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Mechanical properties of composite thermoplastic hdpe / natural rubber and palm oil boiler ash as a filler

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Abstract. This study aims to determine the mechanical properties of natural rubber and High-density polyethylene (HDPE) nanocomposites using 72 nm Palm Oil Boiler Ash (POBA) as filler. The Open mill method is used in the preparation of SIR 20 compounds. Nanocomposite preparation was carried out by varying the composition of nanoparticles (0,5,10,15,20)%. Sample preparation was carried out using 60 cc volume internal mixers with JIS K 6781 standard. The results showed that Young's modulus of thermoplastic elastomer with 20% POBA composition increased to 551.47 MPa and exceeded the pure HD Modulus Young value of 423.58 MPa. Morphological analysis showed that with the addition of nanoparticles, the morphology of the mixture was homogeneous

1. Introduction

Nanoparticles have more superior properties than bulk material. In creating of high quality materials, it is necessary to combine two or more basic elements which different, one of them have function as a matrix and the other have function as a filler.

Palm Oil Boiler Ash (POBA) can be used as an economic and environmentally friendly filler. POBA is ash derived from shells and fruit fibers which have been ground and burned at a temperature of 500-700 °C in a boiler furnace. Palm ash from the remaining combustion of shells and palm fruit fibers contains chemical elements of silica (SiO₂) 31.45%, (CaO) 15.2%. The burned shell and fiber produce a grayish hard crust caused by combustion at high temperatures with a silica content of 49.2% [1]. POBA can be used as a filler between thermoplastic and rubber which can produce a material called thermoplastic elastomer (TPE). there are several previous studies that use palm oil boiler ash as filler such as making thermoplastic with HDPE as matrix and POBA as filler [2]. Research on POBA mixture with polypropylene matrix [3], and Geopolymer Material [4].

TPE products have the potential to develop into commercial products using carbon black and POBA as fillers. Thermoplastic elastomers have properties and functions similar to vulcanized rubber and can be melted like thermoplastics at high temperatures. The unique characteristics of TPE are very useful as an alternative material and can be used in various industries such as the automotive industry [5]. The purpose of this study was to process POBA into nanoparticles and used as a filler in thermoplastic elastomers.

2. Method

The materials used in thermoplastic elastomer nanocomposite preparation are POBA (PT. PP. Lonsum), HDPE (Singapore production), Rubber SIR-20 (production of Medan PTPN Testing Hall), Carbon black (Karbomax brand), PE, g-MA (Japanese production), Sulfur, ZDEC (PT Deli rubber),

Paraffin Wax (Chinese production), Zinc oxide, Stearic Acid, and BHT (PT. Brataco). Thermoplastic elastomer nanocomposite preparation was carried out in three stages

2.1. Preparation of Nanoparticles POBA

POBA from the oil palm shell that burned in the boiler furnace was dried in an oven for 30 minutes at 150^o C, then ground using PM 200 ball mill for 1 hour, then filtered using a 74 μ m (200 mesh) sieve. POBA was mixed and dissolved with 2M HCL for 40 minutes with a magnetic stirrer, then filtered with filter paper then mixed and dissolved with 2.5M NaOH for 40 minutes with a magnetic stirrer, then filtered with filter paper washed with distilled water. then dried in an oven to temperature of 70^oC for 4 hours according to previous research methods [6]

2.2. Preparation of SIR-20 Compound

SIR-20 compound is prepared using Open mill at room temperature. the steps and materials used are shown in Table 1.

Table 1. Stages of preparation of compound sir-20 in the open mill

Material	minute
Ruber SIR-20	0
ZnO	5
Stearic Acid	10
Carbon Black	15
ZDEC	20
BHT	25
Wax	30
Sulfur	35
Termination of the mixing process	40

2.3. Preparation of Thermoplastic Elastomer

Thermoplastic Elastomer (TPE) sample which is a compound mixture SIR-20, POBA, and HDPE made using an internal mixer. The order of the mixing process in the internal mixer is shown in Table 2.

Table 2. Stages of preparation TPE in Internal mixer.

Dynamic vulcanization	Minute of
HDPE melting process	3
Compound SIR-20	6
POBA Nanoparticle	8
PE.g-MA	9
Termination of the mixing process	10

The raw material that have been mixed are put into a rectangular mold with a thickness of 0.1 cm, a length of 11 cm, and a width of 11 cm. Previously the material was weighed first with the balance sheet (according to the volume of the mold plate 12.1 cm³). Then it was pressed hot for 15 minutes which consisted of 5 minutes of heating mold, 5 minutes of heating material and 5 minutes of pressure with 37 tons of pressure at 150^o C, then continued with cold pressure for 5 minutes with the same pressure of the last 37 tons of sample in the form of sheets removed from the mold. Test samples were made using a dumbbell sample cutting machine, for tensile test samples made with JIS K 6781 standard. [7-8]

3. Result and Discussion

3.1. XRD characterization

XRD characterization using Shimadzu 6100 (40 kV, 30 mA) at a rate of 2° / minute with an angle range of 2θ = 5° -70°. XRD testing is carried out at room temperature and uses nickel to filter CuKα radiation. The sample crystallite size is calculated based on the Scherrer method analysis of X-ray diffraction patterns

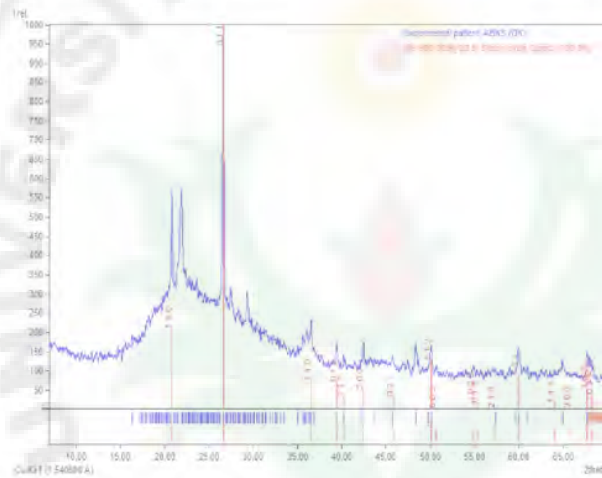


Figure 1. XRD diffraction pattern of POBA nanoparticles

POBA nanoparticle size is shown in Figure 1. The highest peak diffraction pattern is SiO₂ which has trigonal crystal structure and quartz phase, space group P 32 2 1 with maximum peak intensity at dhkl [0 1 1] and lattice parameter a = b = 4,9124 Å and c = 5,4039 Å with α = β = 90° and γ = 60°. From the XRD diffraction pattern, the particle size is obtained by calculating the amount of Full Width at Half Maximum (FWHM) from the diffraction peak through the Scherrer equation approach. FWHM is converted to radians by multiplying π / 180.

$$D = \frac{K\lambda}{\beta \cos \theta}$$

By calculating the equation, the average particle size of palm oil boiler ash is 72 nm so that POBA particles become nanoparticles. The POBA particle size results in this study obtained a better nanoparticle size than the previous study [4], which was 85.35 nm and [2] and 100 nm, because the method used in POBA processing was different from previous studies.

3.2. XRF characterization

The composition of POBA Nanoparticles in Tables 3 and 4 shows the results of the XRF test.

Table 3. XRF Results of POBA nanoparticles with HCL NaOH solutions

Element	Composition (%)weight
Mg	0,7
Al	5,7
Si	79,9
Ni	0,05
Ti	0,88
Mn	0,62
Fe	11,64
Cu	0,24
Zn	0,08
Zr	0,09
Total	100

Table 4. XRF Results of POBA nanoparticles with NaOH solutions

Element	Composition (%) weight
Mg	1,55
Al	16,52
Si	37,03
P	1,89
S	0,95
Ti	1,26
Mn	0,97
Fe	19,51
Co	0,08
Cu	0,43
Zn	0,19
Zr	0,26
Ag	4,12
Sn	13,76
Sb	1,46
Total	100

Table 3 and 4 shows the results of POBA nanoparticles analysis consisting of Si as much as 37.031% wt more dominant than Al and Fe elements which amounted to 19.51% wt and 16.52% wt, the number of Si in less than the number of Si in Table 4, with Si as much as 79.9% wt, Al 5.7% wt and Fe by 11.64% wt. The elements in POBA in Tables 3 and 4 are different. The difference is because the solution used is different, in this study the solution used was HCL and NaOH, whereas in the previous study used HCL and NH4OH solutions [4].

3.3. Mechanical Properties and TPE Morphology

Mechanical testing of HDPE / SIR-20 / POBA nanocomposite using PE.g-MA with Universal Testing Machine (Tensilo 5T), obtained tensile strength, elongation break and Youngs modulus

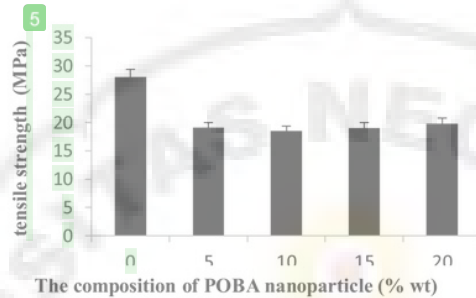


Figure 2. The relationship between tensile strength with POBA nanoparticles

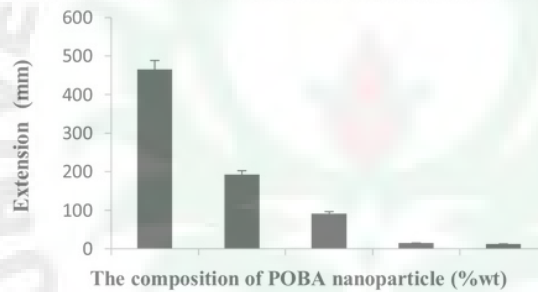


Figure 3. The relationship between elongation breaks with POBA nanoparticles

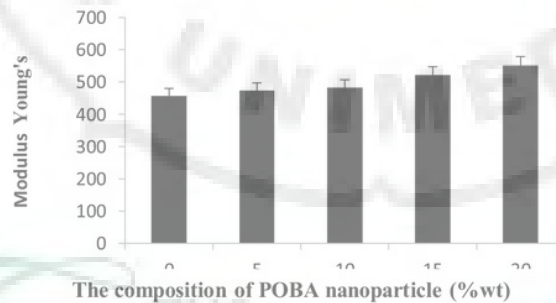


Figure 4. The relationship between Young's modulus and POBA nanoparticles

The tensile strength test of thermoplastic elastomer nanocomposite on the variation of the composition of palm oil boiler ash is shown in Figure 2. It was obtained that the sample experienced a decrease in tensile strength with the addition of palm oil boiler ash nanoparticles in the composition of POBA 5% wt and 10% wt with a tensile strength of 19.06 MPa and 18.46 MPa, but the tensile strength at a composition of 15% wt and 20% wt increased by 19.04 MPa and 19.80 MPa. Even so, the value of tensile strength with POBA addition is lower than the thermoplastic elastomer without filler which is 28.01 MPa with the composition of HDPE 94% wt and SIR-20 Compound 5% wt. In the composition of POBA 0% wt with HDPE 94% wt and SIR-20 compound 5% wt obtained the results of tensile strength better than pure HDPE, because there are still nanoparticles agglomeration when inserted in the internal mixer is not homogeneous.

NR / PP composite research using palm ash filler has good tensile strength and morphology. The best tensile strength is obtained in the composition of palm ash of 30 phr. Under these conditions the tensile strength of the mixture is 9.6 MPa and the tensile strength decreases in the addition of 45 phr palm ash to 6.1 MPa. The decrease in mechanical properties in the higher filler number is estimated to be caused by several possibilities. The first possibility is the occurrence of a filler agglomeration process which indeed tends to occur if the levels are too high. The second possibility is that it is difficult to achieve a homogeneous mixture in the mixing process using an internal mixer for high filler content [3].

The results of thermoplastic elastomer nanocomposite elongation analysis on the variation of the composition of palm oil boiler ash are shown in Figure 3, obtained that the sample with the largest elongation elongation value was in the sample 1 of 465.4 mm without POBA filler material and all four samples had a decreased elongation at each addition POBA. This is because the more number of fillers added, the more rigid the material will be due to the lower extension value.

The results of Young's modulus thermoplastic elastomer nanocomposite analysis on the variation of the composition of palm oil boiler ash are shown in Figure 4, obtained that the sample with the largest young's modulus value is the composition of POBA 20 %wt of 551.47 MPa and exceeds the pure Young's HDPE Modulus value of 423.58 MPa, and also surpasses Modulus Young's from previous research. Modulus Young's highest is 551.43 MPa. Young's Modulus Improvement is caused by palm oil boiler ash having high stiffness properties with purer silica content and the interaction between thermoplastic elastomers with POBA that are dispersed individually which has a large surface contact area so that it can bind strongly to HDPE and PE-g. MA. this is consistent with previous research [2]

The results of the mechanical properties of thermoplastic elastomeric nanocomposites using Universal Testing Machine (UTM) obtained the data shown in Figure 5 the relationship between stress and strain as a result of the addition of a varied POBA filler.

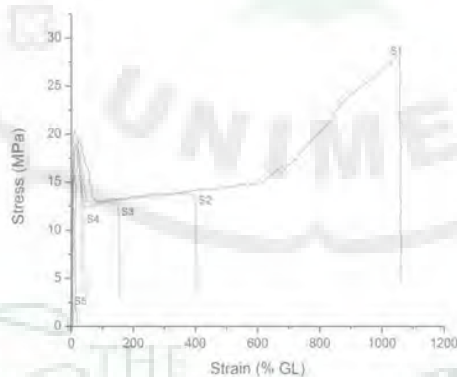


Figure 5. The results of tensile tests on POBA S1 (0), S2 (5), S3 (10), S4 (15), S5 (20)% wt

3.4. Morphological Analysis Results

Morphological testing was carried out using a Scanning Electron Microscope (SEM) tool. Previously samples were coated with gold using JEOL Fine Coat (Ion Sputter).

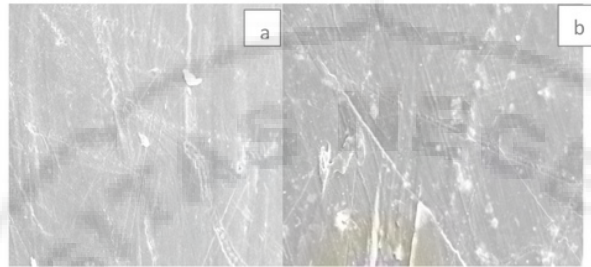


Figure 6. Morphology of TPE with POBA nanoparticles filler (a) 5% wt,(b) (20 % wt

Figure 6 morphology of thermoplastic elastomers with POBA fillers 20% wt showed surface structure that was not evenly distributed and there were more agglomerations compared to the thermoplastic elastomer morphology with a 5% wt filler, it was suspected to be the cause of the elongation break decreased [2].

Conclusion

From the results of the study obtained silica content of 37.03% and the average particle size of 72 nm. The best composition of thermoplastic elastomer nanocomposite with POBA nanoparticle filler is in the composition of 20% wt. The more POBA nanoparticles fillers, the more modest Young's modulus, but the elongation of the break and the tensile strength decreased. In general, the results of TPE mixture morphology are distributed evenly and homogeneously

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Acknowledgments

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