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# PREPARATION AND CHARACTERIZATION OF OIL PALM EMPTY BUNCHES POWDER AS A FILLER OF POLYPROPYLENE / NATURAL RUBBER

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## ABSTRACT

*The objective of this study was to determine the characteristics of Oil Palm Empty Bunch Powder (OPEBP) into nano particles used as Polypropylene (PP) and Natural Rubber (NR) fillers into elastomeric thermoplastic composite nanocomposite (TPE). The method performed in OPEBP processing into nano particles is used by the method of ball mill and coprecipitation. While the manufacture of nano thermoplastic elastomer composite is done by the internal mixer of laboplastomil. The mixing process was carried out with variation of OPEBP nano composition (0,2,4,6,8,10) wt% mixed thermoplastic (PP) and (NR) and PP-g-MA as compatibilizer. The result of internal mixer was done sample mold, to characterize the test of mechanical properties with JIS K 6781 standard. The characterization results of X-Ray Diffraction (XRD) show that the OPEBP crystalline diameter size is 26.91 nm with monoclinic crystalline structure. phase SiO2 and COMgO4Si with dhkl (011), the elements contained are Si, O, C, Mg. The result of mechanical analysis was obtained by increasing the strength of dance and elastic modulus with the increase of OPEBP nanoparticle composition, the result of XRD analysis of TPE composite nano showed that this diffraction angle shift indicates the intercalation between nanoparticles with thermoplastic PP and NR. The morphological results showed that the homogeneous mixture was evenly distributed and had a porosity of 1.254%.*

**Key words:** Nano Particles, OPEBP, PP, NR.

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## 1. INTRODUCTION

The thermoplastic elastomer (TPE) is a mixture of several polymer compounds which are a combination of thermoplastic properties with conventional elstomeric function. The main factor causing the development of TPE production is its simple compound, quick manufacturing, byproducts easy to use and easy to recycle. The use of cheap and expensive thermoplastic elastomers will result in a reduction in material cost, in addition to improving some mechanical properties.

TPE processing by using natural rubber (NR) has the potential to improve the physical and mechanical properties of new materials, and its use can be further expanded, so research on this continues to grow. Modified dynamic vulcanizers to produce elastomeric thermoplastic elastomeric thermoplastic products in natural zeolite mixtures with NR and PP [1], and several types of plastics widely used to make TPE composites include Polyethylene (PE), Polyvinylchloride (PVC) and Polypropylene, [2]. To make the TPE of both plastic and rubber polymeric materials heated above the glass temperature of each polymer by the melt mixing method, [3]. However, the NR/PP was found to exhibit low abrasion resistance, tear strength, and high rolling strength, which make it unsuitable for many practical uses of rubber materials.

TPE processing using natural rubber (NR) has the potential for improved physical and mechanical properties, to become a new material, and its use can be further expanded, so research continues to grow. Modified dynamic vulcanizers to produce elastomeric thermoplastic elastomeric thermoplastic products in natural zeolite mixtures with natural rubber and PP, several studies have reported that blending of NR with PP resulted in characteristic improvement, such as increased thermalstability , decreased fracture elongation , and increased Young's modulus [1], and several types of plastics widely used to manufacture TPE include Polyethylene (PE), Polyvinylchloride (PVC) and Polypropylene, [2]. To make the TPE of both plastic and rubber polymeric materials heated above the glass temperature of each polymer by the melt mixing method, [3] .

Indonesia is one of the largest palm oil producing countries in the world with an area of 3.76 million hectares or 31.4% of the total area of oil palm plantations in the world. The amount of waste (OPEBP) produced can reach 1.7 million tons / year. OPEBP potential is quite abundant and has not been utilized optimally, for production activities that have high economic value added, which is one of the solid waste generated from oil palm plantation industry this material can be used to become one of filler after going through the processing [4]. One of the methods under development is the addition of filler components in the polymer blend to enhance the mechanical and physical properties of the composite. Type of filler that has been developed one of its silica and carbon black. This is based on the analogy that both types of fillers have been shown to improve the mechanical properties of the vulcanized rubber [5]. One of the most common fillers used is carbon black. Carbon black filler material is active or reinforcing fillers are able to add hardness and resilience tear, abrasion resistance, and high tensile strength on the goods produced. Reinforcing fillers generally have a small particle size, surface active chemical, which has a porous surface and a non-uniform shape can improve adhesion [6]. There are many studies on the utilization the

palm oil ash as areplacement of cementing material [7 -13]; however, only few studies have used the palm oil ash as a raw material for silica production

Polypropylene is one of the most recyclable and lightest thermoplastic polymers among other polymeric materials. The selection of polypropylene (PP) thermoplastic polymers as a composite matrix for easy processing, relatively high melting point 180°C, low density, corrosion resistance, heat conduction and relatively inexpensive process cost, readily available on the market, and used as an ingredient in the automotive industry [14].

To improve the quality of polypropylene, the engineering is carried out usually by adding a rubber material in order to improve the mechanical properties of the polypropylene. To increase the usefulness of natural rubber, a mixture of natural rubber with polypropylene thermoplastic is applied. $SiO_2$ mixed on the PP / NR matrix is expected to be able to improve the mechanical properties of the nanocomposite material to be produced, using PP-g-MA as a compatibilizer which is expected to improve the thermoplastic-based mechanical properties [15].

TPE posses many of the physical properties of rubber such as softness, flexibility and resilience . TPE's are normally not use in application such as automobile tires. Instead, most of their applications are in area which requires softness, and flexibility and also less stringent properties in high temperature, such as footwear, automotive moulded part, head lamp casing, handle of heavy duty hand tools, wire insulation and adhesives. There has been much research on thermoplastic and natural rubber mixtures, among others [16, 17,18, 19]

The objective of this research is to make a new material that is thermoplastic elastomer (TPE) with mixture of PP / NR as a matrix with OPEBP nanoparticle filler, nano particles obtained from preparation process using ball mill and coprecipitation method, where OPEBP is expected as carbon black replacement commercial. The products used by TPE are expected to be used as one of the engineering materials that have good mechanical properties.

## 2. EXPERIMENTAL AND METHODOLOGY

### 2.1. Materials

The material used is oil palm empty fruit bunches (OPEBP) from CV. Selaxa Windu, HCl 6M and $NH_4OH$ 3M Used in this work were produced by MERCK, Germany, polypropylene and PP-g-MA Japanese production, Natural Rubber Standard Indonesia Ruber (SIR) -20 from Testing Center and product quality certification Medan PTPN. Polypropylene copolymer was obtained from Production Singapore. This polymer has melting point of 176 ° C, and density of 0.896 g / cm3, Polypropylene grafted maleic anhydride (PP-g-MA) was obtained from Japan.

### 2.2. The process of making nanoparticles is done by ball mill and co-precipitation methods

OPEBP material in furnace with temperature $150^0$C for 1 hour result furnace in milling with Planetary Ball Mill Retsch PM 200 for 2 hours with speed 250 rpm.OPEBP produced in 200 mesh sieve to produce 74 μm particle size. then synthesized using coprecipitation methods. of 50 grams of OPEBP were put into a measuring flask, then dissolved with 6 M HCl solution at a ratio of 1: 2, ie 50 g of OPEBP and 100 ml of HCl 6 M and then stirred and heated in a magnetic stirrer at 700 ° C for 40 minutes, then dissolved with 100 ml $NH_4OH$ solution of 150 ml. stirred and heated in a magnetic stirrer at a temperature of $70^0$C for 40 minutes. The washed solution was then precipitated using a centrifuge at 3500 rpm for 30 minutes. The precipitate was dried in the oven for 4 hours at $70^0$C. The results of OPEBP synthesis were analyzed by XRD and SEM.

## 2.3. OPEBP Synthesis Process

Coprecipitation methods with acid-base reactions, wherein HCl as solvent and $NH_4OH$ as a precipitator are generally described:

$$Na_2O + HCl \longrightarrow 2NaCl + H_2O$$
$$MnO + 2HCl \longrightarrow MnCl_2 + H_2O$$
$$MgO + 2HCl \longrightarrow MgCl_2 + H_2O$$
$$P_2O_5 + 10HCl \longrightarrow P_2Cl_{10} + 5H_2O$$
$$Al_2O_3 + 6HCl \longrightarrow Al_2Cl_6 + 3H_2O$$
$$CaO + 2HCl \longrightarrow CaCl_2 + H_2O$$
$$SiO_2 + HCl \longrightarrow SiCl_4 + 2H_2O \quad (1)$$

OPEBP solution was reconstituted with 3M 150 ml $NH_4OH$ by reaction as follows:

$$SiCl_{4(s)} + 2H_2O_{(l)} + Na_4OH_{(l)} \Rightarrow SiO_{2(s)} + 4NaCl_{(l)} + 2H_2O_{(l)} \quad (2)$$

The washed solution was then precipitated using a centrifuge at 3500 rpm for 30 minutes. The OPEBP precipitate was dried in the oven for 4 hours with a temperature of $70^0$C. The results are shown in Figure 1 (a).

## 2.4. Preparation of mixed thermoplastic elastomer PP / NR / OPEBP

The preparation process was carried out in the Internal Mixer Technical Cooperationby the Government of Japan type of Laboplastomill 30 RI50 volume 60 cc chamber by mixing the material shown in Table 1. PP was melted for 3 minutes, then natural rubber was put on for 4 minutes. After that the OPEBP nanoparticles are mixed for another 2 minutes in add PP-g-MA compatibility for 1 minute. The blending temperature is 180°C and the speed is 60 rpm for 10 minutes. The results of the internal laboplastomil are then fed into a square-shaped molding device with a 1 mm thick plate with a length of 11 cm and a width of 11 cm. The press is then pressed 37 tons Genno Japan done for 15 minutes consisting of a 5 minute heating time, 5 minute heating time and a 5 minute press time, the resulting nanocomposite result is shown in Figure 1 (b). Then the sample sheet is cut using a Jumb K 6781 dumbell cutting machine for the tensile test sample shown in Figure 1 (c).

**Table 1** The Contents Blends Internal Mixer Laboplastomil

| **Materials** | **Contents (Wt %)** | | | | | |
|---|---|---|---|---|---|---|
| | **$S_0$** | **$S_1$** | **$S_2$** | **$S_3$** | **$S_4$** | **$S_5$** |
| PP | 88 | 86 | 84 | 82 | 80 | 78 |
| PP-g-MA | 2 | 2 | 2 | 2 | 2 | 2 |
| NR 20 | 10 | 10 | 10 | 10 | 10 | 10 |
| OPEBP | 0 | 2 | 4 | 6 | 8 | 10 |

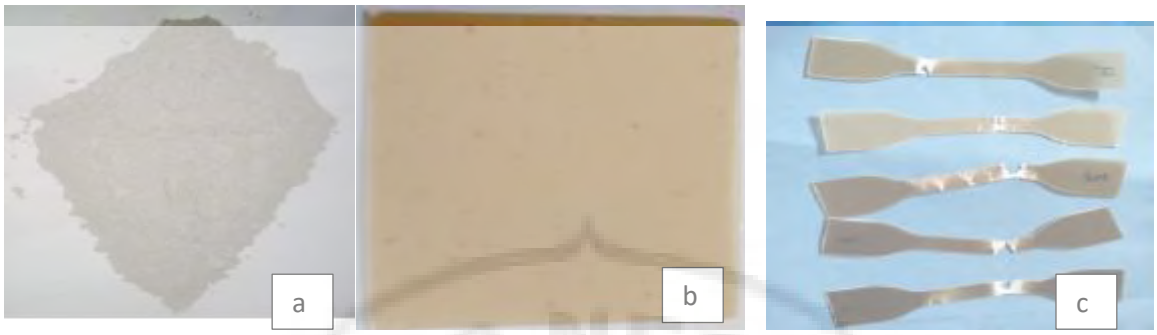



**Figure 1**. (a) OPEFB nano particles (b) Nano composite TPE (c) Sample test mechanis

## 3. RESULTS AND DISCUTION

### 3.1. Analysis of X-Ray Diffractometry (XRD)

Figure 1 (a) characterization and Analysis of OPEBP nanoparticles using XRD type Shimadzu 6100 Cu K $\lambda$ 1,54418 Å, 40 kV, 30 mA).Measurements were made to examine the interlayer activity in the composite as prepared. Bragg's Law ($n\lambda = 2d \sin \theta$) was used to calculate the crystallographic spacing. The diffractograms were scanned from 20° to 60° and $7^0$ to $70^0$ in the 2θ range in the step size of 0.04 s with the scanning speed 2.00 deg/m The samples were chosen in random. from the analysis can be determined phase, crystal structure, lattice parameters, to determine the particle size by Scherrer equation and the $d_{hkl}$ plane of the x-ray diffraction pattern shown in Fig. 2.

Using the Match software we found the lattice parameter a = 14.3300 Å b = 13.8300 Å c = 7.7900 Å β = 117.1 00 º and the highest $d_{hkl}$ plane was on the millier index (0 1 1) with the monoclinic crystal structure. XRD analysis of OPEBP obtained phase ie $SiO_2$ and $COMgO_4Si$. In Fig. 2. has the eight highest peak representation shown in the $d_{hkl}$ (0 1 1), (3 1 1), (1 1-1), (0 2 0), (1 3 1), (2 0-2 ), (1 3 1) and (1 1 2). With density of 2.875 g / cm ³.

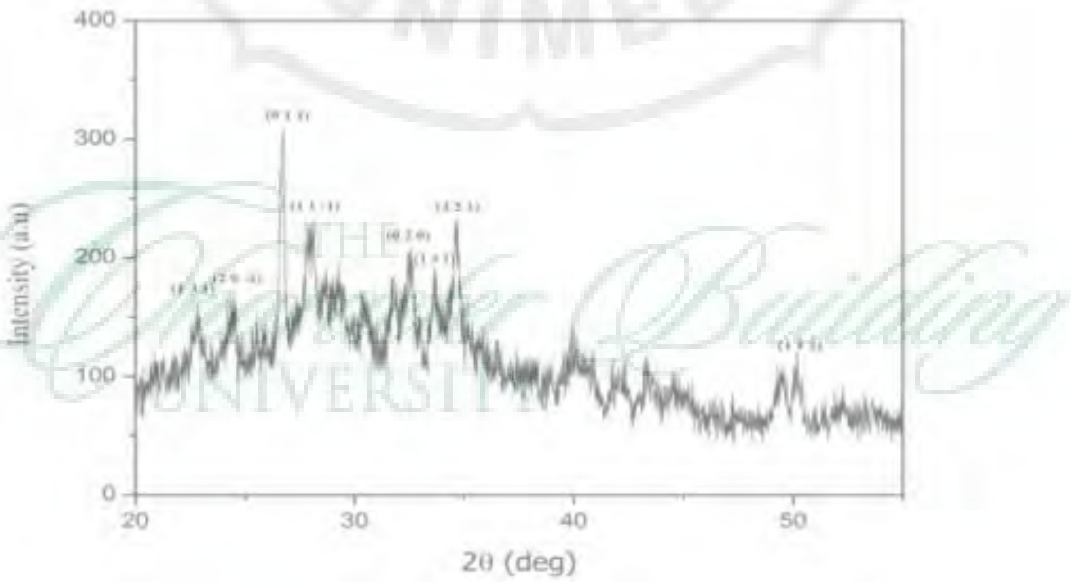



**Figure 2.** XRD pattern of OPEBP.

### 3.2. Nano Composite TPE Analysis (blend PP / NR / OPEBP)

XRD analysis with scan from angle Θ = $7^0$ to $70^0$ on nano TPE composite (mixture PP / NR / OPEBP) from sample Figure. 1 (b) obtained the diffraction pattern shown in Fig. 3 from the analysis result obtained diffraction pattern in general diffraction pattern almost same with the PP thermoplastic diffraction pattern but with the addition of nano particles OPEBP seen there is a change in intetensity and the distance between the bragg diffraction lattice there is a shift, this indicates the intercalation between matrix PP / NR with the addition of variation of nano particle particle composition, and PP-g-MA compatibilers, the occurrence of this intercalation may improve the properties of tensile strength and elastic modulus as well as the elongation at break of the increased TPE nano composites. The greatest increase in the composition of 4 wt%. Addition of the nano composition of OPEBP at 10 wt% can also reduce the dispersibeality that can be associated with the interaction of the filler of the high composition to produce agglomerates, thereby reducing the tensile strength of the elastomeric thermoplastic composite, as is the case [20,21].

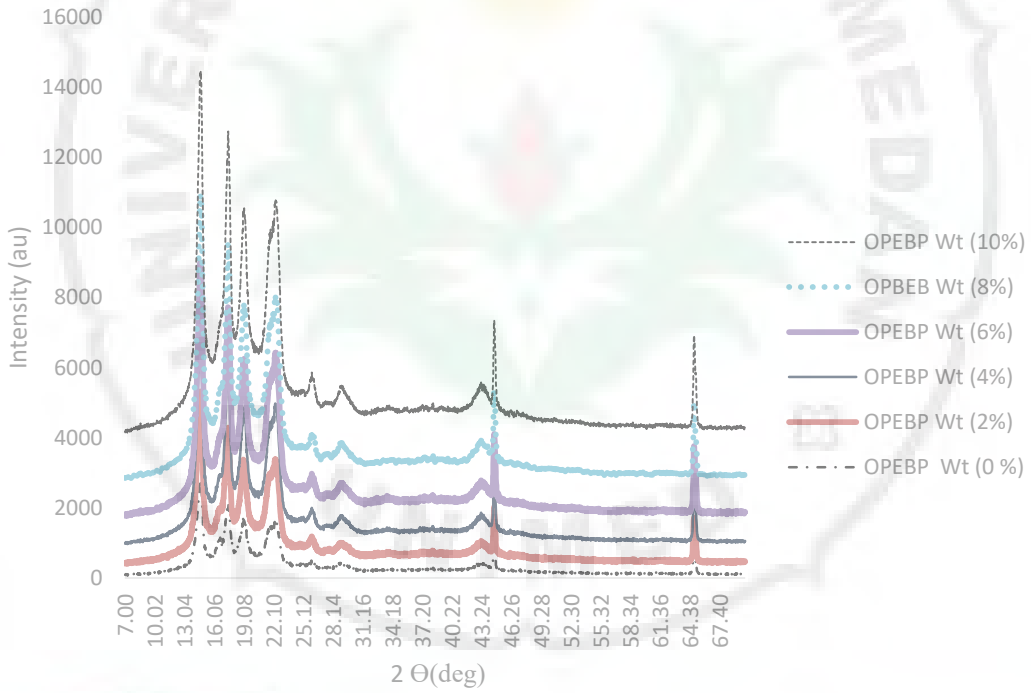



**Figure 3.** XRD patterns nano composite TPE with filler contents OPEBP

### 3.4. Morphological characterization

Morphology studies The structures of nano partikel OPEBP and nano composites TPE were characterized by Scanning Electron Scanning Electron Microscopy (SEM). The synthesis of OPEBP nanoparticles was analyzed by Scanning Electron Microscope (SEM) type Carl Zeiss EVO MA10, EDX (Energy Dispersive X-ray) with magnification 4,000 times, shown in Figure 4. From the picture it can be seen that the OPEBP distrubution looks like a lump of some nano particle. The result of EDX analysis obtained by chemical element contained in OPEBP in general is Si, O, C, Mg with element composition shown in Table 2, while Figure 5 shows mixed composite morphology PP / NR / PP-g-MA / 0, 4, 6) wt% OPEBP. In Fig. 5. (a) shows TPE morphology with 0 wt% OPEBP composition, Fig. 5 (b) Composites with 4 wt% OPEBP and Fig. 5 (c) TPE composites with 6 wt% OPEBP, from morphological results at 4wt% seen mixed which is more homogeneous, it indicates that nanocomposites are more

evenly distributed where less gap or pore arises and small particle size in general, this may result in better interaction between PP and NR polymer matrix and filler than the amount of 10 wt% large composition so that the tensile strength of the sample is better this is in accordance with research [22]. The distribution of the mixture is also affected by the stress history that occurs during the mixing process. Stress history plays a role to break up natural rubber particles and distribute them into the polypropylene matrix, due to mixed systems with dynamic vulcanized [1] . In the 4 wt% composition OPEBP agglomeration on the composite surface is less than the 6 wt% composition. Percent porosity at 4wt% composition of 1.25%. While at the composition of 6 wt% there has been clumping, ie there are more gaps and pores that is equal to 1.61%, this is also appropriate from the analysis of composite mechanical properties resulted in tensile strength decreased, this is in line with the addition of OPEBP nanopartikel resulted in the decline tensile strength [8]. The image of the porosity area of the composition (0.4,6) wt% wt with the OPEBP nanoparticle filler is shown in Fig. 6 (a, b, c). With the addition of PP-g-MA increases the bond between the face between the OPEBP nanoparticle filler and the PP / NR matrix. Similarly, the results of the study [23] using PP-g-MA may increase the interface bond and reduce the fibers involved from the nanocomposite.

From Figure 6b. shows the porosity area of the sample without the addition of filler, a sample of 4wt% composition and a sample of 6 wt% composition can be seen that the addition of filler to the material reduces the porosity of the material. The porosity percentage in the sample without filler addition was 4.538%, compared to the porosity percentage of 4% composition by 1.254% and the 6% composition of 1.615%. From Figure 6 c. which shows the porosity area of the sample without the addition of filler, the composition sample of 4 wt% and the composition sample of 6 wt% can be seen that the addition of filler to the material reduces the porosity of the material. The porosity percentage in the sample without filler addition was 4.538%, compared to the porosity percentage of 4% composition by 1.254% and the 6% composition of 1.615%. the greater the surface area of the adsorbent pore, the greater the adsorption power.

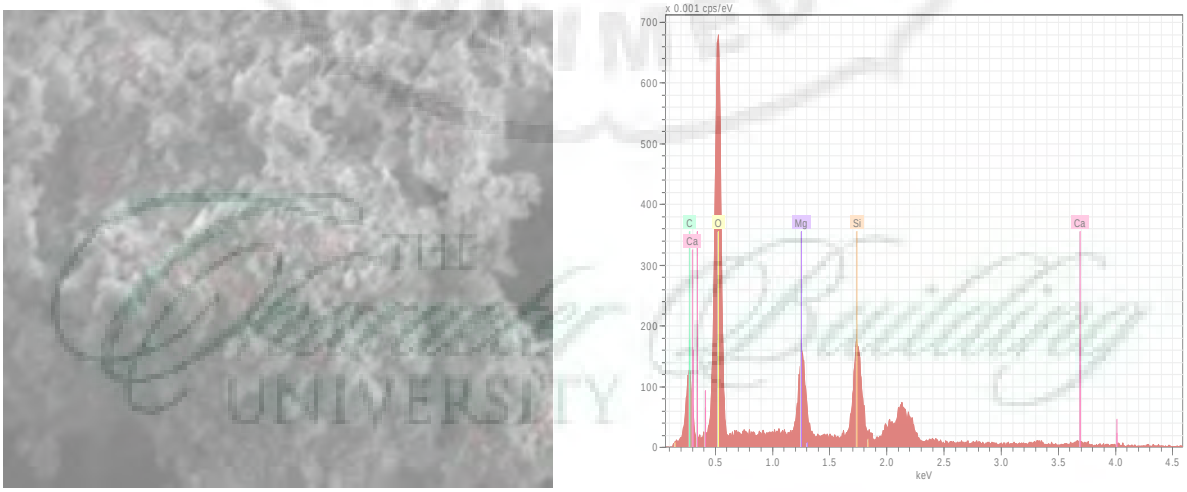



**Figure 4**. SEM images of synthesized products on OPEBP substrate after treatment**.**

**Table 2** Elements Conteints in OPEBP

| Element | The Quantity (Wt %) |
|---|---|
| O | 55.15 |
| Si | 27.09 |
| C | 10.17 |
| Mg | 7.60 |

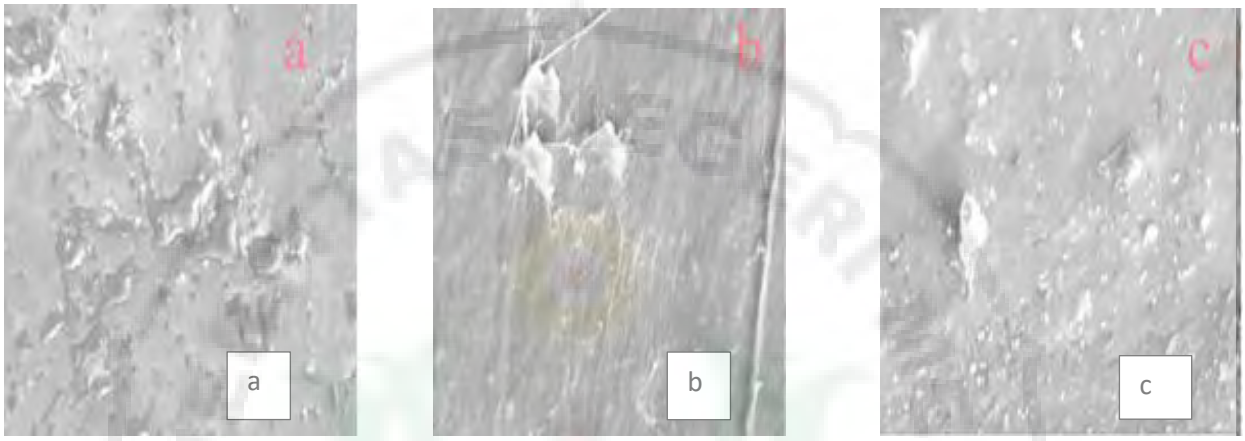



**Figure 5** SEM images of PP/ (NR) / contents OPEBP (a). wt 0% ; (b). 4 wt %; (c). 6 wt %.

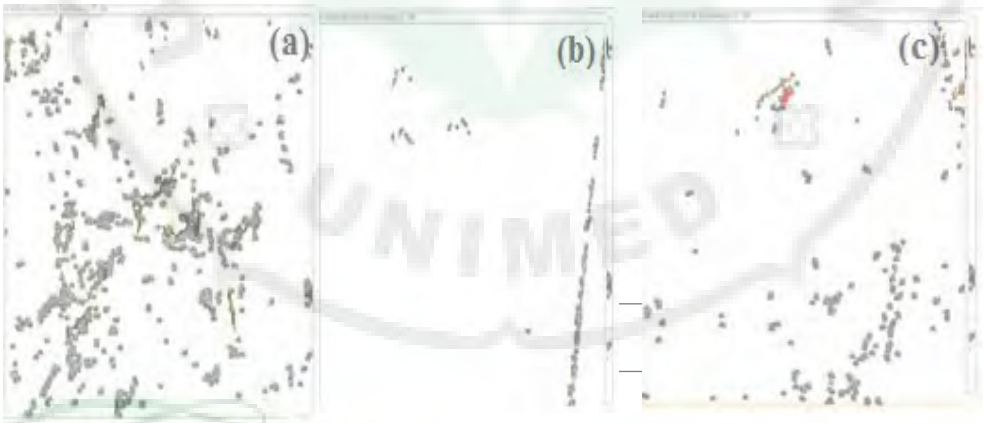



**Figure 6.** Porosity area of sample contents (a). 0 wt%; (b). 4wt %; (c). 6 wt% .

### 3.5. Mechanical properties measurement.

Tensile strength measurement was performed according to JIS K 6781 standard using a e Universal Testing Machine Analysis of Mechanical Properties nano composites produced Tests conducted with a tensile testing machine stograph R-1 brand Toyoseki Japan . All of these were determined at a crosshead speed of 50 mm/min at room temperature and averages of 5 successive tests reported. Young's modulus (E), ultimate tensile strength (σmax), and fracture elogation at break (εb) were determined from the stressstrain curves.

The result of TPE thermoplastic mechanical characterization characterization was obtained tensile strength data, broken strain, breaking voltage, elastic modulus and elongation break in each sample variation (0, 2, 4, 6, 8 and 10) wt%, as shown in Figure 7. Show a mixture of polypropylene thermoplastic and natural rubber with OPEBP filler with the

addition of PP-g-MA as a comboibilizer has a higher tensile strength value than the tensile strength of the mixture without addition of filler.

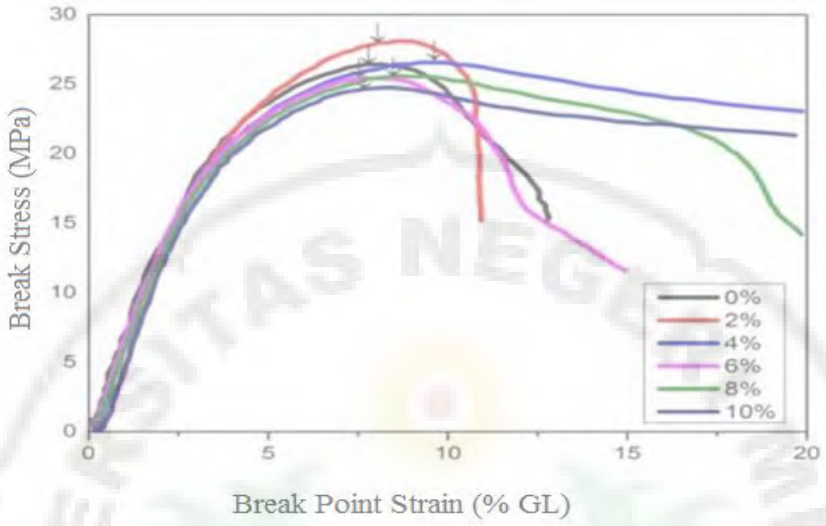
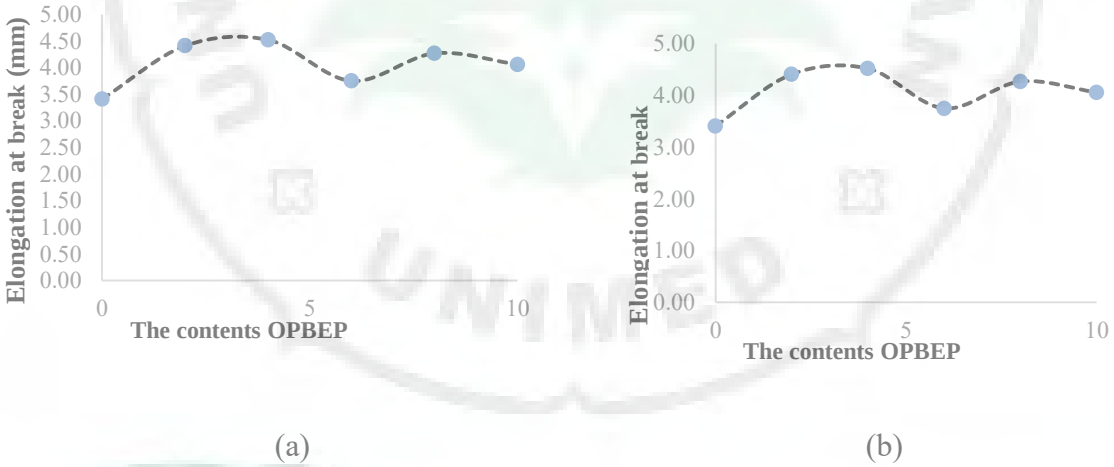
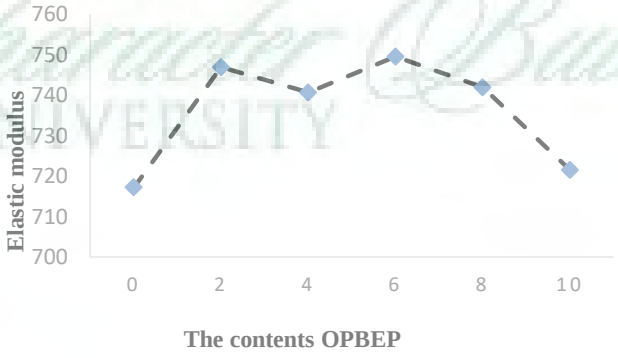



**Figure7.** Relationship tensile strength and breaking strain nano composite TPE of some quantity OPEBPstress-strain curves for samples with different concentrations.




(a) (b)




(c)

**Figure 8.** The contents OPEBP (a) Tensile strength; (b) Elongation at break; (c) Elastic modulus

The mechanical properties of TPE nanocomposites, Decrease in mechanical properties, particularly tensile strengths occurring in the composition of 10 wt% of fillers, this is due to this composition of the process of filler agglomeration in TPE nano composites, the composition of filler is too high, to achieve a homogeneous mixture of the mixing process [8]. The presence of particle agglomeration in morphological results with SEM is shown in general in Fig. 5. This is also the result of tensile strength testing resulting in a decrease in strength. from the result of mechanical analysis resulted tensile strength decreasing and elongation of dropout decrease, this matter because of the increasing of silica content causing decrease of tensile strength, this is in accordance with some research result [24, 25, 26, 27]

Fig. 8 a, b and c, shows the results of the tensile strength test and the elongation of the break and elastic modulus of the PP / NR / OPEBP composite nano with the composition variation of (0, 2, 4, 6, 8, 10) wt%. increase in tensile strength up to 8 wt% OPEBP composition compared to composite without OPEBP nanoparticles, but maximum strength was obtained in 4 wt% state while at 10 wt% composition was decreased Based on Figure 8 b the value of elongation of breaks due to the addition of OPEBP nanoparticles showed an increase up to variation 4% weight. In this case the more filler addition to the mixture the material becomes more rigid so that the value of the elongation breaks down the low [28]. The addition of the value of the elongation at break will affect the modulus of elasticity. The higher the elongation value breaks the elasticity modulus value will be lower [29].

In Figure 8c. it can be seen that the increasing composition of OPEBP nanoparticles in the mixture of PP and NR. The increase in elastic modulus is due to OPEBP having high stiffness properties with a purer silica content so that the increase in Elastic Modulus increases with the addition of this composition in accordance with [1].

The presence of agglomeration of OPEBP particles as shown in SEM Figure 5 also causes a decrease in tensile strength. OPEBP particle agglomeration becomes the place of stress concentration and becomes the beginning of the crack so that the force will decrease. Given the PP-g-MA compatibility it can increase tensile strength and also lead to increased chemical interaction of components. These interactions lower the facial stress and result in better adhesion [30]. The addition of PP-g-MA can increase homogeneity and decrease the size of the distributed natural rubber phase. In mixing PP / NR using internal mixer equipment is expected to produce a more even distribution of phases, as well as an increase in filler composition.

## 4. CONCLUSIONS

XRD analysis of OPEBP obtained by particle size of 26,91 nm with monoclinic crystal structure. contained elements Si, O, C, Mg, whereas for nano TPE composites (blends of PP / NR / PP-g-MA and nanoparticles OPEBP) there is an interaction of nano particles of OPEBP with PP / NR matrix. The tensile strength and elastic modulus of TPE increases with the increase in the nano particle composition of the maximum tensile strength obtained at a composition of 4 wt% to above 10 wt% of a decrease in tensile strength and elastic modulus, this is due to particle agglomeration, resulting in elongation at break also decreasing. Similarly TPE morphological analysis results show homogeneous mixture distribution for composition below 6 wt% and has porosity value of 1.254%

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