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Preparation and Characterization of Rice Husk Ash as Filler Material in to Nanoparticles on Hdpe Thermoplastic Composites

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Abstract
The purpose of this research is to create nano particles rice rusk ash used as a filler in thermoplastic high density poliethylen (HDPE) and nano-mechanical properties of the resulting composites. The method of rice husk ash made way smoothed by ball mill PM 200 for 1 hour, the filter results in a ball mill with a sieve size of 200 mesh (74 µm), rice husk ash is dissolved in 2.5M NaOH for 4 hours and then stirred with a magnetic stirrer, then at Ball mill for 15 hours at a rate of 450 rpm, nano rice husk ash is used as filler in HDPE thermoplastic composition (2,4,6,8,10) wt% were blends in an internal mixer at a temperature of 150 ^{0}C laboplastomil at a rate of 60 rpm for 10 minutes. Nano composite mechanical properties were analyzed with Universal Testing Mechanic. The results obtained rice husk ash silica composition of 89.6 wt%, the particle size of 50.6 nm, the results of the mechanical properties of tensile strength and elongation at break of an increase in the composition of the mixture 2 to 4% by weight.
Keywords : Nano Particle Rice Husk Ash , XRF, XRD, SEM, Mechanic, Analysis

1. Introduction

Waste rice husk is being very abundant in Indonesia, but its use is traditionally limited. Rice husk has now been developed as a raw material to produce ash that is known in the world as Rice Husk Ash (RHA). RHA is one of the raw material to produce silica. Nano silica has now applied in various fields including science and industry. Waste rice husk is being very abundant in Indonesia is an agricultural country. During this limited use of traditional rice husk, which is for organic materials and for burning bricks. Rice husk is an agricultural residue from rice milling process. Central Statistics Agency reported that rice production in 2011 is estimated at 67.31 million tons of milled rice rose 895.86 thousand tons (1.35 percent) than in 2010 amounted to 66.41 million tons of paddy. This means that Indonesia produced 13.462 tons of rice husk in 2011. Material rice husk ash has been used as a filler material.

Silica has been widely used as catalysts, and various kinds of organic-inorganic composite materials (Sun. L et al, 2001). In addition in the form of processed products, silica has also been used directly for purification of oil, as an additive in pharmaceutical products and detergents, as a stationary phase in chromatography columns, fillers and as an adsorbent polymer (Kamath and Proctor, 1998; Sun L, et al, 2001).

There has been much research on the preparation of nano silica from rice husk ash by way of synthesis, among others, (Thuadaij.N. Et al, 2008), (Supakorn Pukird, et al, 2009), as well as (Ezzat Rafiee, et al, 2012). From the results of previous studies have reported that approximately 20% of the weight of the rice is rice husk, and varies from 13 to 29% of the composition husk ash which is always generated whenever the chaff burned (Krishnarao.R, et al., 2000).
Rice husk has now been developed as a raw material to produce ash that is known in the world as RHA (Rice Husk Ask). RHA is one of the most raw materials rich in silica containing about 90-98% silica after complete combustion (Thuadaij, N et. al., 2008).Waste is often defined as waste material/waste materials from the processing of agricultural products. Waste destruction process naturally progresses slowly, so that the waste does not only interfere with the surrounding environment but also interfere with human health. At each rice mill will always we see that the chaff pile mountains even higher and higher. Currently the use of rice husk is still very little, so husks remain a waste material that is disturbing the environment.

The most common value content of silica ($SiO2$) in the rice husk ash is 94-96% and if its value is close to or below 90% may be caused by chaff samples that have been contaminated by other substances that lower the silica content (Prasad. CS, et al., 2001). Rice husk ash when burned in a controlled manner at high temperatures (500 - 600 ^{0}C) will produce silica ash that can be used for a variety of chemical processes.

The rice milling process is usually obtained about 20-30% husk, bran between 8-12% and milled rice from 50 to 63.5% between the initial weights of the data of grain. Husk with a high percentage of these can cause environmental problems. Therefore, today a lot of rice husk ash is used as an adjunct to construction materials. Rice husk is a biomass material such other berlignosellulosa but siliceous high. Amorphous silica is formed when silicon is thermally oxidized. Usually the amorphous silica has a density of 2:21 g/cm3, (Harsono, 2002). Rice husk silica in crystalline form (quartz) and amorphous concentrated on the outer surface and a little on the inside of the husk (Jauberthie. R, et al., 2000).

There has been much research on the preparation of nano silica from rice husk ash by way of synthesis, among others, (Thuadaij.N. et al , 2008), particle size 50 nm was obtained, (Supakorn Pukird , et al , 2009), particle size obtained 40 - 200 nm, as well as (Rafiee, E., et al , 2012).

Material of this nature generally have hydrophilic properties, then the material is generally not compatible with most polymer materials. Therefore, must be chemically modified to make the surface more hidrofobis, it is necessary for a material that is compatible with the polymer matrix , (Jacob, S., et al , 2010).

Nano-sized fillers, better known as nano filler material can be applied to the polymer nano- composite material that results in the improvement of some of the basic properties of polymers, such as thermal resistance properties, mechanical properties, chemical resistance and fuel properties (flammability). Preparation of polymer composites made by combining two different materials so as to enhance the mechanical properties of the material. The materials technology can be made in the nanoscale size, from a few studies mention that preparation composite with nano-sized fillers can improve the mechanical properties.

Results of some studies suggest that the properties of a filler material will be compatible with the polymer matrix, is influenced by several factors, among others, the size of the filler particles, wherein the particle size of a small filler can improve the degree of reinforcement of polymers compared to the larger size, (Leblance, J, 2002), as well as the smaller the particle size the higher the bonding between the filler with the polymer matrix, (Khols J, et al, 2002), the amount of surface area can be increased by the presence of a porous surface on the surface of the filler as well as with the addition of nano can improve the thermal and mechanical properties of nano-composites, (Bukit.N, 2012), as well as with adding bentonite nano filer on HDPE (Bukit. N, et al, 2013). Nano $CaCO_3$ to HDPE, (Zebarjad, S. M, et, al. 2006), nano carbon with HDPE, (Fouad, H., et al, 2011).

The use of rice husk ash in the composite can provide several advantages such as increased strength and endurance, reduce the cost of materials, reducing the environmental impact of waste materials, and reduce CO_2 emissions.

The use of silica in the composite layer can enhance the material properties (changes in cationic capacity, high surface broad, large aspect ratio), (Tjong, 2006), (Utracki, 2007), is essential to improve the physical and mechanical properties, strength tensile, modulus tensile, flexural strength, thermal stability, thermal properties, for some thermoset nanocomposite thermoplastic material and the amount of silica filler is not too much, (Koo, et. al, 2002; Wu, et. al, 2007; Lei, et. al, 2007; Kord, et. al, 2010; Samal, et. Al, 2008).

In this study the process of making nano-particles made of rice husk ash making process that results from the burning of white rice husk in rice plant then milled with a ball mill for one hours, then filtered with a 200 mesh size, equivalent to 74 µm then soaking with a solution 2.5 N NaOH for 4 hours preparation results do the ball mill for 15 hours at a rate of 450 rpm to obtain nano-meter size is used as a filler in thermoplastic HDPE.

2. Experimental

2.1 Matrials and Methods

Materials used in the study of rice husk ash, Copolimer HDPE Production of PT Titan Petrokimia Nusantara Indonesia, melting temperature of 136°C, density 0.941 g/cm3, filter paper, Polietilene grafted Maleic Anhidride (PE-g-MA) production sigma aldrich USA, NaOH.

2.2 Instrumentation

Internal Mixer Laboplastomil Model 30 R150, Hydraulic Hot Press, Cold Press of 37 ton from Genno Japan, Universal Testing Mechanic (UTM), X- Ray Difraction (XRD), Scanning Electron Microscop (SEM), X-Ray Flurosensi (XRF).

2.3 Nano Particles Preparation Process Rice Husk Ash

The procedure of this study conducted by rice husk ash white color from the burning of the rice plant, is processed with rice husk ash manner in ball mill, for 1 hour, strain the results in the ball mill with a sieve size of 200 mesh (74 μm), the ash dissolved in 2.5M NaOH for 4 hours and then stirred with a magnetic stirrer, after filtering with filter paper and vacum pump, the residue was washed with distilled water hasi then the rice husk ash in the warm up with a 100^0C oven for 2 hours, method (Dominic, M., et al .2013), rice husk ash, the results of treatment are both included in a planetary ball mill P 200 for 15 hours at a rate of 450 rpm, according to the method (Bukit. N et al 2013); (Nikmatin. S, 2013).

2.4 Blends of HDPE-PE-g-MA / Nano Particle Rice Husk Ash

HDPE-rice husk ash composites were prepared by mixing HDPE, nano Particle rice husk ash and PE-g-MA, with the composition as presented in Table 1. The mixture was placed in the internal mixer laboplastomill, and mixed at a temperature of 150°C, which is the melting point of HDPE, at a rate of 60 rpm for 10 minutes.

Table. 1 Composition Blends HDPE /Nano Particle Rice Husk Ash With Compatibelizer PE-g-MA in Internal Mixer

Materials	Blends Composition (wt %)					
	HDPE	$S_{absp.1}$	$S_{absp.2}$	$S_{absp.3}$	$S_{absp.4}$	$S_{absp.5}$
HDPE	100	95	93	91	89	87
PE-g-MA	0	3	3	3	3	3
Nano Particle Rice Ash	0	2	4	6	8	10

2.5 Mechanical Properties Measurement

Tensile strength measurement was performed according to JIS K 6781 standard using Universal Testing Machine, at crosshead speed of 50 mm min^{-1}. Young's modulus (E), ultimate tensile strength (σ_{max}), and elongation at break (ε_b) were determined from the stress-strain curves.

2.6. X-Ray Difraction (XRD) Analysis.

The XRD analysis was conducted at room temperature using X-ray difractometer type Shimadzu XRD 6000. The operating conditions used were CuK_λ radiation (λ = 0.15418 Å, produced at 40 kV and 30 mA). Pattern was recorded over goniometer (2θ) ranging from 5° to 60°. The interlayer distance of rice ash in nanocomposite was derived from the peak position (d_{001} = reflection) in XRD diffractograms according to the Bragg equation: $d = \lambda / 2\sin\theta$.

3.Result and Discussion

3.1 Analysis Composition Rice Husk Ash

Table 2. Rice Husk Ash XRF Synthesis with Solutions NaOH

Components	Composition (%wt)
SiO2	89,49
MgO	3,84
P2O5	2,19
CaO	1,08
Al2O3	1,07
Fe2O3	0,72
K2O	0,52
Cl	0,34
MnO	0,30
SO3	0,26
Cr2O3	0,06
ZnO	0,06
MnO	0,04

3.2. Analisys XRD Rice Husk Ash

From the results of X-ray Diffraction seen rice husk ash has shown this is due to the crystal pattern combustion temperatures reached above 900^0C seen from Figure 1 the diffraction patterns did not differn significantly, but there is a shift in the peak of the diffraction peaks in the preparation of rice husk ash before seen the peak maximum at 21.8779 with 2 theta angles , spacing distance d = 4.05928Å and FWHM = 0.26180 and 4784 while the peak intensity after the preparation process of dissolution with NaOH and Ball mill for 15 hours maximum peak at 2 theta angles 21.8748 at a distance d = 4.05984 Å and FWHM = 0.27420, and the peak intensity of 4451. This shows the difference FWHM rice husk ash particles get smaller. In Table 3. Shows the maximum difference between the three peaks of pure rice husk ash with rice husk ash dissolved with NaOH and the ball mill for 15 hours. Crystalline sample size was calculated by Scherrer method analysis of x-ray diffraction pattern.

$$D = \frac{K \lambda}{Br \, Cos\theta}$$

With Br, K, λ and D, respectively half peak width (FWHM) in radians, Scherrer constant (0.9), x-rays wavelength (1.5406 Å), and the crystal diameter (nm). From the XRD data using the equation obtained Scerrer rice husk ash particle size after ball mill for 15 hours at a rate of 450 rpm gained an average of 50.6 nm.

Table 3. Three Largest Difference Peaks of the XRD Results from Rice Husk Ash

Material	1 Theta (deg)	d (Å)	I/I1	FWHM(deg)	Intensity(Counts)
Rice Husk Ash Micro Size	21.8779	4.05928	100	0.26180	4784
	36.0582	2.48885	16	0.26360	771
	20.6000	4.30811	10	0.33480	500
Rice Husk Ash Nano Size	21.8748	4.05984	100	0.27420	4451
	36.0545	2.48910	16	0.28220	690
	31.3507	2.85100	8	0.30750	335

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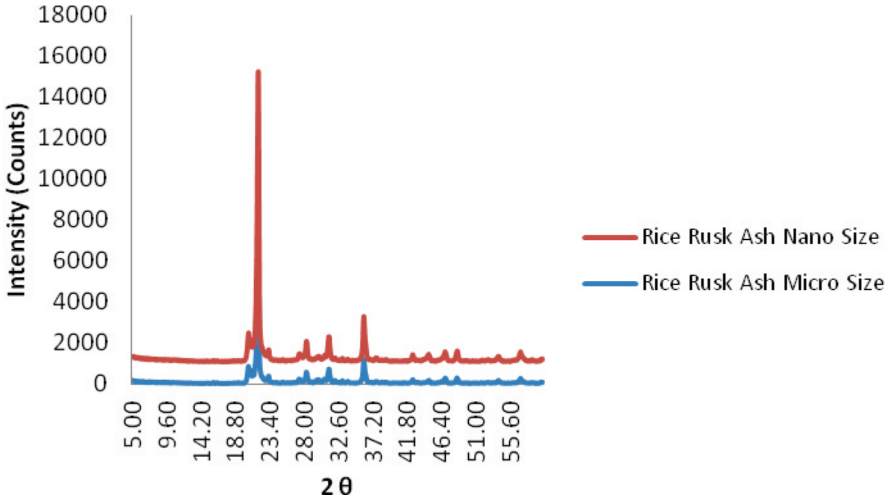


Figure 1. X-Ray Difraction Rice Husk Ash Micro and Nano Size

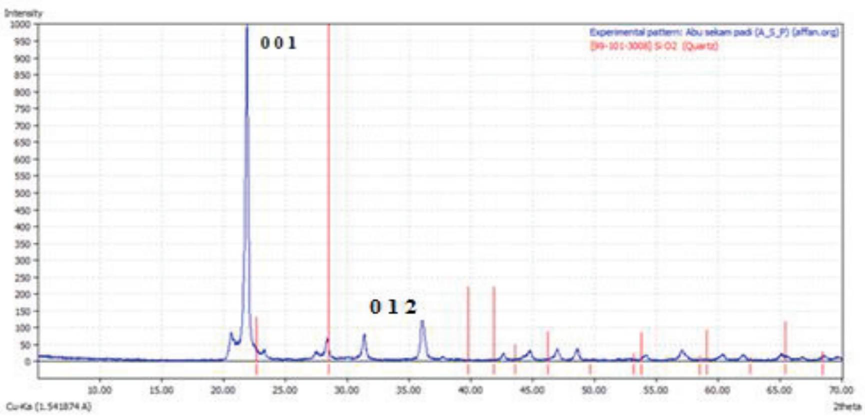
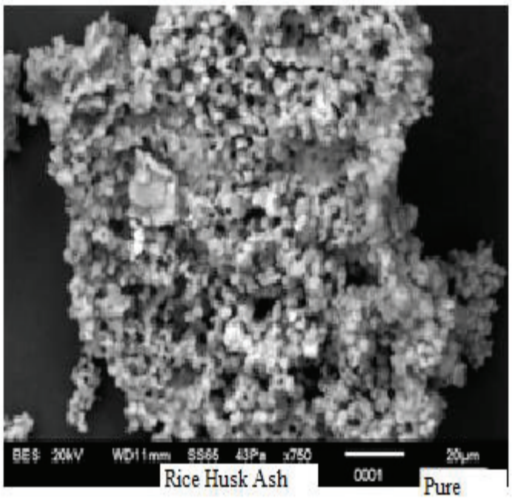


Figure 2. Diffraction patterns d_{hkl} Rice Husk Ash Nano

From the analysis of X-ray Diffraction nano rice husk ash obtained maximum peak d hkl 0 01 with d spacing 3.1274 Å while the d hkl 0 12 d spacing 2.1593 with densities 3.2500 g/cm³ type Quartz crystal with cell parameters a = 4.5350 Å c = 5.1700 Å system hexagonal crystal.

3.3 Analysis Morphology Rice Husk Ash



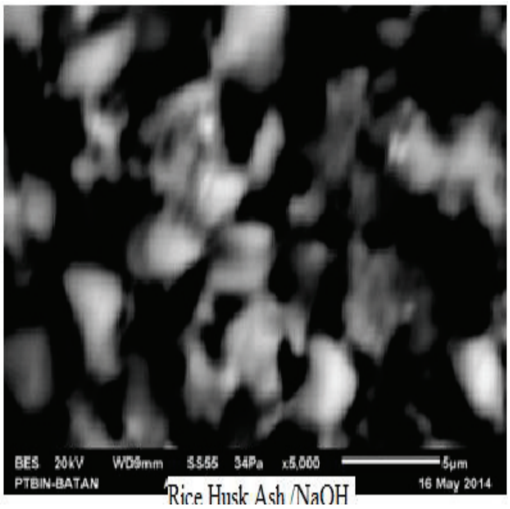


Figure 3. Morphology Rice Husk Ash

Figure 4. Morphology Rice Husk Ash Ball Mill 1 hours with NaOH

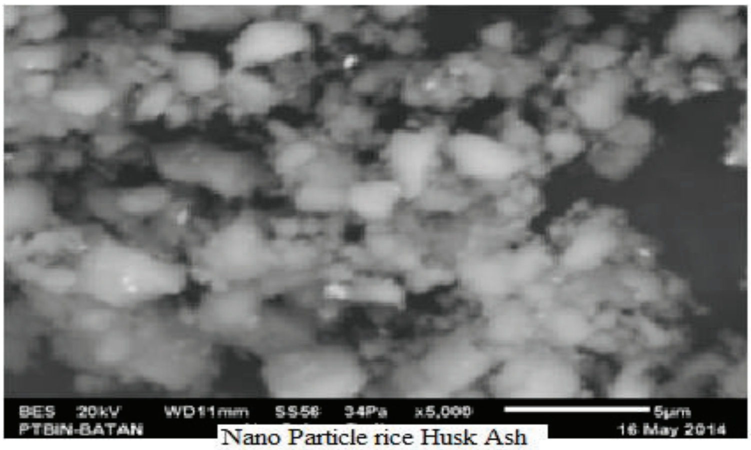


Figure 5. Morphology Rice Rusk Ash Nano Particle Ball Mill 15 hours with NaOH

3.4 Mechanical Characteristics.

In this study, mechanical properties of the samples include tensile strength, elongation at break, and Young's modulus, are measured in order to evaluate of nano Particle rice rusk ash Table 4. On Figure 6 until 8 shows the tensile strengths, elongation at break and Young's Modulus of the samples filled with nano particle rice rusk ash with PE-g-MA .

Table 4. Properties Mechanicals Composites HDPE /PE-g-MA with Filler Rice Husk Ash Nano

Material Blends	Tensile Strength (MPa)	Elongation at Break (mm)	Young's Modulus (MPa)
HDPE	23.54	221.25	547.80
HDPE / PE-g-MA/ Rice Husk Ash Nano 2% wt	27.62	394.46	514.30
HDPE / PE-g-MA/ Rice Husk Ash Nano 4% wt	25.62	312.39	518,29
HDPE / PE-g-MA/ Rice Husk Ash Nano 6% wt	21.86	99.11	510.54
HDPE / PE-g-MA/ Rice Husk Ash Nano 8% wt	21.70	120.60	513.48
HDPE / PE-g-MA/ Rice Husk Ash Nano 10% wt	22.22	195.31	540.81

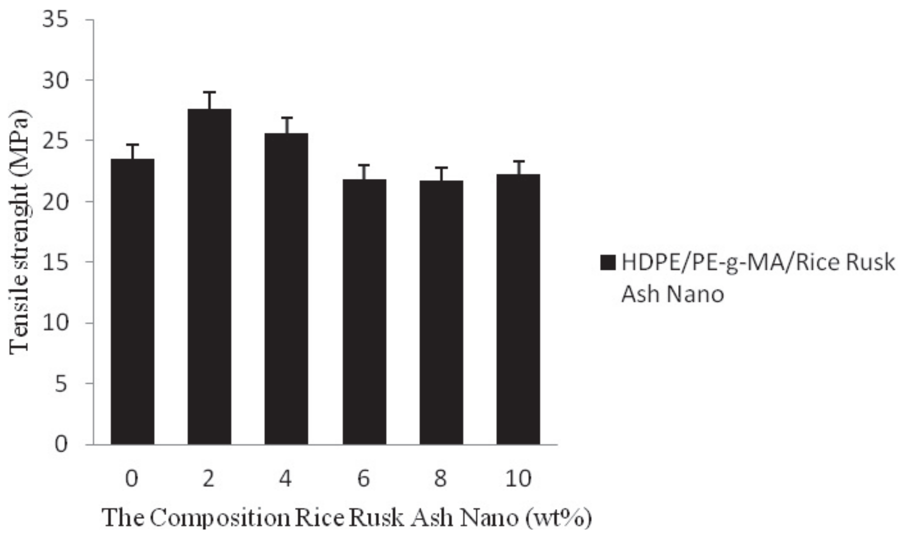


Figure 6. Tensile Strength of the Samples Filler Rice Rusk Ash Nano

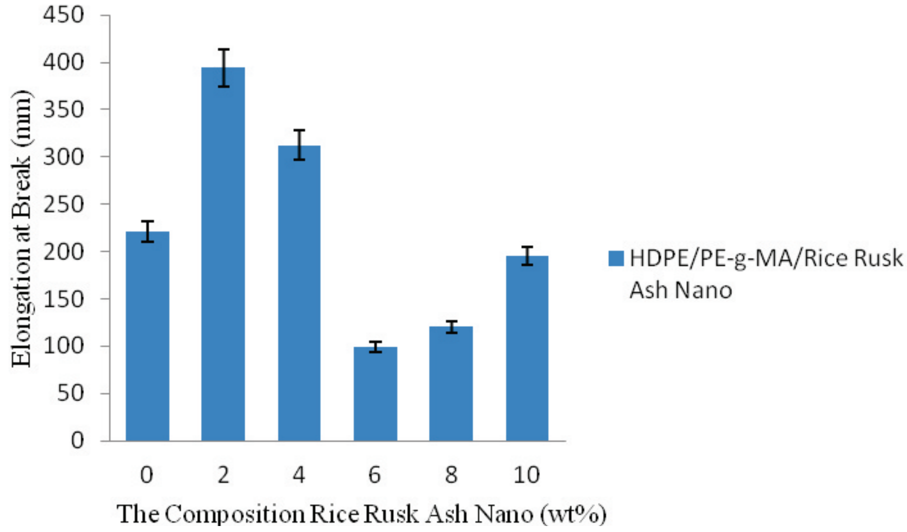


Figure 7. Elongation at Break of the Samples Filler Rice Rusk Ash Nano

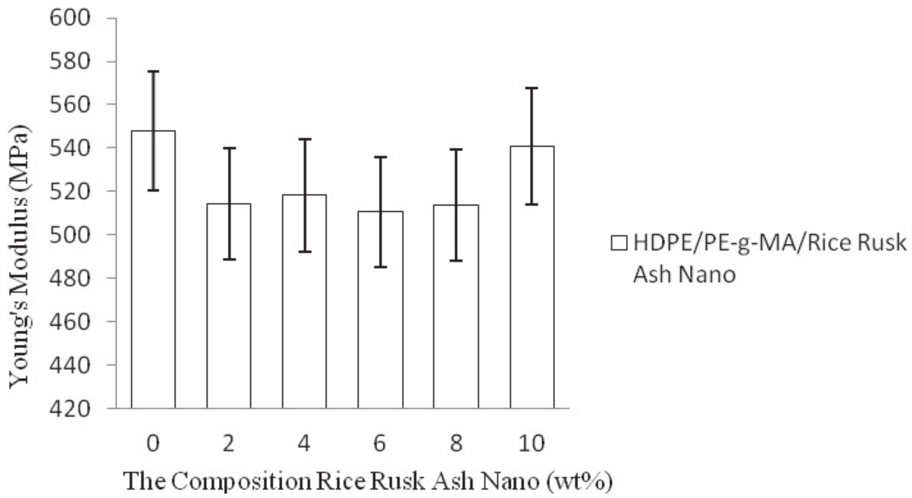


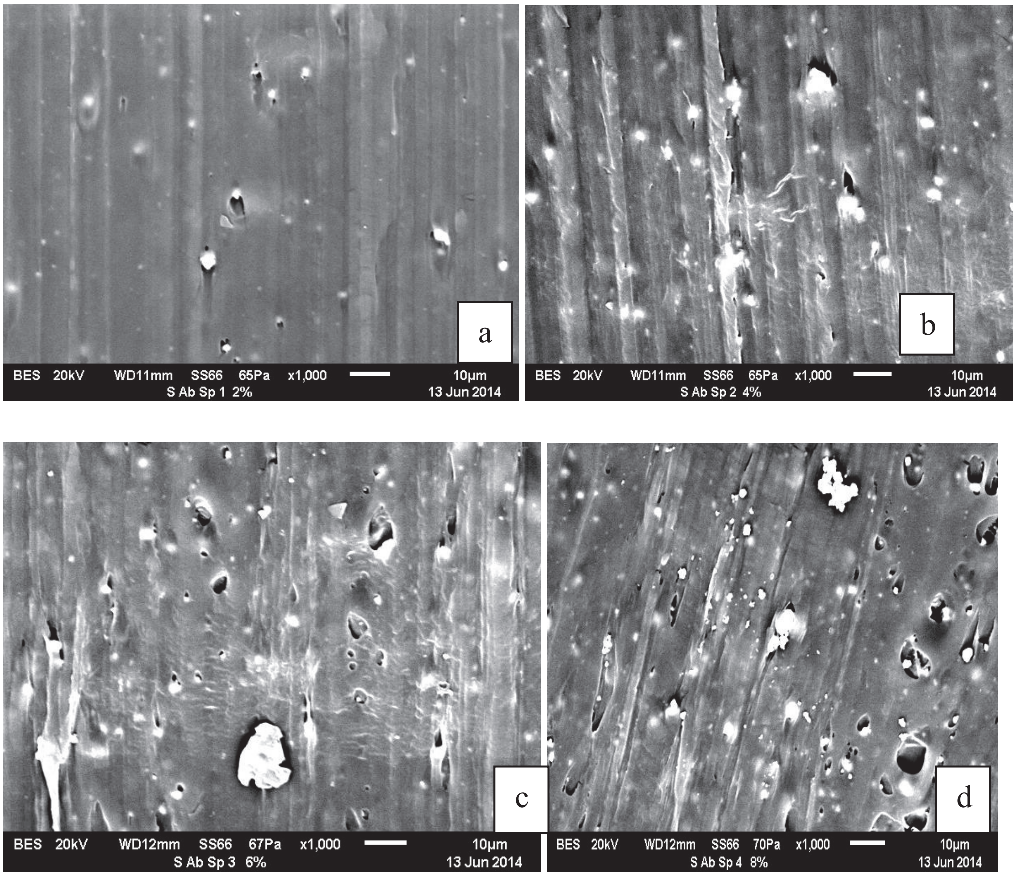
Figure 8. Young,s Modulus of the Samples Filler Rice Rusk Ash Nano

From the data on the mechanical properties of tensile strength increased compared to pure HDPE nano-particles in a mixture of rice husk ash with compatibilizer PE-g-MA on the composition of 2% to 4% and a decrease in maximum tensile strength at 6 to 10%, this is because clumping occurs at a certain composition of rice husk ash, thereby reducing the tensile strength of this can be seen from the results of morphological, as well as the elongation at break and Young's modulus decreased with increasing nano rice husk ash. The increase in tensile strength due to an increase in covalent bonding and hydrogen bonding with the OH group and the oxygen of each of the goup carboxil add bonding between the filler with the matrix which is in line with studies (Bhat, et. al, 2011) .Improved mechanical properties depend on many factors including the aspect ratio of the filler material, the degree disperse and orientation in the matrix, and adhesion at the interface matrix – filler.

The particle size is small filler increases the degree of polymer reinforcement versus large particle size (Leblanc, 2002). Particle size has a direct relationship to the surface area of the filler material. Thus, the small particle size provides a large surface area for interaction between the polymer matrix and filler so improve reinforcement of polymeric materials.

This is probably due to the silicate layer of rice husk ash can be dispersed nanometer-sized randomly and evenly providing exfoliation in nanocomposite structure. Silicate layers that exist on rice husk ash scattered individuals have extensive contacts a large surface that can bind strongly to the HDPE matrix which further gives effect to the increase in tensile strength. The incorporation of rice husk ash with nano compatibilizer PE-g-MA is more than 6% wt contrary negative effects which would lower the tensile strength. This is probably due to the decrease in the degree of spread of exfoliation of silicate layers in the nanocomposite rice husk ash with nano-particle content of rice husk ash high (> 6 wt%). In addition, the nano-particle agglomeration rice husk ash as shown in the SEM image also led to a decrease in tensile strength. Rice husk ash agglomeration is believed to be a stress concentration and the beginning of the crack so that the power will go down. The same thing from the study (Kusmono, et. al, 2010).

This is due to the increasing number of content of silica resulted in a drop in tensile strength, which is in line with the research, (Koo, et. al, 2002; Wu, et. al, 2007; Lei, et. al, 2007; Kord, et. al, 2010; Samal, et. al 2008). Figure 9 morphology of HDPE with a mixture of rice husk ash nano on the composition of 2 to 10% by weight, the greater the visible content of silica, there will be algomerat which in turn reduces the tensile strength and elongation at breaks.



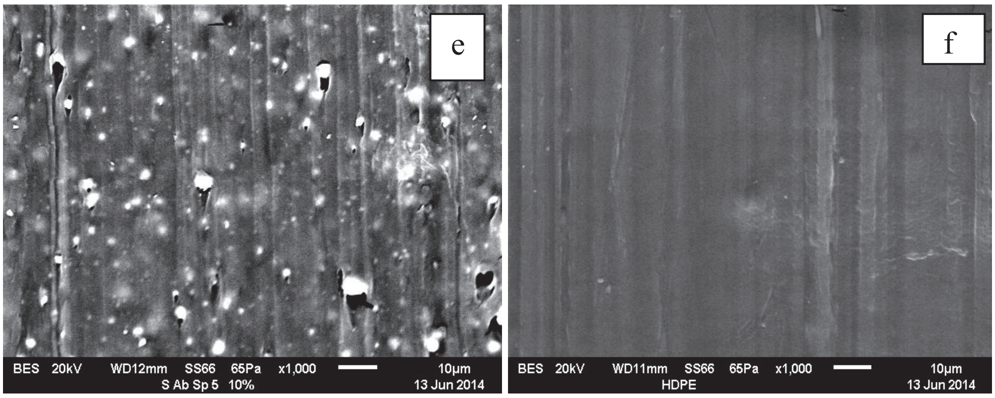


Figure 9. Morphology HDPE/PE-g- MA/Rice Rusk Ash Nano Blends (a) (2 wt%); (b) (4 wt%); (c) (6 wt); (d) (8 wt%); (e) (10 wt%); (f) (HDPE)

4. Conclusion

The results obtained rice husk ash silica composition of 89.6 wt%, the particle size of 50.6 nm, analysis of X-ray Diffraction nano rice husk ash obtained densities 3.2500 g/cm³ type Quartz crystal with cell parameters a = 4.5350 Å c = 5.1700 Å system hexagonal crystal. The results of the mechanical properties of tensile strength and elongation at break of an increase in the composition of the mixture 2 to 4% by weight.

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