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Characterization of Nanoparticles Fe_3O_4 Nanocomposite Blend with Thermoplastic HDPE

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Abstract
This study aims to determine the mechanical properties, morphology and thermal thermoplastic blends of High Density Polyethylene (HDPE) with a filler material Fe_3O_4 nanoparticles. The method is performed by mixing the ingredients on Internal Mixer tool types Labo Plastomill 30 R I50 models with variations in the quantity (2,4,6,8) wt% Fe_3O_4 nanoparticles with a size of 33.11 nm and 2% by weight Polyethylene Grafted Maleic Anhidride (PE -g-MA) and without PE-g-MA, with a chamber volume of 60 cc with a temperature of 150^0C at a speed of 50 rpm for 10 minutes. The results of the mechanical properties obtained an increase in tensile strength maximum with increasing the quantity of nanoparticles Fe_3O_4 without and the compatibilizer PE-g-MA with optimal the quantity at 2% by weight, while the elongation at break decreased with increasing the quantity of nanoparticles Fe_3O_4, but the tensile strength better by using compatibilizer, as well as Young's Modulus, s increased with increasing magnetic nano particles, but in general without compatibilizer better. The results of the dispersion morphology of the occurrence of a homogeneous mixture and intercalates between HDPE thermoplastic matrix with Fe_3O_4 particles and homogeneous mixture. The addition amount of filler increases the thermal stability and crystallinity of composite.
Keywords: Fe_3O_4 nanoparticles, HDPE, PE-g-MA

1. Introduction

Nanoparticles become a very interesting study, because the material that is in nano size particles typically have the chemical or physical properties that are superior to large-sized materials. In this case these properties can be altered by controlling the size of the material, setting the chemical composition, surface modification and control the interactions between the particles.

Nano particles of magnetite (Fe_3O_4) for the last few years has been widely used in various applications, such as storing the information with a high density, the formation of an image with magnetic resonance imaging (MRI), a delivery system for medicines, cosmetics, dyes, as coatings to prevent corrosion, adsorption processes and as a filler for various applications nanocomposite

Compatibility is the level of integration of a mixture,compatibilizer the specific compounds that can be used to integrate incompatible polymers into stable mixture through intermolecular bonds, (Liu, H. et al, 2008).

The addition compatibilizer polietyhilen grafted maleic anhidride (PE-g-MA) is expected to improve the homogeneity and decrease the size of the mixing phase distributed HDPE, the use of PE-g-MA have been done due to very broad applications, such as blending, compatibilizer agent of the polar polymer , adhesive, and on nanotechnologies. (Jayathu.Z.E, et al, 2006)

Several studies of magnetic nanoparticles include Fe_3O_4 nanoparticles with Alginic Acid (Kazmierczak.M,et al,2012), Fe_3O_4 nanoparticles - Au core, (Montazeri,H,et al,2013),Fe_3O_4 Nanoparticles - Carbon (Prakash, Raju ,et al,2013), oxide nanoparticles Fe_3O_4- garfit (Zhang.Xiao,et al, 2014), nanoparticles Fe_3O_4 with thermoplastic LDPE (Zhang,Dong,et al, 2012), nanoparticles Fe_3O_4 with rice Straw (Khandanlou, R,et al, 2013) .

Has been a lot of research using HDPE as the thermoplastic matrix and nanoparticles as fillers, among others; $CaCO_3$ / HDPE (Saeedi.M, et al,2011); Zebarjad, et al. 2006), Graphite / HDPE (M.Sarikanat, et al., 2011), Clay / HDPE (Pegoretti, A, et al, 2007), natural bentonite / HDPE (Bukit.N, et al, 2013), rice husk ash / HDPE (Ginting E.M, et al 2014), zeolite / rice husk ash / HDPE, (Ginting, E.M, et al 2015), zeolite /LDPE, HDPE (Kim.H ,et al 2006).

This study aims to determine the mechanical properties, thermal properties, morphology of HDPE thermoplastic composites with nano filler Nano magnetic particles (Fe_3O_4), using compatibilizer PE-g-MA and without compatibilizer

2. EXPERIMENTAL

Materials

Fe_3O_4 with 33.11 nm particle size of the preparation result (Bukit.N, et al, 2015), Thermoplastic HDPE produced Nusantara PT Titan Petrochemical, PE-g-MA Production Sigma Aldrich USA, HCl with Molarity 37% Sodium Hydroxide (NH_4OH) molarity 25% Merck KGaA 64271 Darmstadt Germany. Poly ethylene Glycol (PEG) – 6000

Equipment

Universal Testing Machine (UTM) stograph R-1 merek Toyoseki Jepang. Internal Mixer Labo Plastomill model 30 R I50 Technical Cooperation Bythe Government of Japan. SEM, (Model Zeiss) ,Thermal Gravimetry (TGA).

The Preparation of Nanocomposite

The preparation of nanocomposite made by blend thermoplastic HDPE with Fe_3O_4 nanoparticles which have been prepared through coprecipitation method with the size (33.11 nm). As this mixing by using Labo Plastomill Mixer Internal models 30 R I50 with variations in composition (2,4,6,8) wt% Fe_3O_4 nanoparticles and 2% by weight of PE-g-MA is shown in Table 1, with a chamber volume of 60 cc with a temperature of 150^0C at a speed of 50 rpm for 10 minutes.

Table 1. The Quantity of Mixed Nano Composite Material

Material	The Quantity blend (%) wt							
	S_{1A}	S_{2A}	S_{3A}	S_{4A}	S_{1B}	S_{2B}	S_{3B}	S_{4B}
HDPE	96	94	92	90	98	96	94	92
Nanoparticle Fe_3O_4	2	4	6	8	2	4	6	8
PE-g-MA	2	2	2	2	-	-	-	-

From internal mixer tool produced samples of granular. The resulting composite mold tool inserted into rectangular plate with a thickness of 1 mm, length 11 cm, width of 11cm. Furthermore, the printing of the printing press and heat by means of Gonno Ramdia 152 mm Ramstroke 150 mm performed for 10 minutes consisting of time heating the mold 3 minutes of heating time material 3 minutes and press 4 minutes with 50 kgf / cm^2 at a temperature of printing 150^0C, followed with cold pressure for 4 minutes with 50 kgf / cm^2 at a temperature of 22^0C.

The mechanical properties of nanocomposite was tested using a Universal Testing Machine (UTM) . Tests conducted using a standard JIS K 6781 with a pulling speed of 50 mm / min

3. RESULTS AND DISCUSSION

3.1 Analysis of Mechanical Properties

From the results of tensile test, it can display a graph the relationship between variations in weight percent filler with tensile strength for each sample using a compatibilizer PE-g-MA and without compatibilizer as in Figure 1 to 3

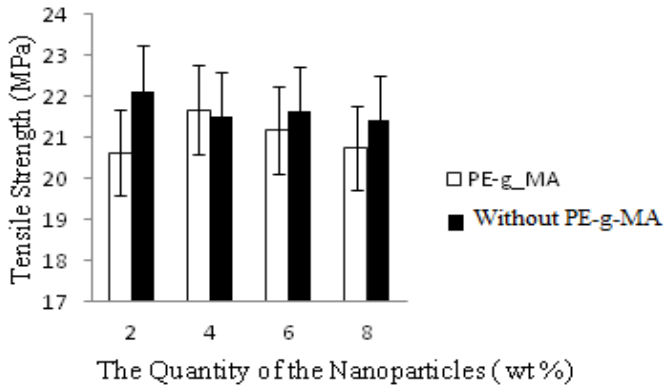

Figure 1. Relationship Between the Tensile Strength with the Quantity of Magnetic Nanoparticles

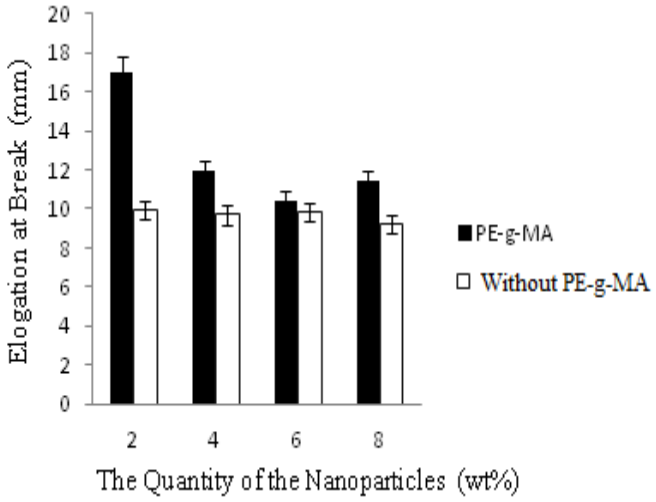

Figure 2. Relationship Between the Elongatiot at Break with the Quantity of Magnetic Nanoparticles

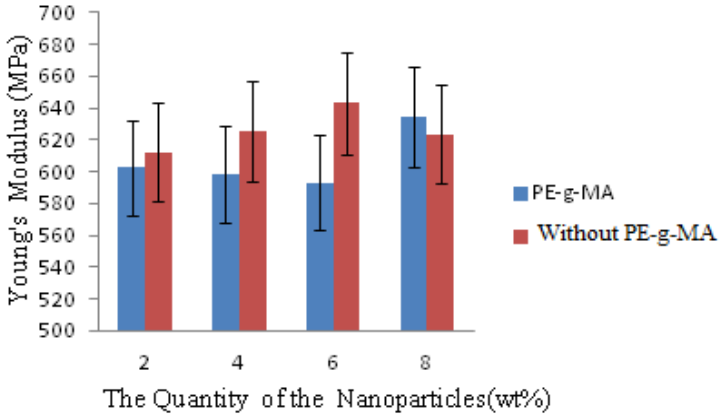

Figure 3. Relationship Between the Young's Modulus with the Quantity of Magnetic Nanoparticles

As shown in Figure 1 relationship of tensile strength to the quantity of filler where in the tensile strength without the use of compatibelizer value is higher compared to using compatibelizer and conversely in figure 2. The relationship between the elongation at break of the quantity of nano particles which by using compatibilizer PE-g-MA is greater , this is due to Force anhydride in PE-g-MA is an important role in improving the mechanical properties of the mixture in the ultimate elongation, of the nature of PE-g-MA addition to depending on the degree of grafting MA, but it can also be determined by the distribution of Maleic Anhidride (MA) within the molecular chain polyethilen (PE). (Machado, et al, 2005).

The optimum value of tensile strength between the use of PE-g-MA and without PE-g-MA on the quantity of the filler (2%) by weight. This shows that the quantity of this nanocomposite has the best tensile strength. As for the elongation at break where the optimum value on the quantity of the filler (2%) by weight and the use of PE-g-MA.

From Figure 3 Youngs modulus optimum is in the quantity of the filler (6%) weight without using PE-g-MA. From the figure shows that the quantity of the nano-particles as a filler in (8%) weight had better grades by using PE-g-MA. This shows a lack of atomic bonding between the particles and fillers for the nanocomposite and no spread of the particles evenly. Crystal phase difference between the filler with the thermoplastic matrix also affects the tensile strength is not good. So the results of tensile testing of nanocomposite HDPE / nano Fe_3O_4 with increasing magnetic particle quantity decreased tensile strength at above 8% by weight of the quantity. By using compatibelizer give better effect to the elongation at break of the composite, while the effect without compatibelizer tensile strength and Young's modulus .

Based on research (Ginting, M.E, et al, 2015), on the quantity of 2% by weight with compatibilizer PE-g-MA where its strength value of 23.97 MPa. When compared with the results of this study in the same the quantity using Fe_3O_4 Nano particles that is obtained at 20 636 MPa. This indicates that the bonding between atoms that occurs in every nanoparticles used as a filler in thermoplastics HDPE vary. Where there is a strong bond that occurs and there is a weak bonds. As for the results of tensile strength using nano-particle filler bentonite as filler material thermoplastic HDPE (Bukit.N,et al, 2013) on the quantity of 2% by weight to produce a tensile strength of 25 377 MPa. This is due to lack of bonding between Fe_3O_4 compared with thermoplastic matrix of silica and

bentonite and clay .In addition clotting occurs due to nanoparticles, causing inequity spread of nanoparticles in which it also greatly affects the tensile strength of nanoparticles.

According to research (Feng et al, 2004), states that the nanometer-size reinforcing materials such as silica, calcium carbonates and clay, a material that can also function as a compatibilizer between polymer blends are not mutually dissolve. Figure 4 shows a sample form JIS K 6781 before was treated and after testing elongation at break.

Figure 4. Sample Nanocomposite (a) before, (b) after the Tensile Test

3.2 Morphology Analysis

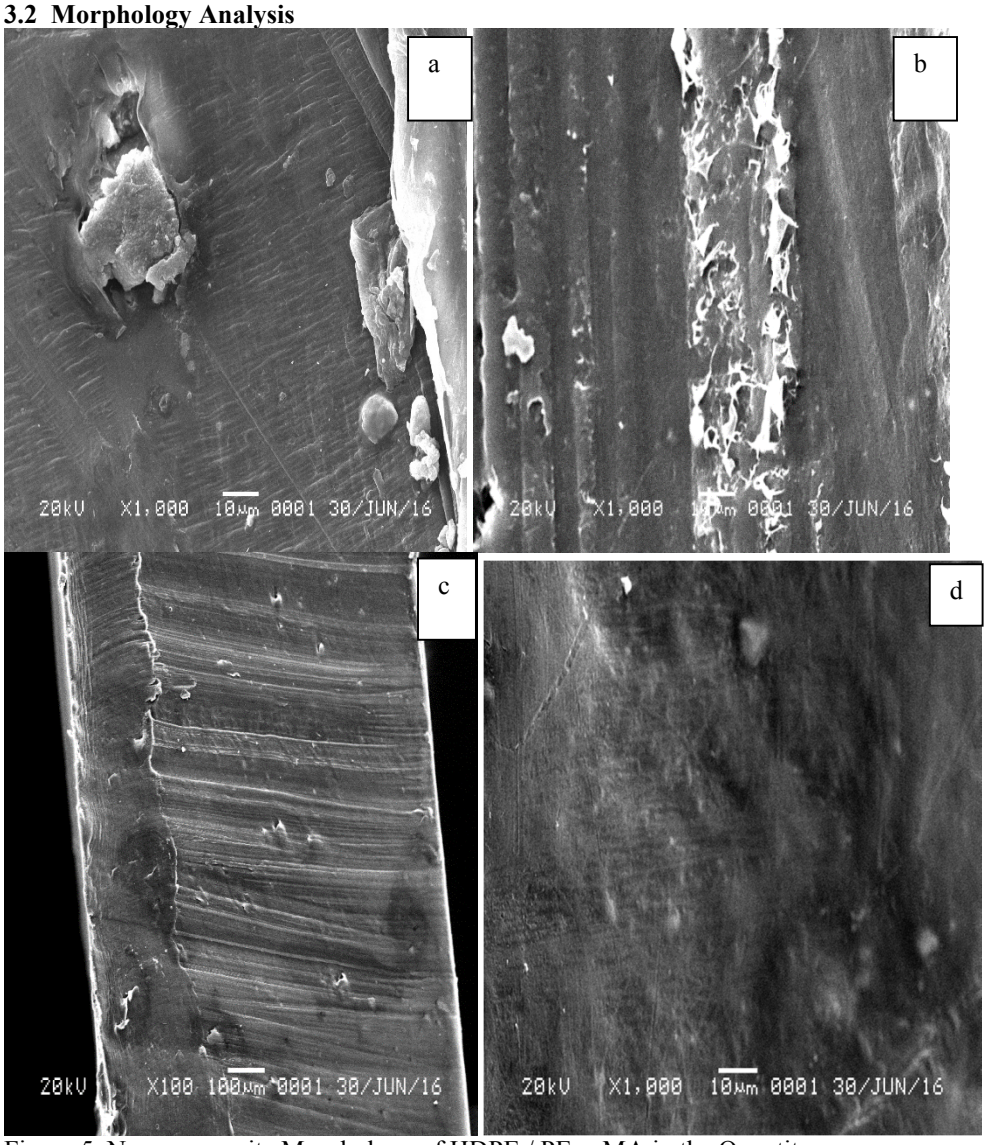

Figure 5. Nanocomposite Morphology of HDPE / PE-g-MA in the Quantity Fe_3O_4 (a), (2%) , (b) 4% , (c) 6% , (d) 8%

From the analysis of the mechanical properties of the mixture of nano Fe_3O_4 more than 8% by weight on the contrary have negative effects that lower the elongation at break but larger if without filler nano-particles, is probably caused by a decrease in the degree of spread of exfoliating particles of Fe_3O_4 on nanocomposite containing nano high particle (> 6% wt), it is their agglomeration or clotting nano particles as shown in photo scanning electro microscope (SEM) in Figures 5 and 6. this case led to a decrease in tensile strength.

Agglomeration Fe_3O_4 believed to be a stress concentration and becomes the beginning of the crack so that the power will go down. The same research (Z.A.Kusmono , et al, 2008) . Nano clay more than 4 phr contrary negative effect that lowers the tensile strength. This is likely due to a decrease in the degree of exfoliation deployment of Fe_3O_4 layer on the nano composites. Similarly, the results of research (T.Serki, et al, 2006), reported by the addition of compatibilizer will form the esterification reaction or hydrogen bonding at interfaces hydroxyl groups on the particle on one side and the carboxylic group in the compatibilizer which diffuses into the polymer matrix on the other side and a homogeneous mixture.

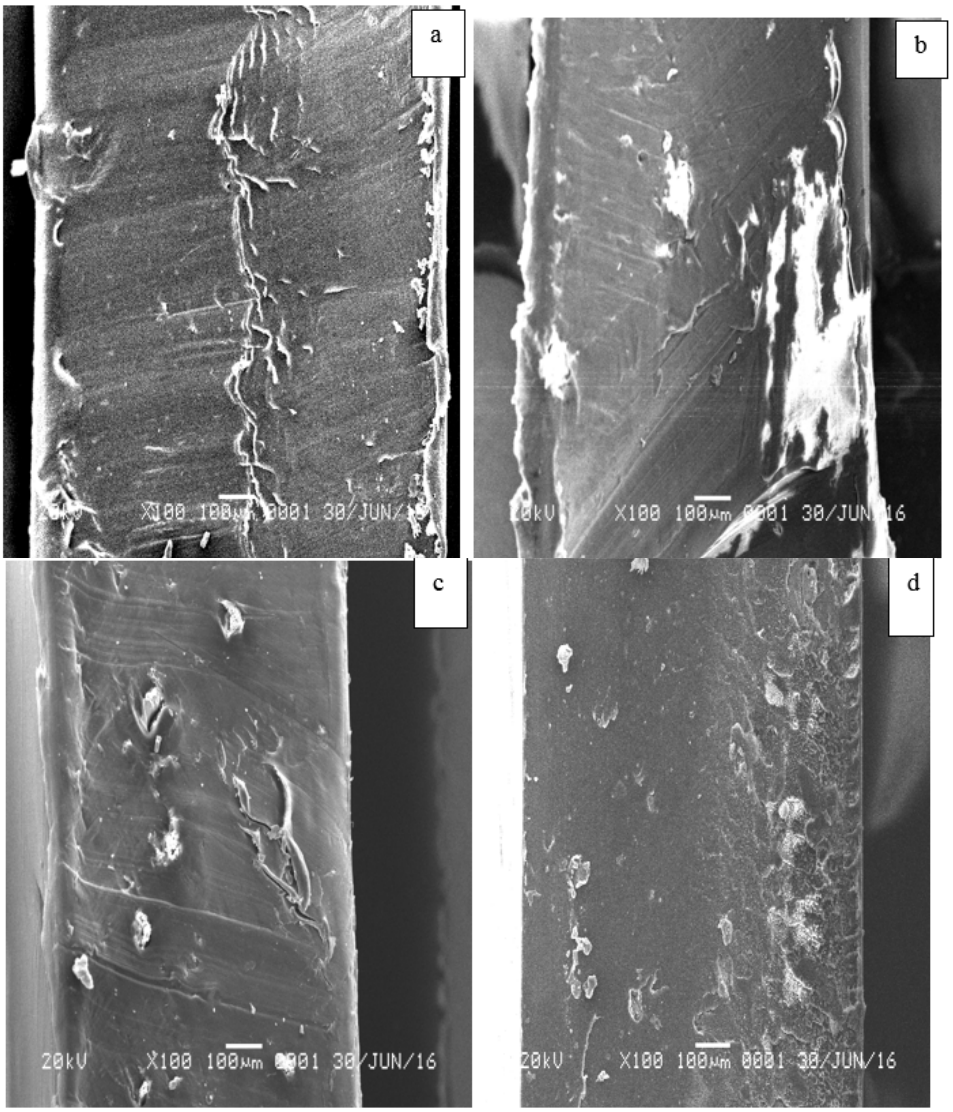

Figure 5. Nanocomposite Morphology of HDPE / Fe_3O_4 in the quantity
(a) 2% , (b) 4 % , (c) 6% ,(d) 8%

3.3 Analysis Thermal

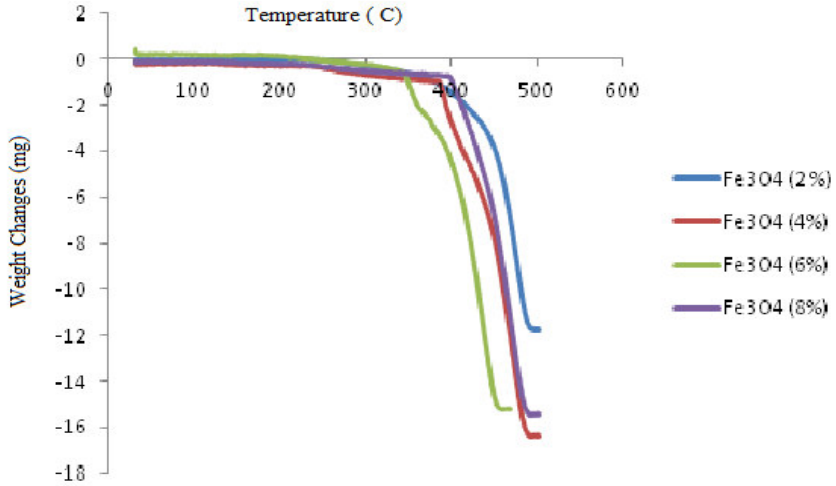

Figure.7 Curva TGA Nanocomposite HDPE/PE-g-MA/ Fe_3O_4 in the Quantity Fe_3O_4 (2,4,6,8)wt %

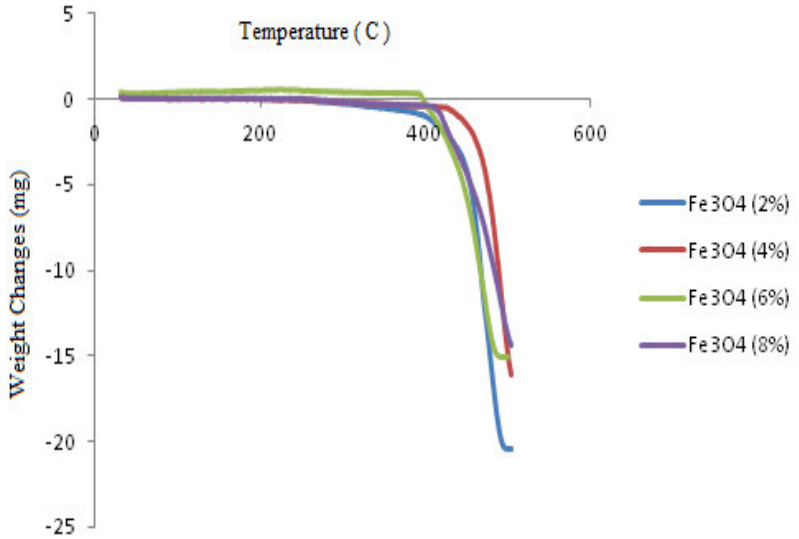

Figure .8 Curva TGA Nanocomposite HDPE/ Fe_3O_4 in the Quantity Fe_3O_4 (2,4,6,8) wt %

Table .2 Weight Changes in Nanocomposite HDPE / Fe3O4 with and without Compatibilizer

HDPE/PE-g-MA/ Fe_3O_4 (wt %)	ΔM (mg)	HDPE/ Fe_3O_4 (wt %)	ΔM (mg)
2	-11,788	2	-20,484
4	-16,153	4	-16,888
6	-15,390	6	-15,590
8	-15,315	8	-14,817

From Figure 7 and 8. and Table 2 shows the quantity of nanoparticles Fe_3O_4 2% weight smaller mass reduction compared with 4% of 16.153 mg of a mixture of HDPE / PE-g-MA / Fe_3O_4, this is because the more content of nano-particles, the greater the decomposition process, so that the thermal stability as well. While on a mixture of HDPE / Fe_3O_4 without compatibelizer seen the quantity of nanoparticles Fe_3O_4 2% by weight, 20.484 mg had a change of times greater than the on the quantity of 8% by weight ie 14.817 mg, the temperature melting point of HDPE about 140^0C, with the addition of filler there is an increase in the melting point the filler particles Fe_3O_4 nanoparticles in the quantity of 4% wt. This is caused by the increased dispersion of the bond between face diatara filler material polyethylene and PE-g-MA and nano particles of Fe_3O_4, the same can be obtained from the study (Pracell, et al, 2006), the addition of compatibelizer PE-g-MA, which could improve crystallization composite polyethylene (HDPE), this is caused by branching chain between maleic anhydride and a better dispersion between PE-g-MA in polymer materials.

To be considered a worthy polymer is heat stable or heat resistant, the polymer should not decompose under the temperature of 400^0C and maintain its characteristics at a temperature close to the decomposition temperature, from figure 7 and 8 at a temperature of 500^0C all polymer materials already have decomposed the rest is Fe_3O_4 nanoparticles.

From the curve TGA decomposition occurs at a temperature of 495 ^{0}C, changes the weight of the smallest 11.778 mg at 2 wt% Fe_3O_4 with compatibilizer PE-g-MA, while the top position in accordance with the curve TGA visible changes in the largest weight as much as 16.153 mg on the composition of 4% at 500^0C rest is Fe_3O_4 nanoparticles. As for HDPE mixture with Fe_3O_4 decomposition occurs at a temperature of 490^0C, the smallest changes in weight of 14.81 mg at 2 wt% Fe_3O_4 with compatibilizer PE-g-MA, while the top position according to the TGA curves visible changes as much as 20.484 mg biggest weight on the quantity of 2 % by weight at a temperature of 500 ^{0}C rest is Fe_3O_4 nanoparticles.This is consistent with the results of the study (Salmah, 2005) that the addition of fillers increase thermal stability and crystallinity of the composite.

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5. CONCLUSION

The results of mechanical properties obtained an increase in tensile strength maximum with increasing the quantity of nanoparticles Fe_3O_4 without compatibelizer or with compatibilizer PE-g-MA with optimal quantity at 2% by weight, while the elongation at break decreased with increasing the quantity of nanoparticles Fe_3O_4, but the power

breakdown better with using compatibilizer, as well as Young's modulus, s increased with increasing magnetic nano particles, but in general without compatibilizer better. The results of the dispersion morphology of the occurrence of a homogeneous mixture and intercalates between HDPE thermoplastic matrix with Fe_3O_4 particles and homogeneous mixture. The addition amount of filler increases the thermal stability and crystallinity of composites.

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