

Heat Deflection Temperature (HDT) Properties of Polypropylene Composite Reinforced Cellulose Microfibrils of Oil Palm Empty Fruit Bunch and Frond

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Abstract

Polypropylene composites reinforcing with natural fiber is potentially applied for automotive particularly on interior part design. Those kind of composites were contributed on renewable material, rapid rate biodegradation, and low cost of production compared to synthetic fiber. Furthermore, the mechanical properties including strength, young modulus, and thermal stability have revealed good performance than glass fiber. Fiber which were fibrillated and have high aspect ratio that correspond to diameter and length ratio of the fiber were noticed as enhancement factor for mechanical properties. Fiber fibrillation processing into microfibrillated cellulose (MFC) attempts for widening surface area of the fiber that improve polymer matrices compatibility. MFC from empty fruit bunches (EFB) and oil palm frond (FB) fibers were performed as pulp by mechanically and chemically treatments. Chemically treatment was conducted with bleach and unbleach procedure. Polypropylene with fiber was mixed using kneader, and injection for molding process. Manufacturing uses needs appropriate size precision, moderate lead time, and low defect. Heat deflection temperature (HDT) provide information for plastic material on indicating temperature condition effect to material deformation during normal loading. Material of origin, additive or filler size, and molding temperature were directly correlated to the HDT performance. Initial temperature of HDT exhibits different value for different kind of fillers and fiber treatments. PP/EFB composite by mechanical treatment gives high value of HDT compared to the fiber processing by chemical treatment both with bleach and unbleach process. Similar result have been performed in PP/FB composites related to initial temperature. PP/ EFB composite with 30% of fiber loading represent HDT in 149.4°C, and for PP/ FB composite with 30% fiber loading gives 150.7°C. By the addition of fiber loading could improve the HDT value of the composites.

Keywords: oil palm empty fruit bunches , frond, polypropylene composite, HDT.

Introduction

Over the demand of sustainable and renewable material in several field such as automotive, medical, and food packaging, hence natural based material widely investigated in last decade. Natural fiber utilization for reinforcement material in polymer matrices especially in automotive part besides reduce the synthetic and metal material consumption that costly high but also could reduce total weight of cars which about 30% (Subyakto *et al.* 2009). Furthermore, by the natural fiber addition could overcome for green house effect since as an organic material, natural fiber could assist on carbon dioxide cycle (Hari 2012). Mostly in automotive manufacture remain use synthetic fiber and metal as main material. Synthetic fiber commonly used as reinforcement for plastic composites applied on interior part. The advantages of natural fiber related to renewable, biodegradable, good properties, low cost production (i.e. synthetic fiber), low density, non abrasive, relatively active surface, low carbon dioxide emission, and most of all as a green movement material (Zimmermann *et al.* 2004; Bhatnagar *et al.* 2005; Kamel 2007; Ashori 2008). Biocomposites is defined as material consisting of natural fiber as reinforcement and polymer as matrix. Polymer usually used in automotive manufacture are provided from petroleum derivatives based such as Polypropylene and Polyethylene. Moreover by the addition of natural fiber,

directly affect for polymer consumption reduction which better properties indeed. Molding process commonly used for automotive part are compression molding and extrusion followed with injection molding. However, the properties related such as mechanical, chemical, and thermal are disconnected for each molding process (Liu *et al.* 2007).

In common with wood, natural fibers have composition consist of cellulose, hemicellulose, and lignin. Secondary wall is imbedded by primary wall, in which cellulose mostly located. Cellulose is as crystalline part, while hemicellulose and lignin are amorphous part (Jonoobi *et al.* 2009). The depiction of composite are expressed in fibers that cellulose act as reinforce and the amorphous part (i.e. hemicellulose, lignin) role as matrix. (Subyakto *et al.* 2009). Crystallinity of the cellulose are required in order to obtain high strength materials, so that some treatment are applied to degrade amorphous part particularly. Crystalline cellulose possessed 150 GPa in elastic modulus, higher than fiber glass (85 GPa) and aramid also (65 GPa) (Samir *et al.* 2004). Cellulose from non wood source could be an options such as flax, rutabaga, sisal, pineapple, coconut fiber, kenaf, and abaca (Bhatnagar *et al.* 2005) and also from alga, tunicate, and enzymatic isolation (Iwamoto *et al.* 2007).

Potential lignocellulosic source especially from industrial and agricultural or plantation waste is oil palm solid waste such empty fruit bunch (EFB) and palm frond (PF). According to the data oil palm plantation in Indonesia

reach 8.9 million hectare in 2011. The CPO production upgrading positively affect to increase oil palm solid waste. The potency are predicted 25 million tonne/ year for EFB and PF could achieve 89 million tonne /year. The utilization of EFB have been conducted with some kind of matrices such Polypropylene (PP), Polyvinyl chloride (PVC), Natural rubber, Phenol Formaldehyde (PF), and Polyurethane (PU). Chemical modification was carried out in order to enhance compatibility between fibers and polymer matrix (Shinoj *et al.* 2011). Several researcher reported the uses of EFB for reinforcing agent to PP matrix through out physical, chemical, and mechanical treatment (Sharkh *et al.* 2004; Khalid *et al.* 2008; Haque *et al.* 2009; Norul *et al.* 2013). Fiber composition strongly affected for mechanical properties of the composite (Khalid *et al.* 2008), similarly the surface modification with chemical treatment resulted strong hydrogen bonding between fibers and matrices (Haque *et al.* 2009; Norul *et al.* 2013). Norul *et al.* (2013), reported good resistance expose from PP/EFB composites including climate resistance and thermal stability under UV lightening.

Heat Deflection Temperature (HDT) is applied as an important parameter for material design. This property is used to decide appropriate processing technology particularly for high temperature manufacturing. HDT were applied to binary polymers and plastic composite for thermal property investigation since along time (Pava *et al.* 1974; Wong *et al.* 2003). Polymers having high degree of crystallinity with presence of fillers enhanced for HDT (Wong *et al.* 2003; Huda *et al.* 2006). Nano sized particle used for reinforcement or fillers could increase the HDT value even in small amount of fibers addition with well dispersion on matrices. (Thomas *et al.* 1996; Martins *et al.* 2009). Some factors that could enhance the HDT value were glass transition temperature, crystallinity, and fibers loading (Huda *et al.* 2006).

This research were aimed to investigate the HDT value from PP composites reinforced with empty fruit bunch and palm frond fibers. The effect of fibers composition both EFB and PF onto PP also analyzed toward to HDT value.

Experimental Methods

Materials

Polypropylene (PP) copolymer impact grade for automotive was used as the matrix and Maleic anhydride Polypropylene (MAPP) was used as a coupling agent. Empty fruit bunch (EFB) and Palm frond (PF) were obtained from Subang, West Java. The fibers firstly processed with mechanical and chemical treatment before applied onto polymer matrices.

Mechanical and Chemical Pulp Preparation

The fibers by mechanical treatment were cutted using drum chipper, ring flaker, and hammer mill to obtain 0.5-1 cm of fibers. Further, obtained fibers were immersed in hot water with 100°C within 1 hour, ratio between water and fiber was determined at 1 : 10. Subsequently, treated fibers

were milled with coarse disc refiner in 2 times, and finer in 5 times of circulation. Mechanical pulp were sieved with Jhonson screen, and kept under freeze condition.

Kraft pulping process were carried out as the chemical treatment. 400 OD of cuted fibers were subjected onto NaOH (19% w/w) and Na₂S (30% sulfidity) with krafting solution ratio larutan pemasak 5 : 1. Conditioned fibers were pulped using rotary digester under 160-170°C within 3.5 hours (lead time in 2 hours and processing time in 1.5 hours). Unbleached pulp were continuously washed, refined, and filtrated with Jhonson screen. Whereas, bleached pulp were processed under C-E-H (Chlorination-Extraction-Hypochlorotation) methods. Chlorination step were conducted using Cl₂ with 3.5% in consistency under 70°C in processing temperature. NaOH (1.5% w/w) were determined for extraction with 10% in consistency under 70°C within 90 minutes. Hypochlorotation were carried out with NaOCl (2% w/v) under 40 ° C within 240 minutes and 2% in consistency.

Production of Composites

Dried pulp were fibrillated using disc mill which have been dispersed in dry condition. PP were mixed with treated fiber (i.e. mechanical, unbleached pulp, bleached pulp) using kneader Daton under 160-170°C along 30-90 minutes of process. The mixed compound were milled with crusher to obtain uniform particle size and assist for feeding process. Then, were molded using injection molding (capacity 70 tonne) under injection condition at 230, 220, and 210°C respectively from feeder, and cooling time within 25 seconds. Resulted test piece were kept under vacuum bag to maintain the moisture content.

Heat Deflection Temperature Analysis

HDV 1 manual DTUL/ Vicat System ATLAS were subjected to analyzed the HDT value for all samples. The analysis based on ASTM D-648 technical test. The samples were placed edgewise with specimen dimension in 127 x 12.7 x 6.35 mm. Silicon oil GE SF 81-50, AK 1000 were used for heater medium under minimum flash temperature seted at 20°C above test temperature. Heating rate were determined in 2°C/minutes untill maksimum temperature reached 200°C. HDT value were noted as the sample undergo on deflection along 0.25 mm with normal loading.

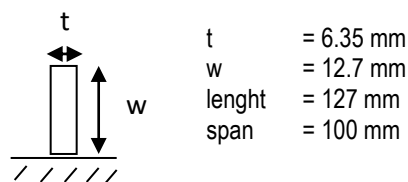


Figure 1. Configuration of HDT sample test.

Results and Discussion

Characterization of Composites

Cellulose content from EFB and PF relatively high. On PF fibers, cellulose content reach 62.34% and lignin 14.81% (Syamani *et al.* 2012). While cellulose content for EFB fibers is known in 41.72%, and other content such hemicellulose in 24.26%, lignin 22.49%, and extractive 11.54% (Anita *et al.* 2011). As depict on Fig. 2, that bleached pulp rather bright

than unbleached pulp, while mechanical pulp almost like bleached pulp. Delignification and pulping process could enhance for cellulose content about 20.48%, 79.45%, and 85.4% respectively for mechanical pulp, unbleached pulp, and bleached pulp, simultaneously lignin content reduce and significantly decrease for extractive content of EFB fibers (Kusumaningrum *et al.* 2012). Beside of cellulose crystallinity, -OH groups on cellulose gives strong hydrogen bonding with polymer matrices.

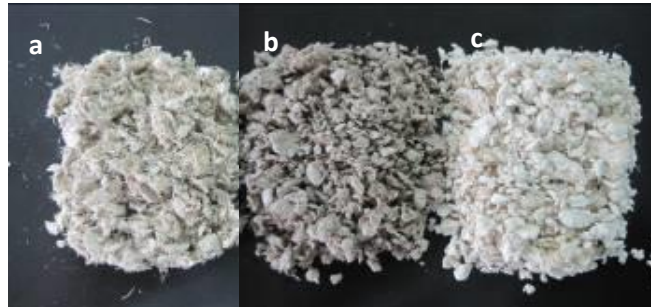


Figure 2. EFB pulp (a) mechanical pulp, (b) unbleached chemical pulp, (c) bleached chemical pulp.

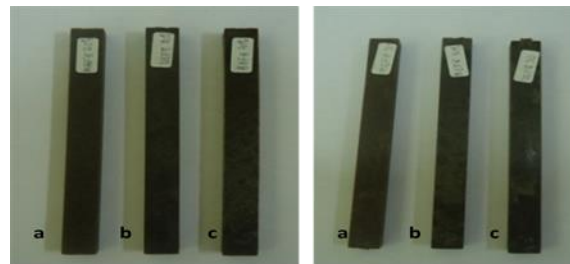


Figure 3. PP/EFB composites (a) mechanical pulp (EFB M), (b) unbleached pulp (EFB UB), (c) bleached pulp (EFB B) (Munawar *et al.* 2012).

Table 1. Composition of PP/EFB fibers composites and PP/PF fibers composites

Composites	Composition
EFB M 20	Mechanical EFB Fibers pulp 20%, PP 75%, MAPP 5%
EFB M 30	Mechanical EFB Fibers pulp 30%, PP 65%, MAPP 5%
EFB UB 20	Chemical Unbleached EFB Fibers pulp 20%, PP 75%, MAPP 5%
EFB UB 30	Chemical Unbleached EFB Fibers pulp 30%, PP 65%, MAPP 5%
EFB B 20	Chemical Bleached EFB Fibers pulp 20%, PP 75%, MAPP 5%
EFB B 30	Chemical Bleached EFB Fibers pulp 30%, PP 65%, MAPP 5%
PF M 20	Mechanical PF Fibers pulp 20%, PP 75%, MAPP 5%
PF M 30	Mechanical PF Fibers pulp 30%, PP 65%, MAPP 5%
PF UB 20	Chemical Unbleached PF Fibers pulp 20%, PP 75%, MAPP 5%
PF UB 30	Chemical Unbleached PF Fibers pulp 30%, PP 65%, MAPP 5%
PF B 20	Chemical Bleached PF Fibers pulp 20%, PP 75%, MAPP 5%
PF B 30	Chemical Bleached PF Fibers pulp 30%, PP 65%, MAPP 5%

Table 2. Density of PP/EFB and PP/PF composites.

Composites	Density (g/cm ³)
EFB M 20	0.942
EFB M 30	1.014
EFB UB 20	0.977
EFB UB 30	0.989
EFB B 20	0.976
EFB B 30	1.017
PF M 20	0.947
PF M 30	0.963
PF UB 20	0.978
PF UB 30	1.029
PF B 20	0.982
PF B 30	1.027

Composites composition between PP and EFB (PP/EFB) also PP and PF (PP/PF) with various fiber loading are given in Table 1. The opposite properties of PP and fibers which PP in hydrophobic and fibers in hydrophilic require MAPP as a coupling agent in order to improve polymer compatibilities onto fibers. According to previous study (Gopar *et al.* 2010), optimum MAPP loading in 5% (w/w) could increase the mechanical properties for PP reinforced with EFB fibers. Over limit MAPP could cause self entanglement that directly decrease the mechanical properties, similarly under limit MAPP could cause MAPP does not well grafted onto PP chain and further lacking of hydrogen bonding.

Composites feature from PP/EFB and PP/PS with mechanical and chemical pulp both unbleached and bleached are not significantly different particularly in colour appearance, as shown in Fig. 3. Some spot appeared in the

composites surface, that probably created by greatly different of morphological and physiological properties between PP and fibers, because of gap, shear stress, and quenching during injection process. Table 2 is informed the enhancement of composites density by addition of EFB also PF fibers, even in slightly differences. Composites that reinforced with mechanical pulp gives lower density than chemical. It could be cause higher volume fraction of chemical pulp both unbleached and bleached after milling treatment using disc mill.

Heat Deflection Temperature (HDT) of PP/EFB and PP/PF Composites

HDT is widely used in material automotive industry and predicted some kind of problems related materials deformation. HDT value for PP/EFB composites are explained in Fig. 4, and Fig. 5 is revealed for PP/PF composites. The addition of EFB also PF fibers on PP matrices could enhance the HDT value. The HDT for pure PP were known at 113.6°C (Zhang *et al.* 2002) testing based on ASTM D 648 with deflection point at 0.25 mm under 1.82 Mpa loadings. Range about 8.71-32.66°C of HDT enhancement were obtain from PP/EFB, whereas 23.6-31.51°C from PP/PF composites. Presence of fibers could be as nucleating agent in crystallisation process, so that improve strength and thermal stability of composites either PP/EFB and PP/PF and continuously affected on HDT value enhancement. Huda *et al.* (2006) reported some parameters that influence HDT value of polymer, such crystallinity and reinforcement agent or filler. Kenaf fibers reinforcing onto PLA could improve the HDT value reaches 2 times than pure one in 40% fiber loading, because of spherulite formation during crystallisation process (Huda *et al.* 2008).

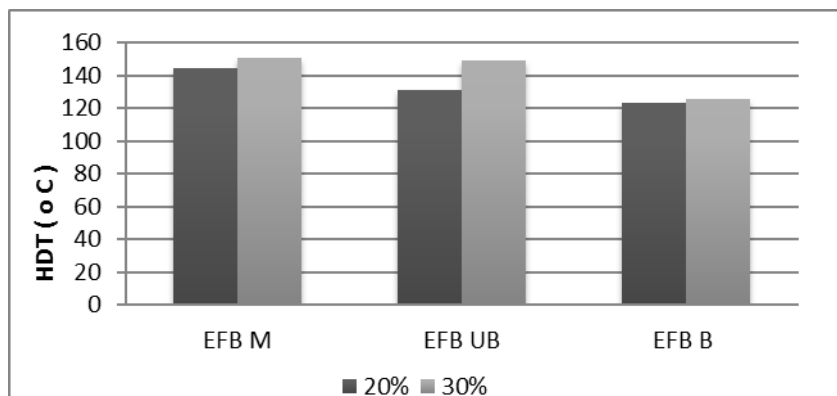


Figure 4. HDT value of PP/EFB composites for each treatment and fiber loading.

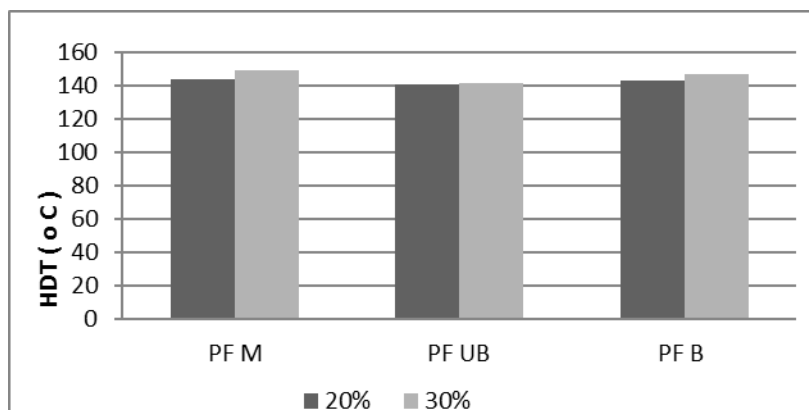


Figure 5. HDT value of PP/PF composites for each treatment and fiber loading.

Fiber loading in composite positively affect on HDT value. HDT value were increased with higher proportion of fiber loading for all kind of pulp, mechanical and chemical both for PP/EFB and PP/PF composites. HDT enhancement by fiber loading addition level result about 3.92%, 12.08%, and 1.59% respectively for mechanical pulp, chemical unbleached pulp, and bleached pulp for PP/EFB composite. While for PP/PF composites about 3.61%, 0.5%, and 2.72% respectively for mechanical pulp, chemical unbleached pulp, and bleached pulp. Fiber loading on CAP composites improved about 13 and 30% for composition under 15 and 30% (Mohanty *et al.* 2004). While, HDT value from PHBV/ wood flour composites increased 24°C through 40% in composition (Singh *et al.* 2007). As previous report at Huda *et al.* (2008), trancrystallinity spherulite formation were affected by nucleating agent quantity.

With optimum composition, presence of fibers could improve the strength and tensile modulus that corelated to HDT value. Pulping process of fibers were aimed for delignification in order to improve cellulose content that could refer to crsytallinity form. Beside that, could extensified suface contact with matrices because smaller particle size facilitate for hydrogen bonding that directly affect for mechanical and thermal properties. As reported before in Morreale *et al.* (2008) that finer particles having greater immobilisation ability on polymer chain. Previous study, Munawar *et al.* (2012) reported PP/EFB M having greater strength modulus achieved 45.51 MPa than PP/EFB UB and PP/EFB B. The result supported for corelation between strength modulus enhancement and HDT value improvement. As depicted from Figure 4 PP/EFB M have greater HDT value than PP/EFB UB and PP/EFB B. Similary to PP/EFB, PP/PS M also have higher HDT value than PP/PF UB and PP/PF B. It could be fiber under milling process using disc mill caused lower aspect ratio of chemical pulp both unbleached and bleached due to horizontally cuted. Aspect ratio that correspond to lenght and width ratio of fibers significantly influence for plastic HDT value (Morreale *et al.* 2008). Some bubbles that were suspected as traped volatile chemical compound and water

vapor, gradually create some cavity in composite reinforcing chemical pulp as shown in Fig. 6. Nunez *et al.* (2002) adduced that volatile compound during heating process formed microcavity affected debonding between fiber and matrices.

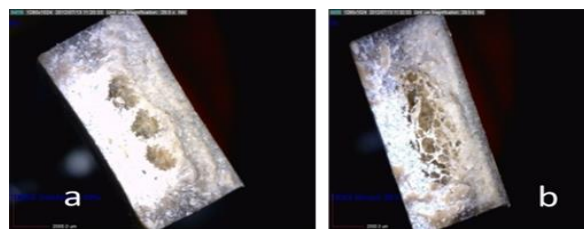


Figure 6. Microcavity on composite PP/EFB by chemical pulping, (a) EFB UB , (b) EFB B.

Conclusions

Heat deflection temperature of PP/EFB and PP/PF composites significantly improve by the addition of EFB and PF fibers with mechanical and chemical pulping. Composites reinforcing with mechanical pulp original from EFB (PP/EFB M) and PF (PP/PF M) have greater HDT value than reinforcing with chemical pulp both unbleached and bleached. Higher fiber loading relatively enhance for HDT value in all fibers treatment, mechanical pulp, chemical unbleached pulp, and chemical bleached pulp.

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