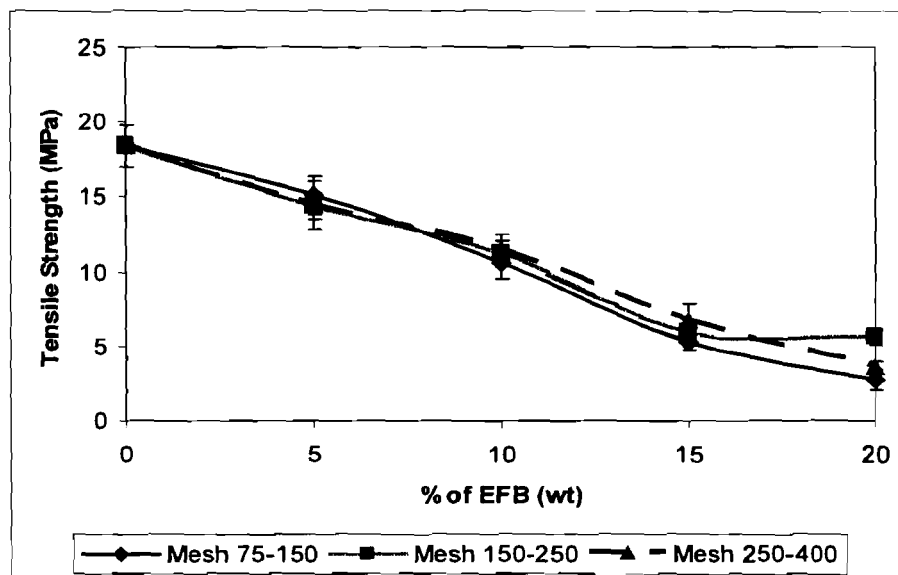


Figure 5 Tensile Strength for Varies Filler Loading and Different Sizes of Filler to EFB-PE Composites.

3.2 Flexural Test

Flexural modulus for various treatments of EFB and PE composites are shown in Figure 6. From this figure, flexural modulus was decreased when coupling agents was added. This may due to the incompetence of tensile test piece preparation; this includes non uniform specimen specification in width, thickness and gauge length. Besides, this may be attributed to the low interaction and poor dispersion of the fiber in matrix because the extensive fiber bundle pulled-out, which is dominant on the bending plane, as reported by Mohd. Ishak *et al.* [8]. The author believed that the integrity of the fiber bundle is still intact, that is, the holocellulose that constitutes about 65% of the EFB is well binding by the lignin. This will provide hindrance for the coupling agent to form an efficient interaction with the holocellulose fibres.

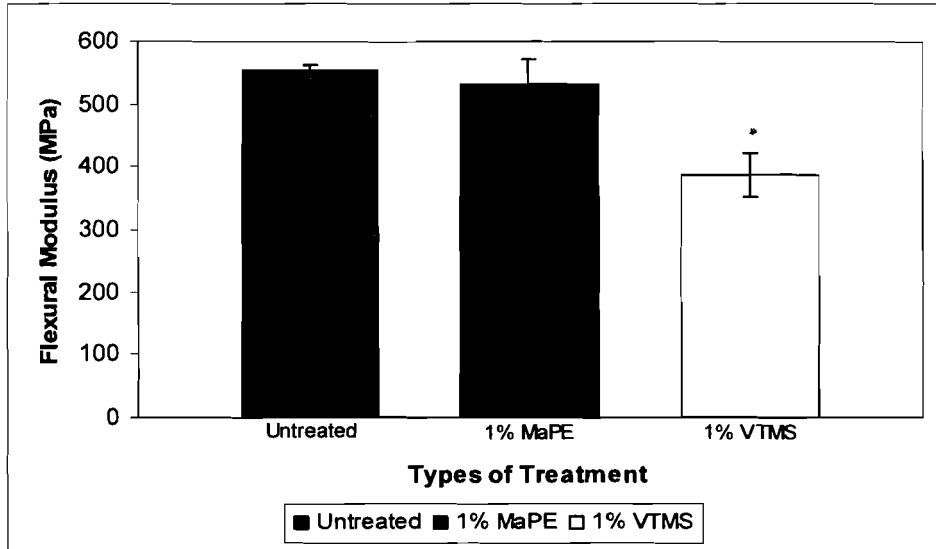


Figure 6 Effect of Various Treatments on the Flexural Modulus for EFB-PE Composites.

Figure 7 shows the effect of various treatments on the flexural strength for EFB-PE composites. The results indicated that MaPE was able to impart greater strength to the composites together with the role played by the MaPE to improve the dispersion of the filler within the matrix, and a reduction in the tendency for the filler to agglomerate. However, the flexural strength of VTMS was lower than the untreated value. This may be attributed to the low interaction and poor dispersion of the fiber in matrix because the extensive fiber bundle pull-out, which is dominant on the bending plane.

Flexural modulus for EFB and PE composites with different size and fillers loadings are shown in Figure 8. Generally, the composites showed maximum flexural modulus for sample with 5% EFB. It can be seen that those with mesh 75-150, 150-250 and 250-400 μ m attained maximum modulus at 5% EFB. After this optimum 5% EFB, dropping sharply at 10%. This is because at higher volume fraction of fiber, the fiber acted as flaws and crazing occurred, thus creating stress concentration area which lowering the stiffness of composites. Rozman *et al.*, [9] has reported that generally, it appears that sample with higher particles size produce higher flexural strength, where factors other than fiber length such as mode of filler, plasticization and better tendency for consolidation in compounding equipment.

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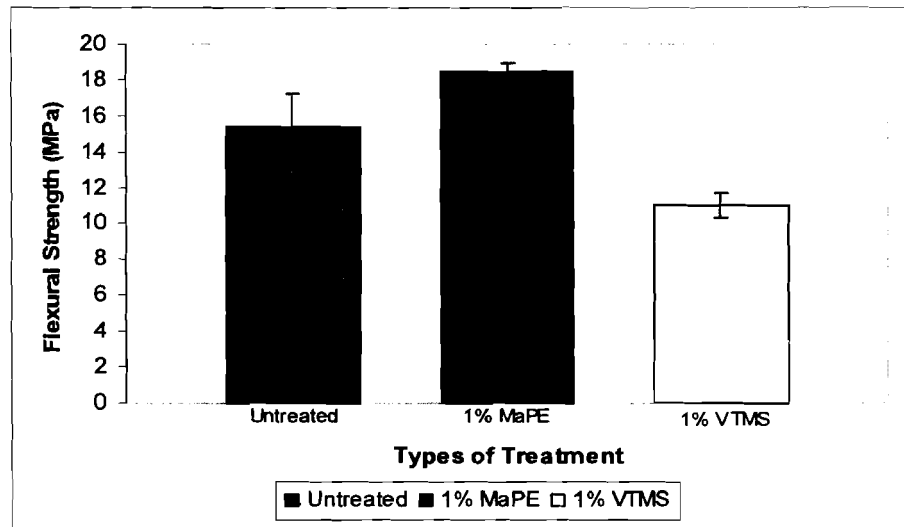


Figure 7 Effect of Various Treatments on the Flexural Strength for EFB-PE Composites.

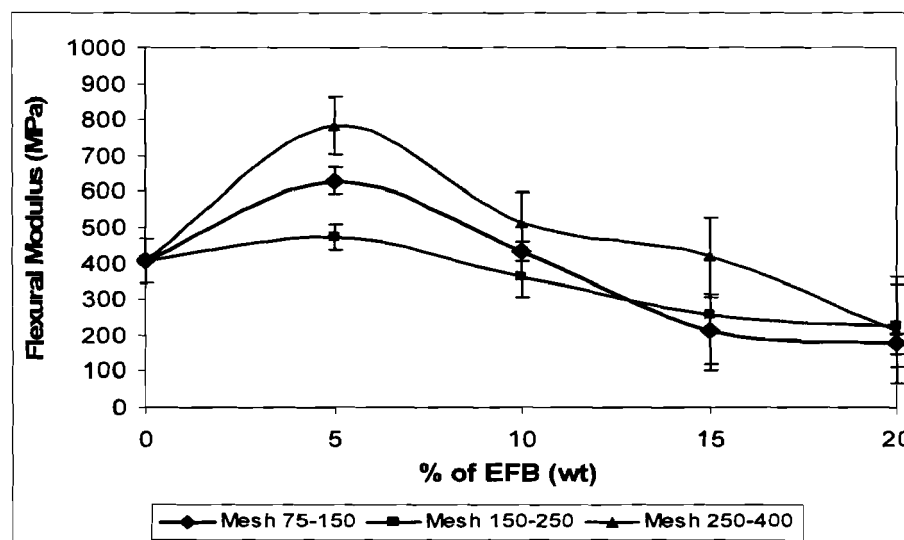


Figure 8 Flexural Modulus for Various Filler Loading and Different Size of Filler to EFB-PE Composites.

Figure 9 shows the effect of different filler loadings and different size of fillers on flexural strength. Similar to figure 4.8, trend of flexural strength decreased after optimum point at 5% EFB. Again, the filler size of mesh 250-400 μ m showed the highest value of flexural strength at 5% EFB. It can be seen that the strength of composites decreased as the proportion of EFB increased. This is in agreement with the trend observed in other lignocellulosic filled composite as stated in the work of Rozman *et al.* [2]. Unlike fibre which have a uniform cross section and a relatively high aspect ratio (length to diameter (l/d) ratio), the capability of irregularly shaped fillers such as EFB to support stress transmitted from the thermoplastic matrix was rather poor. The effect would be amplified if the proportion of EFB were increased.

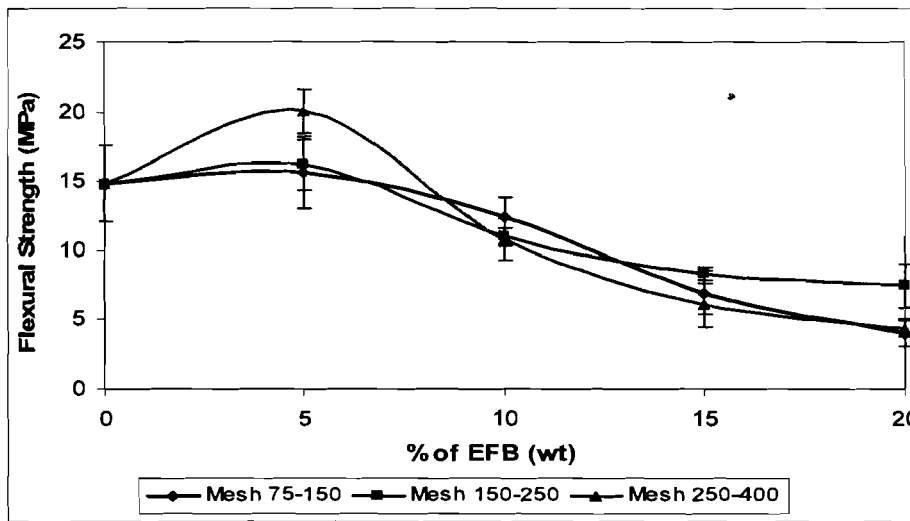


Figure 9 Flexural Strength for Various Filler Loading and Different Size of Filler to EFB-PE Composites.

3.3 Impact Test

Figure 10 shows the trend of impact strength with different types of treatment. It is obvious that composites with MaPE showed higher impact strength than those with VTMS. This is an agreement with the earlier trend observed in the case of tensile modulus shown in Figure 1. Rozman *et al.* [5] has reported that, this can be attributed to an increase in adhesion between matrix and EFB filler, together with the role played by the MaPE to improve the dispersion of the filler within the matrix, and a reduction in the tendency for the filler to agglomerate. In addition to that, MaPE, which contains PE residues in its structure, may promote better bridging between PE matrix and filler.

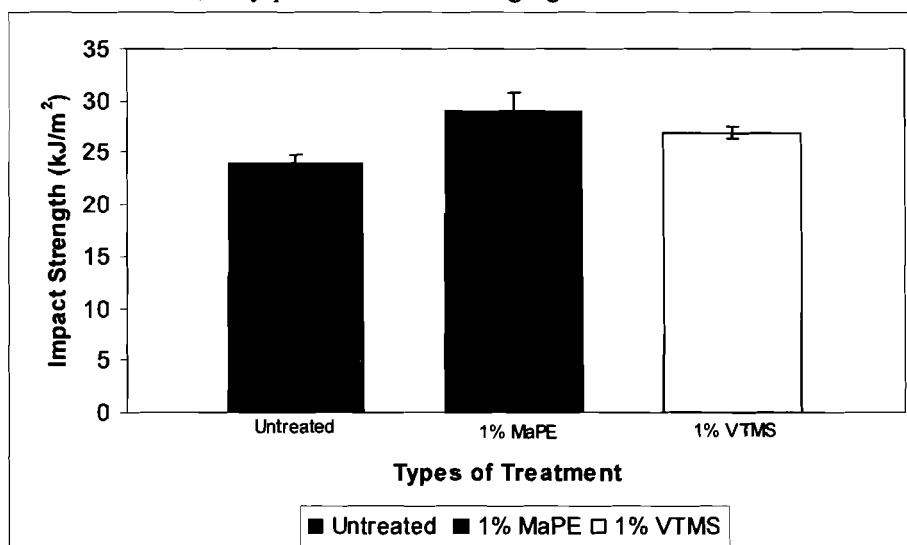


Figure 10 Effect of Various Treatments on the Impact Strength for EFB-PE Composites.

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Figure 11 presents the results for impact strength with different fibre loadings and different size of fillers. Generally, the composites showed maximum impact strength for sample with 5% and 10% EFB. It can be seen that those with mesh 75-150 and 150-250 μm attained maximum modulus at 10% EFB, whilst, those with mesh 250-400 μm attained maximum modulus at 5% EFB. As fibre content increased, the matrix became more brittle and the ability of the material to absorb impact energy decreased whatever the fibre used. Torres and Arago [7] have reported that in the unreinforced region, where bubbles are present, the morphology of a ductile fracture can be observed.

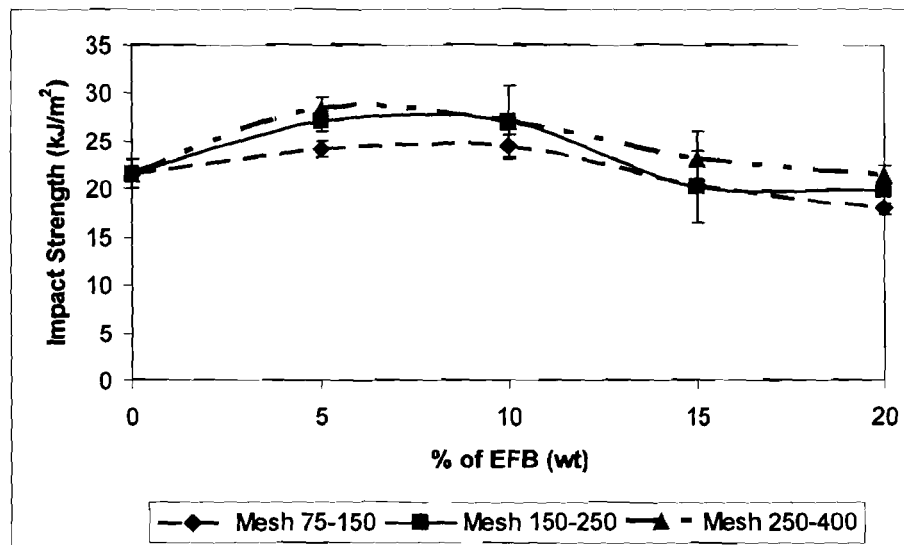


Figure 11 Impact Strength for Various Filler Loading and Different Size of Filler to EFB-PE Composites.

4.0 CONCLUSION

The results of this present study showed that a useful composite with good properties at certain ratio of EFB and PE could be successfully developed using EFB as reinforcing agent for the PE matrix according to their size of fillers. From this, several conclusions can be drawn regarding to mechanical properties of composite to the effect of various types of treatment, fibre loadings and different size of fillers, namely tensile, flexural and impact properties.

All coupling agents used imparted considerable improvements in the tensile modulus and impact strength of EFB-PE composites. Of the both coupling agents tested, MaPE showed the highest enhancement in the tensile strength, flexural strength and impact strength. However, only MaPE was observed to improve the tensile strength and flexural strength. Because MaPE is derivative of PE, it should be very compatible with PE. The condensation reaction taking place between the carboxyl groups in MaPE and hydroxyl groups in EFB is believed to result in the formation of ester linkages between MaPE and EFB.

Finally to summarize everything, EFB has enhanced tensile properties in tensile modulus, flexural as well as impact properties of the PE. This study has demonstrated that optimum fibre loading for peak performance is below that 10 wt%. It can be concluded that those with mesh 75-150 attain optimum at 10% EFB, whilst, those with mesh 150-250 μm

and 250-400 μ m at 5% EFB. Fibre matrix interaction is well adhered and compatible with the use of coupling agent at this concentration.

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