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# Preparation and Characterization of Nano Composites HDPE Blend with Rice Husk Ash Nanoparticles

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**Abstract** : The purpose of this research is to know the nano characteristic of high density polyethylene (HDPE) thermoplastic composite. with nanoparticle ash husk filler (RHA) particle size of 52.22 nm with compatible and non-compatible Polyethylene-grafted maleic anhydride (PE-g-MA) compatibilizers .The composite nano-making process was carried out in an internal mixer of laboplastomill, RHA nano with composition (0,2,4,6,8, and 10)% by weight. From the composite nano result analyzed the mechanical properties, Thermal, morphology and diffraction pattern. The results of tensile strength, elongation at break and elasticity modulus analysis increased with increasing RHA nano composition of elongation and elastic modulus, the results of mixed nano composite HDPE nano analysis with RHA were uniform and homogeneous. Similarly, XRD analysis results in the intercalation of HDPE thermoplastics with RHA nano filler has good thermal stability properties.

Keywords: RHA, HDPE PE-g-MA, Mechanical properties of Thermal morphology, XRD.

# Introduction

Rice husks are an abundant by-product of rice mills, and have been used only for fuel for burning red bricks, burning for cooking or simply throwing away. Inadequate rice husk handling will cause environmental pollution. Waste is often defined as waste material / waste material from agricultural processing. Nano silica has now been applied in various fields including science and industry. RHA materials that have a silica content have been widely used as fillers. Silica has been widely used as a catalyst, and various types of organic-inorganic composite materials [1]. In addition to the processed products, silica has also been used directly for oil purification, as an additive in pharmaceutical products and detergents, as a stationary phase in chromatography columns, polymer fillers and as adsorbents [1,2].

Nano silica has now been applied in various fields including science and industry. RHA has been widely used as a filler. Silica has been widely used as a catalyst, and various types of organic-inorganic composite materials [2]. There has been much research on the making of silica nano from RHA by synthesis, among others, [3,4,5,6].

RHA natural materials generally have hydrophilic properties, they are generally not compatible with most polymeric materials, and therefore chemically have to be modified to make their surfaces more hydrophobic with polymeric material for which an ingredient is compatible with the polymer matrix.

The use of a silica coating on the composite may improve the properties of the material (cationic capacity change, high permissive area, large comparative aspect), [7,8].

Rice husk ash containing> 90% silica is widely used as adsorbent, ash, filler, and auxiliary materials in the manufacture of building materials such as cement and concrete [9,10,11]. The organic part of the rice husk can be further processed to produce chemicals such as xylose, furfural, xylitol, ethanol, acetic acid, lignosulfonic acid [10], organic substances in rice husks such as minerals in small quantities can be removed by treatment with acids using  $H_2SO_4$ , HCl, or HNO [12]. According to[11], hydrochloric acid is a highly effective chemical to reduce impurities contained in rice husks

It can essentially improve physical and mechanical properties, such as tensile strength, tensile modulus, flexural strength, heat stability, thermal properties, for some thermoplastic materials and thermoset nanocomposites on a relatively small amount of silica fillers [13,14,15].

The purpose of this study was to investigate the mechanical, thermal, morphological and nanocomposite nano-mixed structure of Nano ASP and HDPE thermoplastics with PE-g-MA and PE-g-MA compatibles.

## Experimental

#### **RHA Nano Particle Preparation**

RHA white from burning result from rice plant in the form of powder included by tool of planetary ball mill PM 200 type retsch for 1 hour at speed 250 rpm. then the results of the ball mill were sieved using a 200 mesh sieve (74  $\mu$ m).

The 74  $\mu$ m RHA was dissolved in 2M HCl (Merck KgaA 64271 Darmstadt Germany) as much as 40 ml and then stirred for 40 minutes at 70<sup>o</sup>C using a magnetic stirrer. After the solution is formed filtering with filter paper. Further dissolved using 2.5 M NaOH and stirred using a magnetic stirrer. The mixture of rice husk ash with NaOH solution (production of Merck KgaA 64271 Darmstadt Germany) is separated by filter paper and then washed with aquades and filtered back to separate aquades with rice husk ash Then dried at oven with 70<sup>o</sup>C for 4 hours. RHA yield of 10 g dissolved in HCL 2 M and filtered using filter paper. Then the solid-shaped PEG-6000 is heated and melted at 50<sup>o</sup>C for 15 minutes. The melted PEG-6000 is added to the solution at a ratio of 1: 3 then stirred using a magnetic stirer at 70 ° C for 40 minutes. Then 2.5 M NaOH is added to the PEG-6000 mixture with a rice husk ash solution while stirring using a magnetic stirer.Furthermore the solution is separated by filter paper and washed with aquades and filtered back to separate addes and filtered back to separate. Then 2.5 M NaOH is added to the PEG-6000 mixture with a rice husk ash solution while stirring using a magnetic stirer.Furthermore the solution is separated by filter paper and washed with aquades and filtered back to separate ash rice husk with aquades. Then dried in oven at 70<sup>o</sup>C for 2 hours.

# **Nanocomposites Preparation**

The production of Nanocomposite is done by mixing HDPE thermoplastic production of PT Titan Petrokimia Nusantara Indonesia with ASP or RHA nanoparticles (Rice Husk Ask preparation with size 52,22 nm) mixing this by using internal mixer laboplastomil with 60 cc chamber volume with 70% with 60 g, mixed temperature at 150 ° C with a rotor speed of 60 rpm for 10 min with mixed variation of Nano ASP (0.2,4,6,8,10)% by weight to thermoplastic HDPE and compatible material (Polyethylene-grafted maleic Anhydride (Sigma Aldrich USA) 2% by weight and without (PE-g.MA), in Tables 1 and 2 showing the composition of the mixture. The nanocomposite results were characterized by mechanical properties, tested by a R-1 storm tensile test to obtain maximum tensile strength, elongation of breaks, and elastic modulus. The test was performed using JIS K 8671 standard

Materials	Blend Composition (%) wt							
	S <sub>0A</sub>	S <sub>1A</sub>	S <sub>2A</sub>	S <sub>3A</sub>	S <sub>4 A</sub>	S <sub>5A</sub>		
HDPE	100	96	94	92	90	88		
PE-g-MA	0	2	2	2	2	2		
Nanoparticles	0	2	4	6	8	10		
RHA								

# Table 1 Mixed Composition In Internal Mixer laboplastomill

# Table 2 Mixed Composition In Internal Mixer laboplastomill without PE-g-MA

Materials	Blend Composition (%) wt							
	S <sub>0B</sub>	S <sub>1B</sub>	S <sub>2B</sub>	S <sub>3B</sub>	S <sub>4 B</sub>	S <sub>5B</sub>		
HDPE	100	96	94	92	90	88		
Nanoparticles	0	4	6	8	10	12		
RHA								

# **Results and Discussion**

# **Results Analysis Of Mechanical Properties Of Nanocomposite**

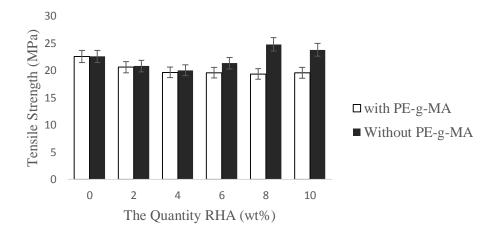
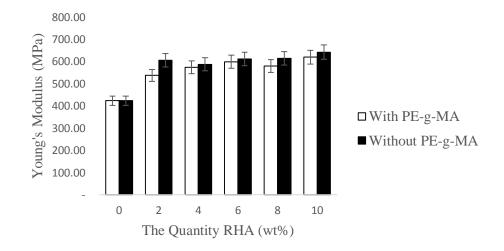
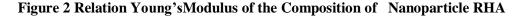


Figure 1. Relation Tensile strength of the Compositionof Nanoparticle RHA





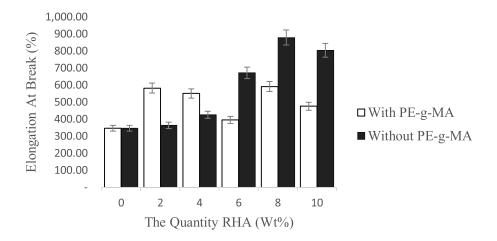


Figure 3 Relationship Elongation at break of the Composition of nanoparticles

#### RHA

From the data on the mechanical properties of tensile strength in Fig. 1 for each sample it was found that the sample with the largest tensile strength value in the 8% RHA composition without the PE-g-MA compatibilizer while the elastic modulus was increased by the addition of RHA filler either without compatible or compatible compatibilizer.

Particle distribution greatly affects the tensile test of a composite material, homogeneously distributed particles increasing the interaction through polymer uptake on the surface of the filler. In contrast, the particles that are not homogeneously distributed may produce agglomerates in the polymer matrix. This weakens the interaction or bonding between the filler and the matrix and results in a decrease in the mechanical properties of the polymer material. [16], as well as the weak bond between the HDPE matrix and the RHA filler due to a gap or pore along with the addition of filler.

The immiscible blend has physical attraction between the weak components at the phase margin, thus causing phase separation under certain conditions and causing the mechanical properties of the mixture to be poor, as the addition of RHA or silica nanoparticles results in a decrease in tensile strength, [17]. As for Young's modulus in Figure 2 there is an increase with the addition of nanoparticles beyond the value of Young's HDPE Modulus of pure 423.58 MPa, and also surpass Young's Modulus from previous studies[18] where Young's highest modulus is 547.80 MPa. The increase in Young's modulus is due to RHA having high stiffness

properties with a purer silica content so that Young's Modulus increase increases with the addition of RHA composition.

Based on the results of mechanical testing (elongation at break) shown in Figure 3 it is known that there is an increase of mechanical properties of HDPE nanocomposite with RHA filler with both compatible and without compatibilizer, value added of mechanical properties including Young's modulus, breaking stress and breaking strain compared to pure HDPE.

The increase in tensile strength is due to an increase in covalent bonds and hydrogen bonds with the OH Group and oxygen from the carbocyte goup each adding a bond between the filler and the matrix according to the study [19]. Likewise, the smaller the size of the filler particles the larger the surface area and the power of interaction or adhesion between the two materials will be greater so that the mechanical properties will be better.

#### **Result of Analysis of Thermal Properties of Nanocomposite**

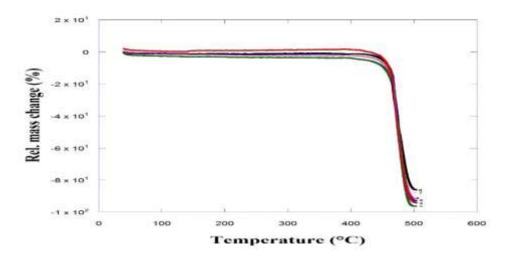


Figure 4. Termogram of TGA Mixed RHA and HDPE With PE-g-MA

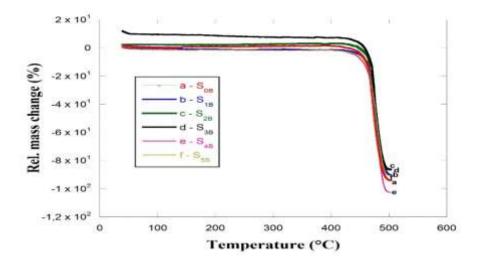


Figure 5 Termogram of TGA Mixed RHA and HDPE Without PE-g-MA

Figures 4 and 5 of TGA temogram analysis for nano composite HDPE / PE-g-MA / nano RHA and without compatibilizer are seen with the increasing composition of RHA addition at decomposition temperature of 500°C the smaller mass changes, this is due to the increasing number of nano content RHA then the decomposition process gets bigger, so thermal stability is better [20] Thermal stability is generally a function of bonding energy as the temperature rises to the point where the vibrating energy causes a breaking of the bond, the corresponding polymer decomposes. as well as for composite nano RHA without compatibiliser. In order for a suitable polymer to be considered heat stable or heat-resistant, the polymer should not decompose below 400°C and should retain its properties at a temperature near the decomposition temperature, where from the TGA analysis it appears that the decomposition temperature is at 500 °C. So it can be concluded that HDPE nano composites with RHA nanoparticle fillers have good thermal properties.

#### Characterization of X-Ray Diffractometry (XRD).

Characterization of nanoparticles using XRD was performed to determine the diffraction pattern and crystalline structure of RHA nanoparticle particles which were then used as HDPE thermoplastic filler. Nanoparticles used are synthesis using PEG 6000 versus 1: 3. with wavelength Ka  $\lambda$  Cu = 1.54060 Å = 0.15406 nm. with the size 52.22 nm [6] diffraction pattern shown in Figure 6. In the picture above can be seen the highest peaks that is at 2 $\Theta$ : 20.530°; 21,729°; 23,282°; 27,392°; 30.076°; 36,100°. The maximum peak is at an angle of 2 $\Theta$  = 21,729 with a distance of 4.0867Å. The result of X-ray diffraction pattern of RHA with PEG-6000 (1: 3) has cristobalite (SiO<sub>2</sub>) phase with lattice parameter a = b  $\neq$  c with a = b = 4,9930 Å and c = 7,0050 Å tetragonal crystal system and has a density of 2.28500 g / cm<sup>3</sup>

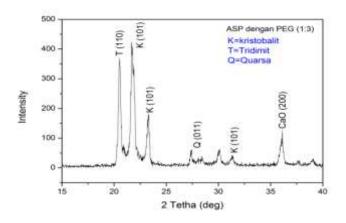


Figure 6. RHA NanoParticle Diffraction Pattern

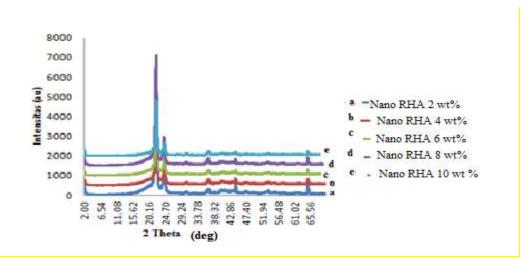
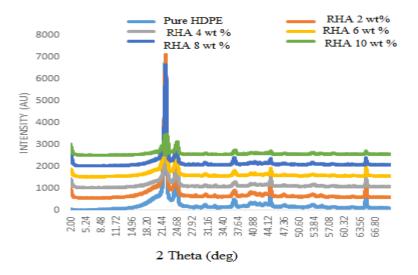


Figure 7 Mixed Diffraction Patterns of RHA and HDPE With PE-g-MA



#### Figure 7 Mixed Diffraction Patterns of RHA and HDPE Without PE-g-MA

From the analysis of X-ray diffraction pattern seen the pattern of HDPE / RHA mixture in Figures 7 and 8 and almost the same as HDPE pattern but with the addition of ASP nano intensity and the distance between the bragg diffraction gratings this indicates the intercalation between HDPE and RHA, with the compatibles and without the PE-g-MA compatibles which can improve the tensile strength and elongation properties of the composite nano. The largest increase in the composition of 8 and 10% for RHA without compatibiliser. The addition of rice husk ash husk composition may reduce the dispersibeality of rice husk ash which may be associated with high filler interaction of RHA resulting in agglomerates, so the intercalation of HDPE matrix melting into RHA interlayer becomes more difficult as is the case[21].

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#### Figure 8. The morphology of HDPE / PE-MA / nanocomposite with RHA(a) 2 %wt (b)10 wt%

# Morphological Analysis

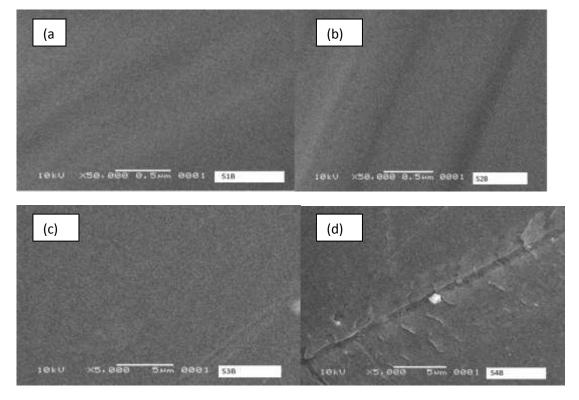


Figure 9. The morphology of HDPE / PE-MA / nanocomposite with RHA (a) wt 2%; (b) 4wt %; (c) 6 wt %; d 8 wt%

Figure. 8a and b exhibited a mixture of RHA nano particles with HDPE respectively in compositions 2 and 8 wt.%, from SEM images seen to homogeneous mixtures and no algomerates resulting in increased tensile strength and elongation of nanoplastic composite nano, as well as Scaning electron microscopy (SEM) figure 9 (a, b, c, d,) with HDPE thermoplastic mixture with RHA nanoparticles.

## Conclusion

The result analysis of composite nano mechanical properties (tensile strength, elongation at break and Young's modulus). increased with RHA nanoparticle composition from XRD analysis result of intercalation between HDPE thermoplastics with RHA nanoparticles so as to add Young's modulus, s and the breaking extension while the thermal analysis of the TGA thermogram shows a decomposition temperature at 500 ° C. Thus, HDPE nano composites with RHA nanoparticle fillers have excellent thermal stability properties. The result of morphological analysis of homogeneous composite nano mixture and no algomerate.

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#### **References:**

- 1. Sun, L., Gong, K., Review, silicon-based materials from rice husks and their applications. Ind. Eng. Chem. Res., 2001,40 ;.5861–5877
- 2. Kamath, S.R., Proctor, A. Silica gel from rice hull ash: preparation and characterization. Cereal Chemistry., 1998, 75; 484-487.
- 3. Thuadaij, N and Nuntiya, A. Synthesis and Characterization of Nanosilica from Rice Husk Ash Prepared by Precipitation Method. CMU.J. Nat. Sci. Special Issue on Nanotechnology.,2008), 7(1) ;206-211

- 4. Supakorn Pukird, Pattanasuk Chamninok, Supon Samran, Pristanuch Kasian, Kiattisak Noipa And Lee Chow. ,Synthesis and Characterization of SiO<sub>2</sub>Nanowires Prepared from Rice Husk Ash, Journal of Metals, Materials and Minerals.,2009,19 (2) ;.33-37
- 5. Ezzat Rafiee, Shabnam Shahebrahimi, Mostafa Feyziand Mahdi Shaterzadeh, Optimization of synthesis and characterization of nanosilica produced from rice husk (a common waste material nternational Nano Letter., 2012, 2;.29
- 6. Ginting ,E..M.,Bukit,N ,Sinulingga,K, Harahap ,M.H,Padang, M. M , Karakterisasi Abu Sekam Padi Disintesis PolietilenGlikol-6000 Sebagai Bahan Pengisi Nano Komposit Termoplastik HDPE ,Jurnal Spektra.,2016,1(1); 36-40
- 7. Tjong,S.C,structural ang mechanical properties of polymer nanocomposites A.review,Journal of Material Scince and Enginneri.,2006, 53;73-197.
- 8. Utracki,L.A.,Sepehr,M., and Boccaleri, E. "Synthetic layerd nanoparticels for polymeric nanocomposites (PNCs); A review."Journal of Polymer Advanced Technology .,2007, 18;1-37
- 9. Sun, L., Gong, K. 'Silicon-based Materials from Rice Husks and Their Applications' Ind. Eng. Chem. Res., 2001, 40;5861-5877.
- Chandrasekhar, S., Satyanarayana. K. G., Pramada, P.N., and Raghavan, P. 'Review Processing, Properties and Applications of Reactive Silica from Rice Husk— An Overview', Journal of Materials Science., 2003, 38; 3159 – 3168.
- 11. Chandrasekhar, S., Pramada, P.N., and Majeed, J. 'Effect of calcination temperature and heating rate on the optical properties and reactivity of rice husk ash', Journal of Materials Science., 2006,41;7926-7933.
- 12. Chakraverty, A., Mishra, P., and Banerjee, D. 'Investigation of Combustion of Raw and Acid-Leached Rice Husk for Production of Pure Amorphous White Silica', Journal of Materials Science., 1998,23; 21-24.
- 13. Wu,Q.,Lei,y.,Clemons,C.M.,Yao,F.,Xu,Y.,and Lian, K,Properties of HDPE/Caly/Wood Nanocomposites, Journal of Plastic Technology.,2007 27;.108-115
- 14. Samal, S. K., Nayak, S., and Mohanty, S. "Polypropylene nanocomposites. Effect of organo-modified layered silicates on mechanical, thermal and motphological performance," Journal of Thermoplastic Composite Material., 2008,8 ;.243-263.
- 15. Lei, Y., Wu, Q., Clemons, C. M., Yao. F., and Xu, Y., "Influence of nanoclay on properties of HDPE/wood composites," Journal of Applied Polymer scienc., 2007, 18;1425-1433.
- 16. Chand, N., dan Jhod, B. D., Mechanical, Electrical, And Thermal Properties Of Maleic Anhydride Modified Rice Husk Filled PVC Composites, BioResources.,2008: 3(4);1228-1243.
- 17. Kord, B., Nanofiller Reinforcement Effects on The Thermal, Dynamic Mechanical, and Morphological Behavior of HDPE/Rice Husk Flour Composites, BioResources., 2011, 6(2);1351-1358.
- 18. Ginting, E. M., Wirjosentono, B., Bukit, N., dan Agusnar, H., (2014), Preparation and Characterization of Rice Husk Ash as Filler Material in to Nanoparticles on HDPE Thermoplastic Composites, Chemistry and Materials Research., 2014,6 (7), ; 14-24
- 19. Bhat, A.H., Abdul Khalil H.P.S, Nano Filler Based on Oil Palm ash Polypropilene Composites, Bio Resouces .,2011,6(2) ;1288-1297
- 20. Bukit, N., Mechanical And Thermal Properties Of Polypropylene Reinforced By Calcined And Uncalcined Zeolite Makara, Technology., 2012, 16(2);121-128
- 21. Ma, J.S., Qi, Z.N., and Hu, Y.L.. "Synthesis and Characterization of Polypropylene/Clay Nanocomposites". Journal of Applied Polymer Science, 200182;3611-3617

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