HIGH DENSITY POLYETYLENE (HDPE) - OIL PALM EMPTY FRUIT BUNCH FILLED MICRO COMPOSITES USING MELT BLENDING PROCESS

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Abstract.
In this research, high density polyetylene (HDPE) reinforced oil palm empty fruit bunch fiber micro composites to improve the mechanical properties were prepared using melt blending and hot press technique. Oil palm empty fruit bunch fiber as a filler reduces in sizes 63, 75, 90, and 106 µm. Oil palm empty fruit bunch fiber were mixed with in 10 mL MAPE 8% (in xylene) solution up evenly. The variation composition ratio of HDPE in filler is : (80: 20, 70:30 60:40, 50:50). The good results showed that composite boards are tested viable filler and matrix ratio 30%:70%, 3.969 to 12.243 Mpa of tensile strength, the value of the melting point (Tm) of 400.05 to 497, 07^{o}C for thermal properties, FT-IR analysis results indicate that the groups contained in the matrix and the filler is still present in the composite board products.The SEM photograph tests showed that the best products are the smallest filler size is 63 µm, and 4943.4 J/m^2 of Impact strength test obtained. The results of this study can be applied as part of an electronic element and the casing, because high heat resistance, is a semiconductor, not easily broken and durable.

Keywords: High-Density Polyethylene (HDPE), oil palm empty fruit bunch (EFB) fiber, mechanical properties, filler, melt blending, composite

Introduction

High-Density Polyethylene (HDPE) is a type of plastic waste that are found in everyday life. This waste can be used as a composite which has high mechanical properties by adding filler into it, using melt blending nd hot press process.

EFB of oil palm fresh fruit has fiber that is strong and not easily decompose, when used as a filler in the composite fibers will grow durable and long lasting because it is already covered by the matrix, so it can not decompose by the entry of microorganisms, as it also because wetted with chemicals (wetting agent), so that the fibers remain durable. EFB used as filler smoothed micrometers in size. EFB can be used as a filler to produce a composite, with a maximum tensile strength of the composite is obtained when the filler content of 20% (by weight), although the elongation showed a decreasing trend with increase in filler. The addition of acrylic acid will increase the compatibility between the filler and polypropylene (Basuki et al., 2004).

EFB has the characteristics of hydrophilic (hydroxyl groups in cellulose, lignocellulose and hemicellulose), good interfacial adhesion properties, and low resistance to moisture absorption when used in lignocellulosic composites. For this reason lignocellulosic fibers treated with appropriate chemicals (Rozman, et, al., 2002).

Thermoplastic composites made from lignocellulosic materials such as wood and cellulose at this time continue to be developed. Lignocellulose as filler has many advantages over inorganic fillers, such as: low density, deformability properties of large, flexible, does not generate heat in the equipment during the process, low prices, and derived from renewable resources. Thermoplastic materials from plant fibers and filler material principle is incompatible, because of differences in polarity, so that the necessary modifications to the process such as: in situ crosslinking, addition of compatibilizer and copolymerization of functional groups on the polymer and filler. (Basuki, et al., 2004 and Khalid, et, al., 2007).

Some chemicals are developed for compatibility between the two materials is: maleic anhydrate-modified polypropylene, poly [methylene (polyphenyl isocyanate)], poly (propylene-acrylic acid) and silanes (Rozman, et, al., 2002).

Reaction Maleic anhydrate (MAH) with EFB been observed by FTIR analysis shows the nature of the filler EFB MAH peaks. Composites with MAH-treated filler exhibits high flexural and impact. SEM showed the adhesion properties and good compatibility between EFB and PP matrix as a result of chemical modification using MAH. (Rozman, et, al., 2002).

Treated cellulose is used as a filler thermoplastic potential compared to untreated EFB the PP matrix. Tensile and flexural properties of cellulose treated higher than untreated EFB. The mechanical properties of the composites increased with the use of coupling and bonding agent Maleic Anhydride grafted polypropylene (MAPP) and multi-functional acrylates (MFA) to improve bonding between cellulose and matrix polymers. (Khalid, et, al., 2007).

Processing of cellulose fibers affect the improvement of mechanical properties (tensile strength, modulus of elasticity, hardness) polymer composites. Treatment with agents lead to good results of chemical bonds with cellulose fibers in improving the mechanical properties. Immersion in water affect the polymer composite electrical properties of the composite. Samples were processed polymer composites with cellulose treated giving the resistivity after immersion in water. Processing of cellulose fibers by increasing the chemical bonding agents (in particular amino groups) and to develop the mechanical and chemical properties of polymer composites in water. (Notingher, et, al., 2006).

Modification process of polymer using reactive processing techniques have been reported by several investigators. The end result of the modification reaction to improve the compatibility of polymeric materials using lignocellulosic filler. Impact strength, dynamic fracture, the effect of water on electrical properties, flexural and tensile properties of polymer polypropylene with fiber reinforcement EFB has been reported by several investigators.

Compability between the microfibrils of EFB with HDPE is very low, this is due to the different nature of the polarity between the cellulose microfibrils of waste EFB with HDPE. With the addition of plasticizer will increase the compability in the biocomposite, which can be seen in the mechanical properties, thermal properties, surface morphology, and the FTIR spectrum. This weakness can be improved by mixing the polymer material reinforced with fibers such as, among others, micro-sized EFB fiber. In this study HDPE composites and EFB fiber in micro size will be processed further by using a melt blending method in its application to the material elements of furniture and electronic boards.

Materials and Methods

The materials used in this study is the fiber of oil palm empty fruit bunches (EFB), HDPE plastic, xylene solvent, NaOH 5% solvent, coupling agent maleic anhydride polyethylene (MAPE).

The equipments used crusher, grinding mill, vibrating sieve, three-neck flask, hot plate, condenser, set mixer. Pressing down the upper mold measuring 200 x 200 x 1 mm. Mold pressing middle-sized 115 x 80 x 2 mm, specimen mold and hot press. Differential Scanning Calorymeter test equipment (DSC), test equipment Tensile Strength, Impact test equipment, test equipment Scanning Electron Microscopy (SEM) and Fourier Transform Infra-Red (FTIR).

This study consists of four stages: the first stage of processing EFB fiber, HDPE matrix processing stage, processing composite (melt blending and hot press) and testing stage.

Results and Discussions

The resulting composite visually shows the appearance of a clearer and better homogenity, the physical texture of the composite looks smooth with the delicate size of the filler.

Tensile Strength Properties

Tensile test is one of the mechanical test aims to determine the tensile strength of the material, the test material is pulled off. In composite board with 63µm size of the obtained values between 12,243 MPa tensile strength, composite board with 75µm filler size values obtained tensile strength of 11,592 MPa, composite board with filler size 90 µm obtained values between 6.209 MPa tensile strength, whereas the composite board with size 106 µm filler tensile strength values obtained amounted to only 3,969 MPa, filler size affects the values of tensile strength composite board.

Table 1 Tensile Strength analysis of HDPE/EFB micro composites with different size filler

No.	Filler size (µm)	Tensile Strength (Mpa)	Elongation at Break (%)	Break (mm)
1.	63	12,243	4,718	5,019
2.	75	11,592	4,130	3,299
3.	90	6,209	3,969	2,980
4.	106	3,969	3,176	2,470
Max.	**106**	**12,243**	**4,718**	**5,019**
Min.	**63**	**3,969**	**3,176**	**2,470**

From Table 1, it can be seen that the composite board which has a larger filler size, then the value of tensile strength of the composite board is getting smaller. This happens because the mixing process, the finer the size of the filler will occur uniformly homogeneous mixing and vice versa, the larger the size of the filler will experience a lack of uniformity in the mixing process.

FT-IR analysis

FTIR analysis useful to obtain intensity are available on the groups bonding composites. FT-IR analysis results for HDPE composites with EFB fibers as shown in the Figure 2.

From FT-IR spectra analysis results for oil palm empty fruit bunches fiber (EFB), HDPE matrix and composite board product, it can be seen that the functional groups contained there in (Table 2). Table 2 shows that the functional groups contained in the EFB fiber is O-H, C-H, C=C, CC and C-H_2. In the HDPE matrix contained functional groups CH, C=C, C-C, CH_3 and CH_2, while the composite board products contained the entire functional groups listed in Table 2, the O-H group, C-H, C=O, C=C, C-C, CH_3 and CH_2.

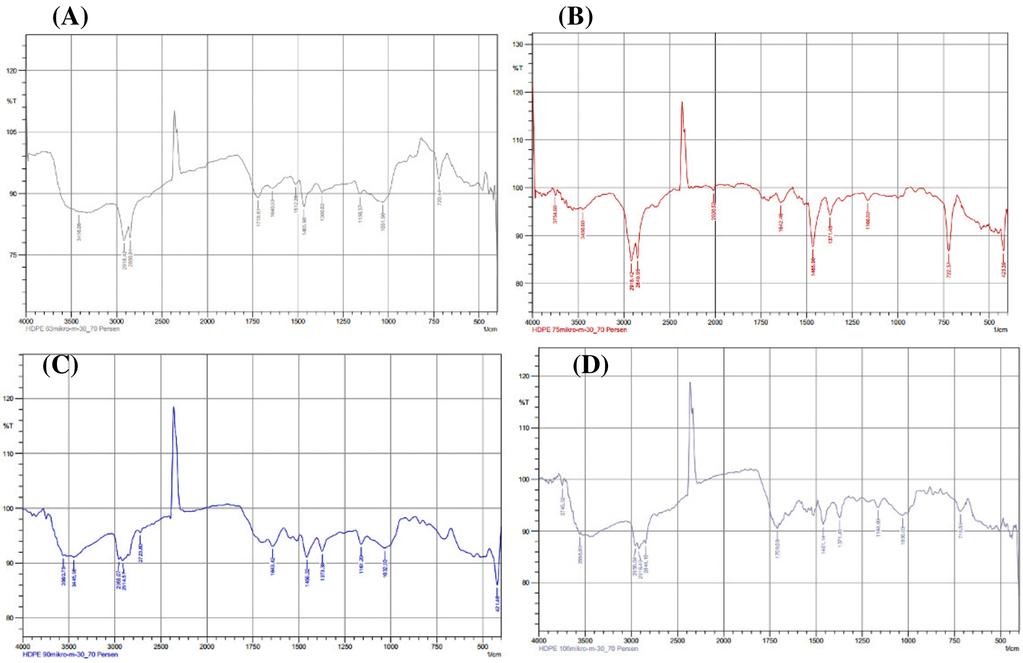

Figure 2 FT-IR spectra results based on differences in the size of the HDPE composite filler, (A) 63 µm, (B) 75 µm , (C) 90 µm and (D) 106 µm.

Table 2. FTIR spectrum of functional groups HDPE/EFB micro composites

Functional groups	Wave number (cm^{-1})	EFB	HDPE	Micro composites
O-H	3500 – 3200	∨	-	∨
C-H	3300 – 2850	∨	∨	∨
C=O	1725 – 1700	-	-	∨
C=C	1650 – 1475	∨	∨	∨
C-C	1650 – 1600	∨	∨	∨
Alkil				
$-CH_3$	1465 – 1365	∨	∨	∨
$-CH_2$			∨	∨

Note: ∨ = viable

Results of SEM Analysis

Observation through Scanning Electron Microscopy (SEM) was conducted to look at the surface of the composite. The existence of differences in the nature of the polarity between the filler particles and the matrix due to the influence of temperature, causing the filler particles of oil palm empty fruit bunches tend to be group/clot, resulting in an uneven distribution throughout the matrix, and cause the formation of spaces (spaces) along the particle becomes open. This shows the compatibility and the low nature of contact between the EFB filler particles and the HDPE matrix. SEM images of the specimen with a composite board composition ratio of 70:30 with the filler size 63, 75, 90, and 106 µm are shown in Figure 3. From the SEM photograph shown that a mixture of oil palm empty fruit bunch fibers with HDPE has demonstrated the existence of a strong interaction between the oil palm empty fruit bunches fibers with HDPE matrix, although there are still visible cavities between the fibers of oil palm empty fruit bunches. In figure A and B show that the matrix and filler distributed evenly, this means that the reaction is very good.

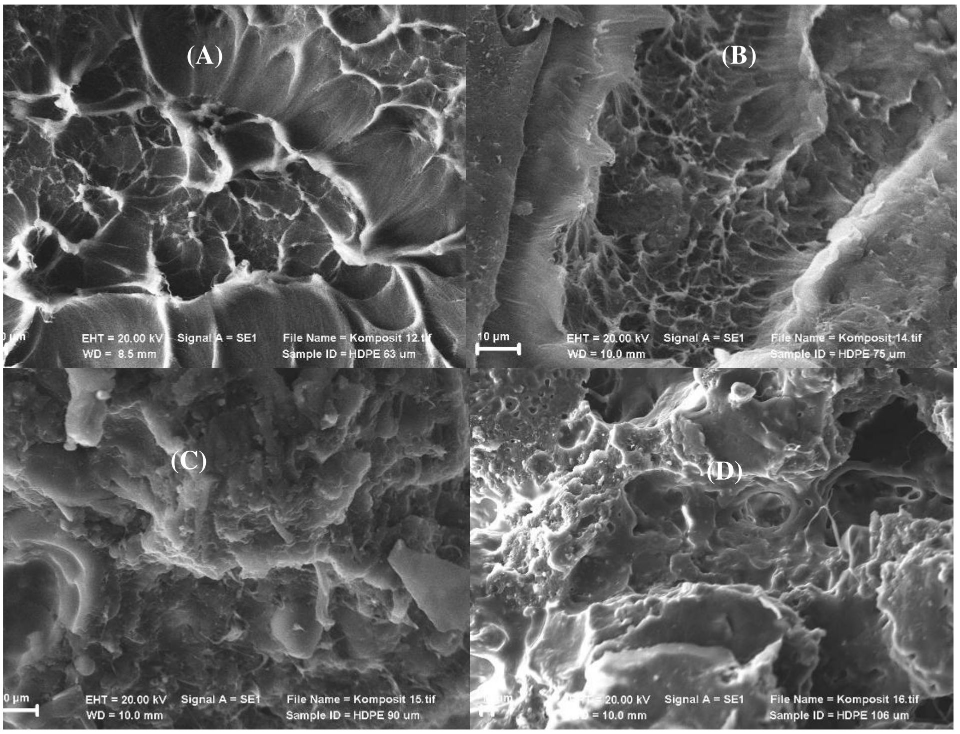

Figure 3. SEM photograph results based on different sizes of HDPE composite filler, (A) 63µm, (B) 75 µm, (C) 90 µm and (D) 106 µm.

In images C and D when observed properly, there are clumps which means they are not subtle still found large cavities due to the filler matrix not distribute evenly, these cavities are caused by oxygen that gets in when the process of compression with hot forged instrument, the effect of the cavity on the strength of the resulting material is very low when compared to the power generated in the samples A and B.

Thermal properties analysis by differential scanning calorimetry (DSC)

DSC (Differential Scanning Calorymeter) analysis done to see the value of the melting point of the composite board to heat, and to see the amount of heat required to reach the melting point. Describe melting point phase change from solid to the liquid without changing the composition and melting temperature, which is the critical temperature, where the polymer crystallinity loss overall. Composite materials decreased melting point along with the addition of fillers, this occurs because the polymer chains are physically degraded, meaning that the polymer chains are driven by a filler material thus damaging the polymer molecule chains.

Table 4. Melting point and decomposition temperature HDPE/EFB micro composites with different size filler

No.	Particle size (µm)	$T_{Polymer}$ (^oC)	$T_{Decomposition}$(^oC)	$T_{Melting}$(^oC)
1.	63	130	106,77	497,07
2.	75	130	27,24	400,05
3.	90	130	30,75	496,73
4.	106	130	43,61	493,45
Max. value	**106,77**	**497,07**		
Min. value	**27,24**	**400,05**		

The curve has a typical model with two endothermic peaks. The value of the melting point and decomposition temperature of the composite HDPE/EFB not a single price but lies in a certain range. Figure 4 looks at the first peak, giving information about the melting point of the material, while the second peak gives information about the temperature of decomposition.

Temperature values contained in Figure 4 can be seen in Table 4, on the table very clearly visible increase in the melting point of the composite board in the test, basically the melting point of HDPE is not mixed with the EFB

130°C, but after the blending process is done, then the point melting of the composite products increased almost 4 times that of HDPE melting point, the melting point of the composite reaches 497,07°C, and the effect of filler size is very significant, clearly visible in Table 4, T melting of composite filler size is smaller, reaching up to 497,07°C filler when compared to its bigger size is 493,45°C, so it can be concluded that the smaller the size of the filler is used, the greater the melting point of the composite produced, so the better the quality.

Decomposition temperature is the temperature at which the material begins to experience a change and a shift in the composition of the molecules contained in the constituent materials. In this study, the filler composite composition measuring 63 µm higher temperature than the filler composite measuring 75, 90 and 106 µm. The temperature of the composite filler 63 µm is 106,77°C while the temperature of the filler composite which 75 µm is only 27,24°C, this is due to the bond that occurs between the smaller filler stronger so that the temperature needed to loosen the bond is very high when compared with that particles larger composition.

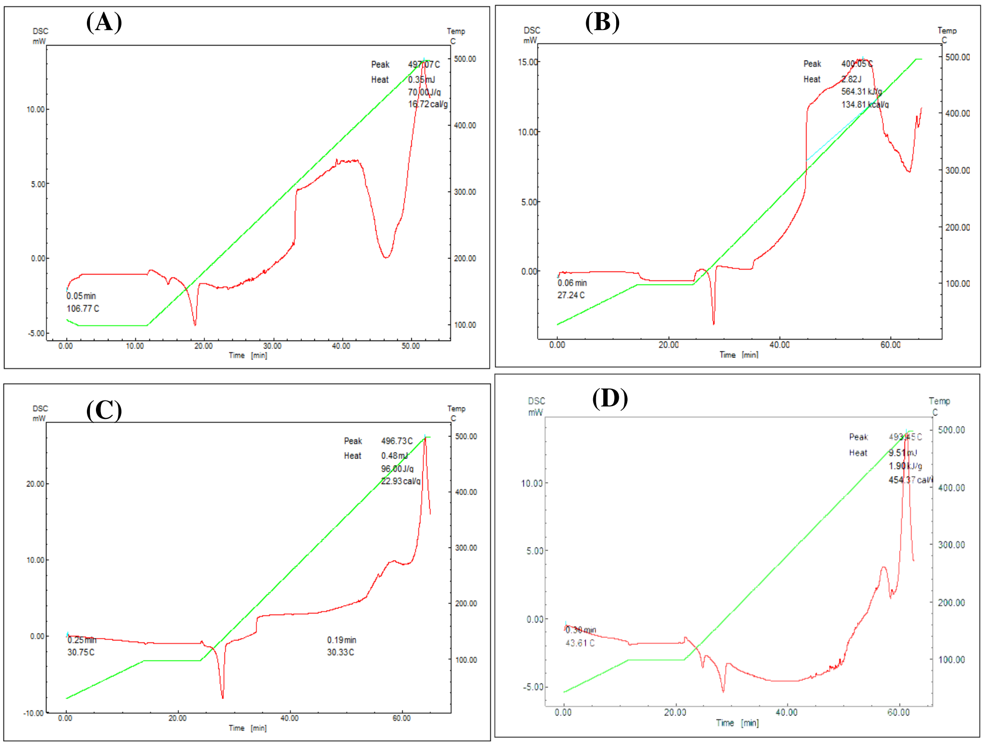

Figure 4. DSC analysis results based on differences in the size of the filler, (A) 63 µm, (B) 75 µm, (C) 90 µm and (D) 106 µm.

In Figure 4, we can see the increase of or decrease in the melting point and decomposition temperature of each of the composite material. Changes in the value of the melting point of the HDPE matrix due to the influence of EFB filler, so the lower the temperature needed to melt the material. For the decomposed temperature, the composite test results show a temperature rise compared with the properties of HDPE matrix, can be the caused by the stronger interaction between the mixture of materials so that the results obtained have a high burning temperature. The difference in the size of the filler does not significantly affect the thermal properties of the composite.

Impact analysis

Impact analysis aims to find a collision stronger composite bump given load. Impact is a measure of the energy needed to break the material. An agent may be able to determine the level of impact is acceptable, the power bump material is larger then possessed a good rate.

Strong decline bump can be damaged, therefore a strong need to bump conventionally measured or where it was done by Impact testing/clash. In carrying out the Impact testing process, the procedures adopted must fit certain standards that apply to the results obtained and widely applicable. Various standards that can be used is the

ASTM (American Standard for Testing Materials), JIS (Japanese Industrial Standard), DIN (Deutsche Industrie Normen).

Table 5 Impact analysis HDPE/EFB micro composites

No.	Size(µm)	W*(mm)	T(mm)	*Impact*(J/m^2)
1.	63	11	2,17	4943,4
2.	75	11	2,44	2537,2
3.	90	11	2,11	1573,6
4.	106	11	2,41	2516,0

*W = Width (mm), T = Thick (mm)

Oil palm empty fruit bunch composites with HDPE matrix with a high value provides enhanced Impact Impact on filler size 63 µm, as much as 4943.4 (J/m^2). Impact test data plots for recycled HDPE matrix and oil palm empty fruit bunch fibers shown in Figure 4 effect of heat treatment on the polymer causes a decrease in the value homopolymer Impact generated, the heat treatment turns into a brittle polymer, which is in line with the increase of data tensile strength values.

Similarly, the composite filler size 63 µm. Impact strong value enhancement occurs in the composite filler size 63 µm after heating at 10^{o}C. No clear trend in the formation of heat treatment on the Impact value is related to the complexity of polymer crystallization in the presence of fibers as reinforcement in polymer

Conclusions

Results achieved from this research is a method processing EFB filler and filler products with micrometer size. In this study the process of blending HDPE with fillers can be done by adding xylene 70% into 0.8 grams of MAPE and blending at temperatures 45-60^{o}C. Composite obtained in this study have filler size 63, 75, 90 and 106 µm. Composite board that has a filler size larger then the value of the tensile strength of the composite board the smaller the amount of 3,969 MPa. Value of the average melting point obtained in the HDPE matrix composite board using the 400,05^{o}C-497,07^{o}C, the value is greater than the value of the HDPE melting point 135^{o}C. EFB filler and HDPE matrix in the ratio 30%: 0% reacted so well that groups on both the raw material still present in the composite boards were formed. The smaller the size the better filler density and the resulting reaction because of all the filler can distribute homogeneously with the matrix. The larger filler used then causes the resulting strong bump commensal reduced. The results of this study can be applied as part of the furniture, electronic elements, casings, etc., because of its high heat resistance, is a semiconductor, and not easily broken and durable.

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