Preparation Natural Bentonite in Nano Particle Material as Filler Nanocomposite High Density Poliethylene (Hdpe)

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Abstract

The purpose of this study is the preparation of natural bentonite in nano particle material as filler and reinforcement of thermoplastic high density polyethylene (HDPE) that has mechanical properties and good thermal properties and can be used as one component in the automotive industry. The method is performed in the preparation of nano particles is done by purification with HCl solution, calcination at a temperature of 600 ^{0}C for 2 hours and the ball mill for 10 hours to get natural bentonite nanometers in size and materials used as a filler thermoplastic HDPE. The results particle size analyzer (PSA) average diameter of the particles 97.5 nm, with the X-rays diffraction (XRD) obtained by means of an average particle size of 49.80 nm and the highest content of EDX analysis on natural bentonite is aluminum (Al) and silicon (Si). with the X-rays diffraction analysis obtained from natural Bentonite is a kind of aluminum silicate mineral and classified types of wyoming (Na-bentonite). The minerals present in the clay consists of a group of minerals crystoballite quartz, mica group minerals from the annite, analcime from feldspar group minerals, and mineral carnegieite of feldspathoid group. The biggest content of samples of clay minerals crystoballite this phase is about 68%, while the result of nano composite (HDPE blend natural bentonite nano particle) with XRD analysis occurs intercalation between nano bentonite with HDPE, thermal analysis were shown enthalpy increased with increasing nano composites bentonite particles, the results of the mechanical properties of nano composite in an increased of the maximum tensile strength on quantity of 2 to 6 wt%, and a decline in the larger quantity of 6 % wt and a decrease in elongation at break with the addition of nano-particles of natural bentonite from SEM analysis HDPE blend with nano bentonite is homogeneous.

Keywords: Nanoparticles, Natural Bentonite, HDPE, Mechanic, Thermal

1. INTRODUCTION

Bentonite is a natural resource that is quite a lot in Indonesia, such as in Java, Sumatra, and Sulawesi, but has not been utilized. In North Sumatra natural bentonite can be found in the District of North Tapanuli Pahae. Bentonite has hydrophilic properties, so that the material is generally not compatible with the majority of polymeric materials, therefore, must be chemically modified to create a more hidrofobis surface, it is necessary for a material with the polymer matrix compatibel [1].

Polymer composite is a composite consisting of a matrix (matrix is the base material forming composite filler with no binding occurs chemically bonds) form of the polymer and the filler material of various types of composite materials that have the properties of the alloy constituent material properties [2]. On the manufacture of polymer composite products will experience reduced physical properties of the polymer matrix, due to the high temperatures in the manufacturing process so that the use of durable materials can not. Besides, the filler will provide varied nature of the material properties in order to obtain the physical and mechanical properties that meet the requirements. [2]

Bentonite is a type of rock that contains a lot of minerals in it that has the properties of a typical montmorillonite namely; may inflate in water, intercalation and ion exchangers are making this material interesting organo clay used to be a catalyst nano and nano clay polymer composites [3].

Until now user an bentonite is not optimal, in the field of engineering technology a material (composite). Bentonite One application is currently under review by many national and international research institutions is its use as a filler material are nano-sized, more known as nano filler. Nano fillers can be applied to the polymer material to produce nano composite material with improved some basic properties of polymers, such as thermal resistance properties, mechanical properties, chemical resistance and flammability.

Problems that are often faced is the lack of elastic properties in the resulting polymer composites due to the addition of fillers. This influence can be on the look with the addition of fillers to a minimum, to produce a composite nanostructures. In general, the addition of natural bentonite into the polymer is highly dependent on the strength of the interaction between the polymer and the filler material will produce nanoscale properties of composites, such as: intercalated, nano composites, nanocomposite and axfloliated flokulated nonocomposit. Observations-fundamentally previous studies involving bentonite as filler as has been done by Rihayat [4]. The fillers are often added to polymeric materials that are capable of homogeneously into the matrix. Homogeneous with respect to the above properties, polymers derived from organic materials with filler derived from inorganic materials not converge uniformly this is caused by differences in surface energies of the two materials. To address these problems, the modified filler material [5]. One of the modified bentonite is to use a different surfactant with cation exchange method.

Results of some studies suggest that the inn Properties of materials will be compatible with the polymer matrix is influenced by several factors namely: a filler particle size, wherein the particle size of a small filler material that can increase the degree of polymer reinforcement compared to the larger size [6], as well as the smaller the particle size the higher the bond between the filler's with the polymer matrix, the number of vast surfaces of the surfaces of the filler with the polymer matrix [7], 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 natural zeolite increased nano mechanical properties of nanocomposite. [8]

2. METHODS

Materials.

The used materials is: Natural Bentonite of North Tapanuli Pahae District, North Sumatra: Indonesia, HCl solution, Aquades, thermoplastic HDPE, PE-g-MA compatibelizer.

Tools.

The tools used in the study are: Mortar muller, Magnet, magnetic stirrer, planetary ball mill P 200, Particle Size Analyzer™ Desla nano (PSA), XRD, SEM, Internal mixer laboplastomil Model 30 R150, Hot Press and Cold Press, dumbell samples cut machine, Universal Testing Machine, DSC.

2.1 Purification Process Development and Nano Particle Natural Bentonite.

At this stage, natural bentonite taken in the form of large chunks of the size, will be made in the nanometer size, then the process is carried out with the following steps; solve large chunks with a hammer and then crushed with a mortar muller use it again until smooth shape with size 74 micrometers (200 mesh). In the process of purification of materials impurities that exist in the natural bentonite content of the activation process is carried out with the steps as follows: to eliminate the impurity levels of Fe used magnets, to remove impurities while Al was a chemical process using HCL solution with 2 M levels. The HCl solution in bentonite into the mix in a container with a ratio of Bentonite: HCl, 1:10, then until homogeneous by using a magnetic stirrer for 2 hours, after which separates the bentonite HCl solution by using filter paper and then do the washing again using with distilled water and re-separate the bentonite with Aquades until a neutral of pH. Bentonite natural distillate that is purified, dried in the sun then calcination at a temperature of 600^0 C ,during 2 hours. Calcination of purified bentonite and incorporated into (PM 200) planetary ball mill for 10 hours to obtain a natural bentonite nanometers in size. Result of natural bentonite nano particles that have been purified and then characterized by SEM morphological analysis, as well as the analysis of chemical constituents contained in natural bentonite nano particles by means of EDX, XRD analysis of the crystal structure and to analyze the particle size of bentonite used nano particle size analyzer (PSA).

2.2 Preparation Nano Composite

The result of natural bentonite is used as a nano-size fillers in thermoplastics HDPE and PE-g-MA compatibilizer, included in the internal mixer with a temperature of 150 ^{0} C at a rate of 60 rpm for 10 minutes, compatibilizer quantity of PE-g-MA 3 wt% ,nano bentonite particles in quantity (0,2,4,6,8, and 10) wt % shown in Table 1. Then, using hot press molding and cold press for 10 minutes and obtained a prototype in thermoplastic sheet specimens for each sample testing based ASTM and JIS standard size for a 6781 K dumbell tensile strength

Results of morphological analysis of nano-composites were characterized with SEM, as well as, the analysis of crystal structure by XRD and mechanical properties and thermal properties (DSC)

Table 1. Material Quantity Blend In Internal Mixer

Material	Mixture Quantity (wt%)						
	S1	**S2**	**S3**	**S4**	**S5**	**S6**	**S7**
HDPE	100	97	95	93	91	89	87
PE-g-MA	0	3	3	3	3	3	3
Natural Bentonite Nano	0	0	2	4	6	8	10

3. RESULTS AND DISCUSSION

3.1. Analysis Morphology of Nano Particle Natural Bentonite.

In Figure 1. Shown morphological observations of natural bentonite sample surface, was shaped like alumenium silicate mineral nanoscale surface morphology of bentonite particles with a magnification of 15,000 and 20,000. Figure 1. This can be seen bentonite sample consists of various phases and distribution and have a variety of color contrast too. Aluminum silicate mineral has a majority element content of aluminum (Al) and silicon (Si). This is confirmed by the EDX results showed that the energy spectrum that arises is Sodium (Na), Alumenium (Al), Silicon (Si), and others.

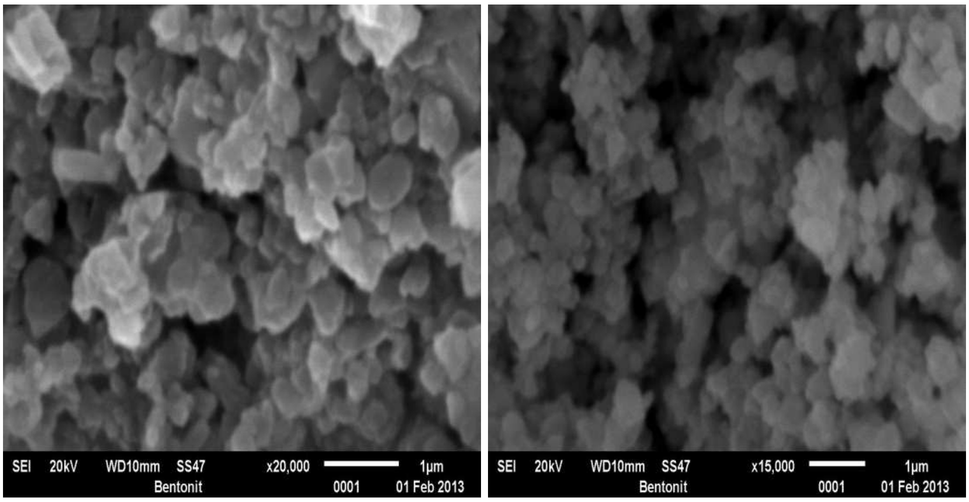


Figure 1. Microstructure Nano Particle Bentonite.

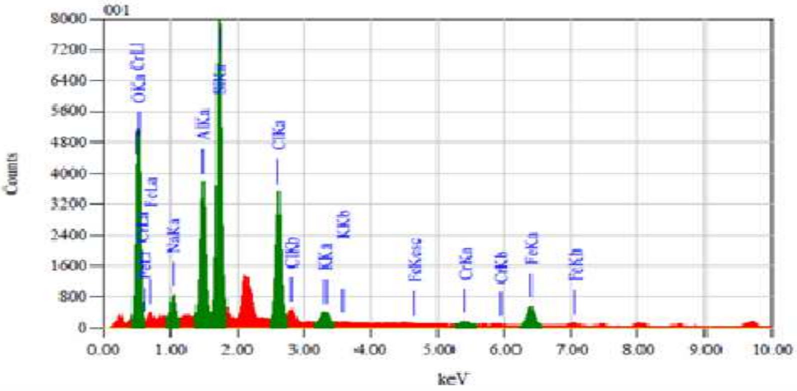


Figure 2. Analysis Elements With Energy Dispersive Spectroscopy

3.2 Analysis of Energy Dispersive Spectroscopy (EDS)

In Figure 2. Shown the results of elemental analysis using energy dispersive spectroscopy on samples of nano bentonite. Energy spectrum show that element that appears is sodium (Na), aluminum (Al), silicon (Si), Samples Nano Bentonite chlorine (Cl), potassium (K), chromium (Cr), iron (Fe), and oxygen (O) are respectively 1.041 keV energy; 1.486 keV, 1.739 keV; 2.621 keV, 3.312 keV; 5.411 keV; 6.398 keV and 0.525 keV. In detail, the content of elements in the nano particles of bentonite are shown in Table 2.

From Table 2 that the sample has the highest content of aluminum (Al) and silicon (Si). While the presence of chlorine (Cl) in the bentonite sample because the sample has to be within the chlor acid (HCl). Thus it seems clear that this is a class of bentonite samples of aluminum silicate minerals.

Table 2. Results Of Elementary Analysis Using Energy Dispersive Spectroscopy

No..	Element	(wt %)
1.	Sodium (Na)	2.81 ± 12:16
2.	Aluminum (Al)	9:37 ± 0:12
3.	Silicon (Si)	24.28 ± 12:13
4.	Chlorine (Cl)	11:55 ± 12:12
5.	Potassium (K)	1:08 ± 0:12
6.	Chromium (Cr)	0.73 ± 12:16
7.	Iron (Fe)	5.92 ± 12:29
8.	Oxygen (O)	44.26 ± 0.23

3.3 Analysis Of Nano Particle Bentonite With XRD.

In Figure 3. Shown the results of measurements of X-ray diffraction (XRD) bentonite. Size crystallites was calculated based on analysis of samples scerrer method of X-ray diffraction pattern using the formulation:

$$D = \frac{k\gamma}{B\cos\theta}$$

Where as B, K, λ, and D, respectively half-peak width (FWHM) in radians, Scherrer constant (0.9), x-ray wavelength (1.5406 Å), and the diameter of the crystallites (nm). Based on the calculation of average crystallite size obtained rat that he meter bentonite sample crystallite size of 49.80 nm.

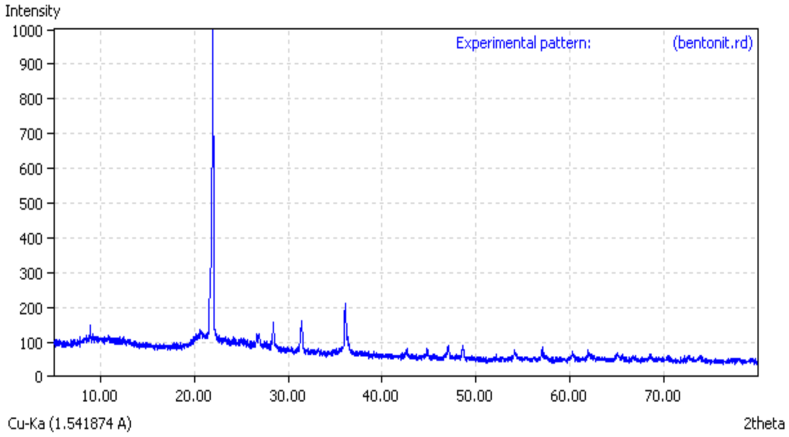


Figure 3. Identification Phase of The Diffraction Pattern X-Ray Nano-Bentonite

In Figure 4 are shown the results of the identification of X-ray diffraction pattern of the sample of bentonite. Phase identification that appears on bentonite samples suspected crystoballite phase (SiO_2), phase annite ($Fe_3Al_{3,448}K_{0.99}Na_{12:01}O_{12}Si_{2552}$), analcime phase ($Al_4Na_{1.806}H_{1.71}O_{14}Si_{4,194}$), and carnegieite ($AlNaO_4Si$), which respectively refer to the results of research Pluth [9], Redhammer [10], Gatta [11] and Barth [12].

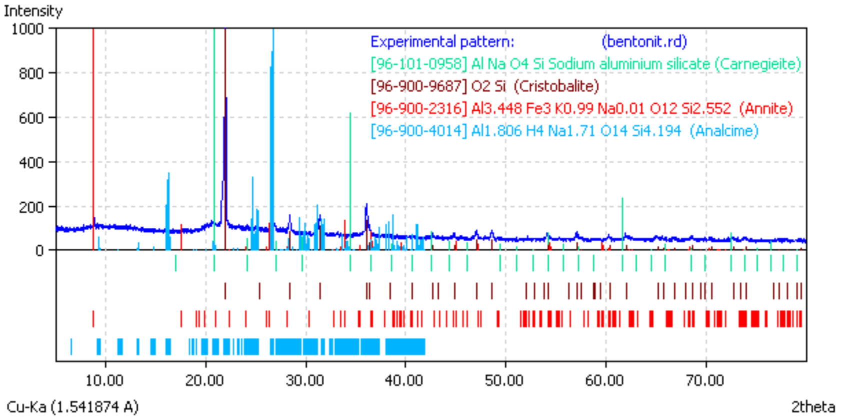


Figure 4. Identify The Phase Of The X-Ray Diffraction Patterns Of Samples Of Bentonite

Thus further analysis is needed to be able to show that the samples have four mineral phase. Bentonite is an aluminum silicate mineral species and classified types of wyoming (Na-bentonite). Minerals in the bentonite consists of a group of minerals crystoballite of quartz, mica group minerals from the annite, feldspar mineral analcime of the group, and a group of minerals carnegieite feldspathoid. Contens greatest of these is bentonite sample crystoballite mineral phase around 68%.

3.4 Size Analysis Of Natural Nano Bentonite With Particle Size Analyzer

Figure 5 sample of particle diameter measurements using particle size analyzer (PSA). Based on the results of measurements of particle diameter using a particle size analyzer and particle morphology using a scanning electron microscope shows that the magnitude of the average diameter of the particles of bentonite sample is 97.5 nm. While the particle size distribution from the smallest to largest size is 74 to 117 nm.

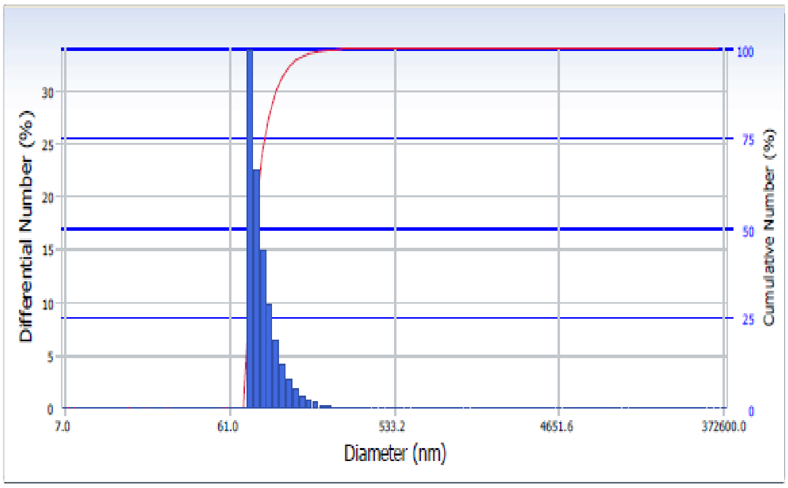


Figure 5. Nano Size Natural Bentonite Use Particle Size Analyzer (PSA)

3.5 Mechanical Analysis Of Nano Composite Bentonite

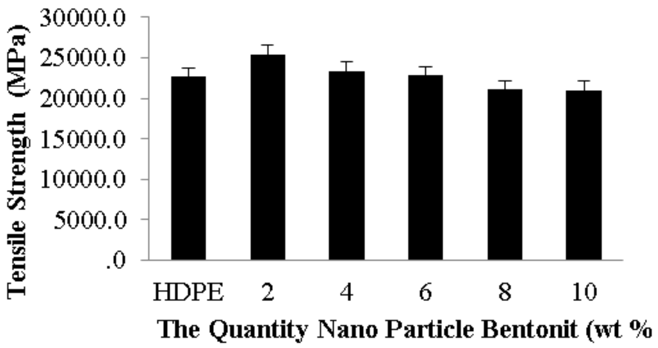


Figure 6. Tensile Strength of the Samples Filler with Nano Particle Bentonite

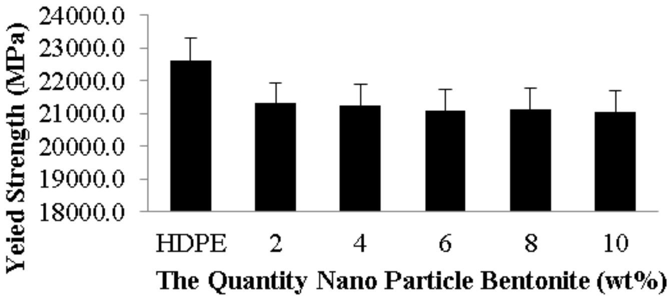


Figure 7. Yield Strength of the Samples Filler with Nano Particle Bentonite

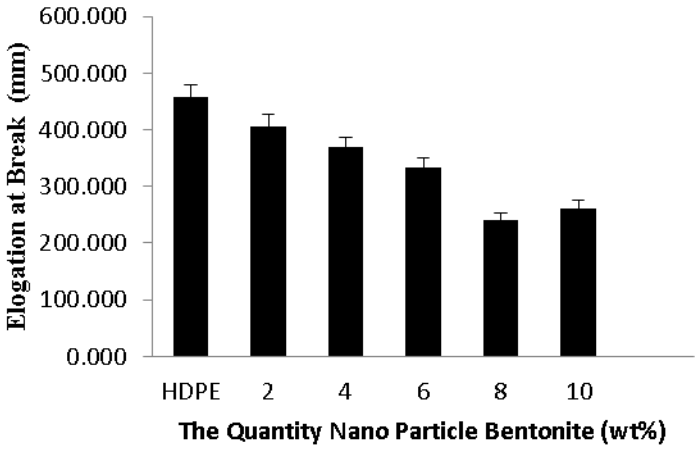


Figure 8. Elongation at Break of the Samples Filler with Nano Particle Bentonite

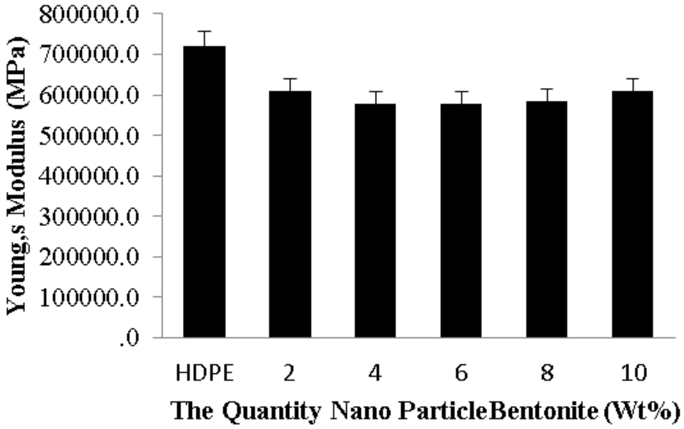


Figure 9. Young's Modulus of the Samples Filler with Nano Particle Bentonite

From the data on the mechanical properties of tensile strength increased in HDPE with nano-bentonite mixture 2% to 6% and a decrease in the maximum tensile strength of nano particle natural bentonite 8 to 10%, this was due to the specific quantity of natural bentonite clumping occurs thereby reducing the tensile strength, while for yield Strenght relatively the same with the addition of natural bentonite, as well as with elogation at break and Young's modulus decreased with increasing natural bentonite nano material particle size small increase compared to the size of the polymer reinforcement large part. Particle size has a relationship directly to the surface area per gram of filler material. Therefore, small particle size provides a large surface area for interaction between the polymer matrix and filler material so improve mastery tan emakin polimer. Small particle size the higher the interaction between the matrix material and the in Polimer. Results research which states that the surface area can be increased with the shaft surface or cavity on the surface of the filler [7]. It is possible that polymers can penetrate into the surface of the shaft when the mixing process. Homogeneously dispersed particles increased interaction through polymer adsorption on the surface of the filler material. Instead, the particles are not homogeneously dispersed agglomerates or clumping may result in the matrix agglomerates polimer. Presence interactions between the filler and matrix and the resulting decline in the physical properties of polymer materials.

From Figure 6 tensile strength obtained for nano-particle nature of purified bentonite largest obtained in a mixture of 2% wt quantity that is 25.377 MPa and 4% wt of 23.296 MPa, whereas, for without the use of nano particles of natural bentonite tensile strength acquired by a large 22.62 MPa. Tensile strength is better to use a mixture of natural bentonite nano particles carried by the calcination process. This is because the silicate layers in nanometer-sized natural bentonite can be spread evenly and randomly providing exfoliation in nanocomposite structure. Silicate layers that exist in the bentonite are widely dispersed individuals have a large contact surface that can bind strongly to the matrix HDPE and PE-g-MA compatibilizer which further gives effect to the increase in tensile strength. Incorporation of natural bentonite nano more than 8% wt opposite negative effects that lower tensile strength but greater if no nano bentonite fillers. This is due to the decrease in the degree of spread of exfoliation of silicate layers in the natural bentonite content of nano-particle nanocomposite with high natural bentonite (> 6 wt %). In addition, the nano-particle agglomeration or natural bentonite as shown in the microstructure also cause a decrease in tensile strength. Agglomeration bentonite is a natural place to start stress concentration and cracking so strengh will go down. This is in accordance with the study of more than 4 wt % clay just the opposite which is to reduce the negative effects strenght drag, caused by a decrease in the degree of spread of exfoliation of silicate layers of clay in nano composites [13]. With adition compatibilizer material will form the esterification reaction or hydrogen bonding at interfaces of existing hydroxyl groups on the particle on one side and the carboxylic group compatibilizer which diffuses into the polymer matrix on the other [14].

3.6 Thermal Analysis Of Nano Composite

From the differential scanning calorimetry (DSC) analysis can be used to determine the enthalpy by measuring the differential heat flow required keeping reference samples and inert materials at the same temperature. This temperature is usually programmed to scan various temperatures with increased linearly at a rate that has.

Information which can obtained at semi-crystalline crystallinity of the polymer material is content. Both mechanical properties, physical and chemical results are important, it depends on quantity blend and conditions crystallization such as temperature, pressure, weight, orientation, molecular, and thinners [14]. Temperature melt constitute important parameter for thermoplastics because represent temperature minimum required for processing polymer. Behavior melt highly dependent on structure chemistry of materials along with size and regularity cristality found in crystalline phase [16]. Temperature crystallization can obtained from DSC, refrigeration of temperature melt scan above ambient conditions. Crystallization temperature polymer information can characterization more informative. This is due to sensitivity inherent higher than this test, to detect subtle differences in make-up of polymer given [16].
From Figure 10, a decrease in the enthalpy ΔH with the addition of nano-particles with a good natural bentonite PE-g-MA compatibilizer. However, the melting temperature of nano composites of HDPE pure after combined by the number of certain natural bentonite nano particles increase not sinifikan at 130 ^{0} C range where the melting point of HDPE in the range (125-130) ^{0}C. complete data are presented in Table 3.

Table 3. Thermal Analysis of Thermopalstic HDPE with Nano Particle Bentonite Blend

Nano Bentonite (wt%)	Melting Temperature (^{0} C)	Area (mJ)	ΔH (J / g)
0	130.16	526.232	150.324
2	130.03	592.133	155.824
4	130.37	558.323	146.927
6	129.15	508.390	137.402
8	129.44	484.674	131.047
10	130.88	502.817	128.927

This suggests that crystal in HDPE nano composites thicker and more perfect of the pure HDPE. The results consistent the study [17]. contrary, incorporation of the copolymer, the melting endothermic region is calculated and reported as the heat of fusion ΔH. Where ΔH is an important parameter because it is directly proportional to the amount of the overall level of which is owned by the polymer. Then, enthalpy can be estimated from ΔH [16]. In a HDPE interfering with the incorporation of nano particles of natural bentonite 2 wt% to 6 wt% with HDPE has caused the value of the tensile strength of HDPE increased. This means that the presence of a small amount of natural bentonite nano particles in polypropylene PP nucleation increases it. These results are consistent with the results of recent studies of crystallization behavior and morphology of pure polypropylene/clay nano composites [17]. Mineral filler very likely affect semicristal polymer crystallization process quantitatively. Similar increase in crystallinity has been reported not only for PP/organo-clay nano-composites, but also for other polymer/organo-clay nano-composites such as nano composites of polyethylene [19].

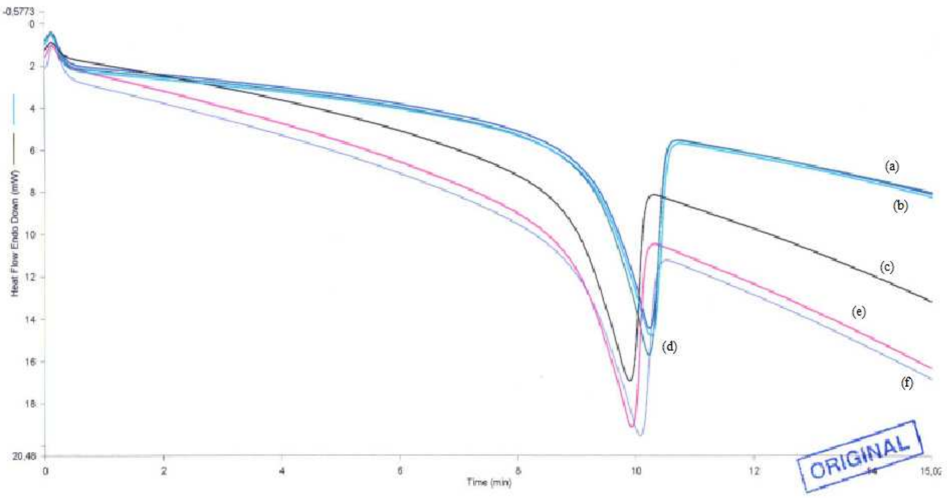
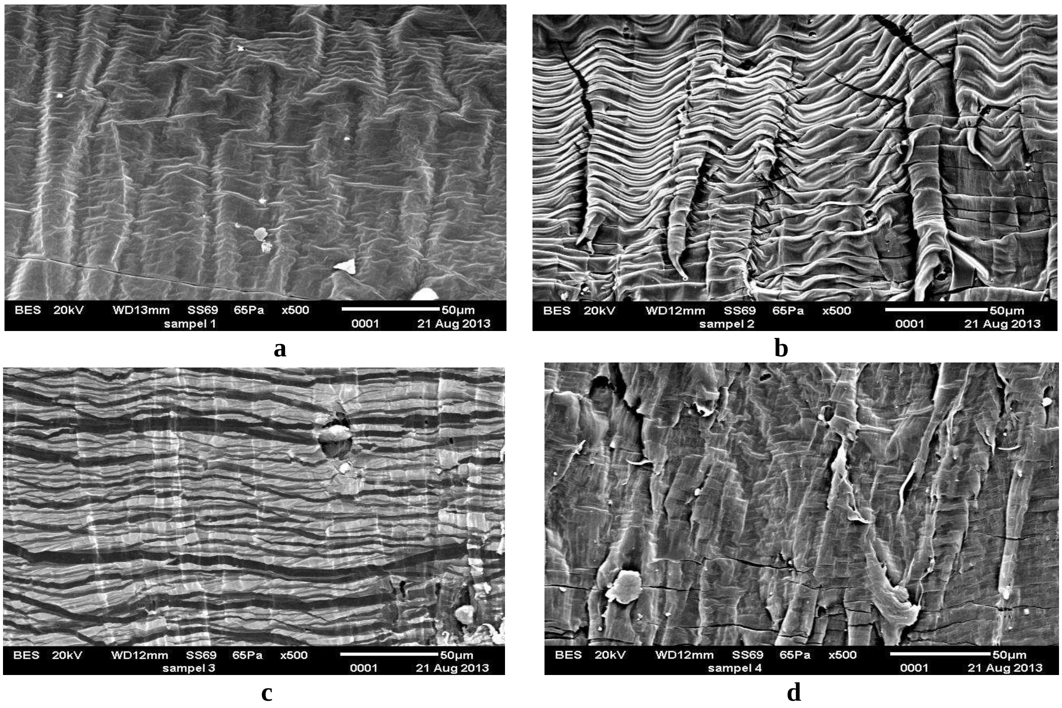


Figure 10 a. DSC thermogram of HDPE Thermoplastic; DSC thermogram of HDPE Thermoplastic with (b. 2 , c. 4, d. 6, e.8, f. 10) wt % Nano Bentonite Blend

3.7 Microstructure Analysis of Nano Composite

From the results of microstructure analysis can be seen that with the addition of nano particle bentonite (2, 4, 6) wt % may be more homogeneous it is in accordance with the mechanical properties are obtained, which is larger tensile strength, whereas the addition of bentonite nano 8% and 10% by weight, of Figure 11, e and f agglomeration can occur s lead to decreased mechanical properties.



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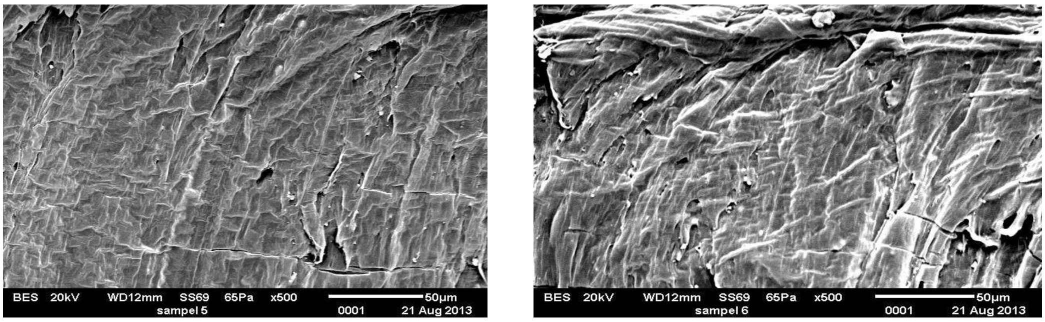

e **f**

Figure 11. a. Microstructure of HDPE Thermoplastic; Microstructure of HDPE Thermoplastic with (b. 2, c. 4, d. 6, e.8, f. 10) wt % Nano Bentonite Blend

4. CONCLUSION

This research indicates that natural bentonite obtained nano as from mineral aluminum silicate and classified types of wyoming (Na-bentonite). The average diameter of the particles using particle size analyzer (PSA) obtained 97.5 nm. which can be used as filler material in nanocomposites. Natural bentonite nano particle mineral phase crystoballite is approximately 68 %. From the test results mechanic, DSC and SEM nano composite obtained that the quatity of bentonite (2 - 6)wt% fallow obtained mechanical properties, thermal properties, and morphology of the good that can be used as a filler in nanocomposites.

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