



Characterization of Low-Density Polyethylene (LDPE) Thermoplastics with Rice Husk Ash Nanosilica Filler Synthesized Using the Coprecipitation Method

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ABSTRACT

LDPE thermoplastics have been made with rice husk ash nanosilica filler, which is synthesized using the coprecipitation method. The nanosynthesis of rice husk ash silica was carried out with HCl 2M and NaOH 2.5M solvents with a ratio of 1:3. The synthesized nanosilica was then characterized using XRD and obtained an average size of 23.09 nm. Based on the results of XRF characterization, it is known that there is 92.99% SiO₂ and the most abundant element, namely Si, 43.47%. The composition of each LDPE/nanosilica rice husk silica was varied (100/0, 98/2, 96/4, 94/6, and 92/8 % by weight), and mechanical properties in the form of tensile strength, elongation at break, and modulus of elasticity were tested using ASTM D638 standard. The results of the mechanical test were obtained with the optimum tensile strength value in the composition (96/4% by weight) of 10.53 MPa, the optimal elongation value at the break in the composition (100/0% by weight) of 201.5%, and the optimum elastic modulus value in the composition (96/4% by weight) of 226.57 MPa. The addition of RICE HUSK SILICA nanosilica as a filler is proven to improve the mechanical properties of LDPE thermoplastic in terms of tensile strength and modulus of elasticity compared to LDPE without filler material (100/0% by weight).

Keywords: Coprecipitation, LDPE, Mechanical Test, Nanosilica Rice Husk Silica, XRD

ABSTRAK

Telah dilakukan pembuatan termoplastik LDPE dengan filler nanosilika abu sekam padi yang disintesis menggunakan metode kopresipitasi. Sintesis nanosilika abu sekam padi (ASP) dilakukan dengan pelarut HCl 2M dan NaOH 2,5M dengan perbandingan 1:3. Nanosilika hasil sintesis selanjutnya dikarakterisasi menggunakan XRD dan diperoleh ukuran rata-rata 23,09 nm. Berdasarkan hasil karakterisasi XRF diketahui bahwa terdapat 92,99% senyawa SiO₂ dan unsur terbanyak yaitu Si 43,47%. Komposisi masing-masing LDPE/nanosilika ASP divariasikan (100/0, 98/2, 96/4, 94/6 dan 92/8 % berat) dan dilakukan uji sifat mekanis berupa kuat tarik, perpanjangan putus dan modulus elastisitas menggunakan standar ASTM D638. Diperoleh hasil uji mekanis dengan nilai kuat tarik optimum pada komposisi (96/4 %berat) sebesar 10,53 MPa, nilai perpanjangan putus yang optimum pada komposisi (100/0% berat) sebesar 201,5%, dan nilai modulus elastisitas optimum pada komposisi (96/4 % berat) sebesar 226,57 MPa. Penambahan nanosilika ASP sebagai filler terbukti mampu meningkatkan sifat mekanik termoplastik LDPE pada nilai kuat tarik dan modulus elastisitas dibandingkan dengan LDPE tanpa bahan pengisi (100/0 %berat).

Kata kunci: Kopresipitasi, LDPE, Nanosilika ASP, Uji Mekanik, XRD



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1. Introduction

Low Low-density polyethylene (LDPE) is a popular thermoplastic with high durability and a wide range of applications; LDPE excels because it can perform optimally in extreme environments with low temperatures and chemicals, making it ideal for various purposes. Despite its many benefits, LDPE is derived from petroleum, and its decomposition process is slow. One way to make synthetic polyethylene more biodegradable naturally is to combine it with natural polymers, where LDPE acts as a base material and natural polymers are added to improve the material's resistance [1]. The effectiveness of fillers has been proven through various studies in increasing mechanical strength and heat resistance and reducing material costs. In addition, the addition of fillers can also promote sustainability by utilizing waste materials. The rice harvesting process produces rice husks as a by-product. These rice husks are often thrown away or burned, which can damage the beauty of the environment and increase greenhouse gas emissions. Rice husk ash is rich in silica, with a content of 87% to 97%, opening up opportunities to use it as a raw material for making nanoparticles. These nanoparticles can then be used as materials to increase resistance in manufacturing nanocomposites [2]. The result of rice husk ash in the form of nano-sized silica is an interesting filler because of its tiny size, so it is a potential material to be used as a filler in various thermoplastic composites.

The field of nanotechnology is currently the prima donna of today's research, driven by its better chemical properties and the physical properties of nanomaterials than materials with larger particle sizes [3]. Nanoparticles with a smaller size are characterized by superior chemical and physical characteristics compared to macro-sized materials. This also impacts increasing the use value of composite materials resulting from material engineering. Nanosilica can be obtained through various methods, one of the most common being the synthesis method, which involves mixing certain materials [4]. Nanosilica synthesis methods are divided into several types, such as coprecipitation, microemulsion, and molding methods. In this study, the researcher chose the coprecipitation method to obtain nanosilica because this method is more cost-effective and the process is easier. The process of this method takes place at low temperatures (70°C), the coprecipitation method also offers easy particle size control and shorter synthesis times. This technique involves the addition of acid (HCl) as a solvent and a base (NaOH) as a precipitate to produce a polymer powder that is nano-sized [5].

Based on this explanation, the researcher will conduct research on the manufacture of thermoplastic LDPE with additional fillers.

2. Methods

2.1. Synthesis of Rice Husk Ash Nanosilica by Coagulation Method

Rice husk ash was calcined in an oven for 5 hours at 60°C and in a furnace at 800°C for 4 hours. The rice husk ash was then processed in a ball mill at a speed of 250 rpm for 1 hour and sifted using a 200-mesh sieve to achieve a size of 74 μm . The calcined rice husk ash was then weighed and dissolved in 2M HCl at a ratio of 1:3. Next, it was stirred and heated using a magnetic stirrer at a speed of 400 rpm and a temperature of 70°C for 1 hour before being filtered. Subsequently, the rice husk ash was dissolved in 2.5M NaOH at a ratio of 1:3, stirred, and heated using a magnetic stirrer at a speed of 400 rpm and a temperature of 70°C for 1 hour, then filtered. The filtered rice husk ash was washed with distilled water repeatedly until a neutral pH (pH 7) was achieved. After reaching a neutral pH, the solution was filtered, and the sediment was dried in an oven at 70°C for 4 hours. The dried material was ground using a mortar, sifted, and characterized by X-ray Diffraction (XRD).

2.2. Preparation of LDPE Thermoplastic with Rice Husk Ash Nanosilica Filler

The manufacture of thermoplastics was done by mixing LDPE and rice husk ash nanosilica in a flask using the reflux method with xylene as a solvent at a temperature of 130°C for 2 hours. The LDPE and rice husk ash nanosilica were weighed according to the composition ratios of 100/0, 98/2, 96/4, 94/6, and 92/8 % by weight. Next, LDPE was placed into a triple-neck flask, xylene was added to dissolve the LDPE, and the mixture was heated while distilling for 2 hours until the LDPE was completely dissolved. The resulting solution was poured into a tray lined with Teflon paper, evaporated, and ground. The mixed material was molded using a hot press according to ASTM D638 Type 4 standard. The molding process took 20 minutes, consisting of 5 minutes for mold heating, 10 minutes for material heating, and 5 minutes for pressing at a pressure of 0.1 MPa and a temperature of 170°C. Table 1 depicts thermoplastic manufacturing composition. The next stage involved conducting mechanical tests, including tensile strength, elongation at break, and modulus of elasticity.

Table 1. Thermoplastic Manufacturing Composition.

Material	Sample A	Sample B	Sample C	Sample D	Sample E
LDPE (weight%)	100	98	96	94	92
nanosilica rice husk silica (weight%)	0	2	4	6	8

3. Result and Discussion

3.1. XRD Analysis of Rice Husk Ash Nanosilica

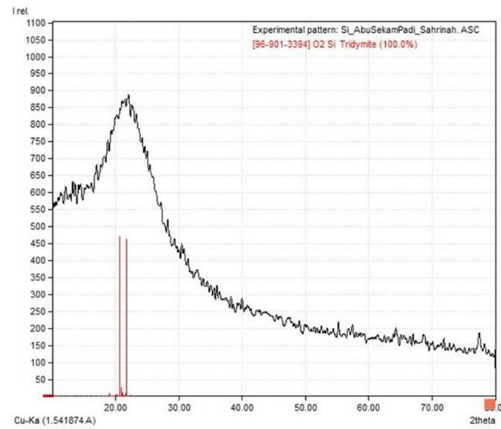


Figure 1. Diffractogram of rice husk ash nanosilica.

Table 2. Rice husk ash nanosilica XRD result data.

Data	SiO ₂
Crystal structure	Triclinic (anortic)
Cell unit	a= 9.9320 Å b= 17.2160 Å c= 81.8640 Å
Intensity	1.50
Density	2.281 g/cm ³

Based on the diffractogram of XRD analysis results given in Figure 1 and Table 2, there is a wide peak, which is at $2\theta = 20.64^\circ$; $2\theta = 21.76^\circ$; $2\theta = 22.50^\circ$ shows that the nanosilica powder from the synthesis of rice husk ash by the coprecipitation method contains SiO₂ (Silicon Dioxide). With the highest peak and the strongest intensity at $2\theta = 21.76^\circ$, it is known that the crystal structure is amorphous, and the silica peaks are identified in the form of tridymite. This result is in accordance with previous research, which stated that amorphous silica is generally in the region of $2\theta = 20-22^\circ$ with a wide and undefined peak [6]. The study [7] obtained an amorphous silica phase with a wide peak at $2\theta = 22.24$. In line with the research conducted by [8], it is also stated that the silica synthesized from rice husk ash has an amorphous structure, and the silica present in plants generally has amorphous properties.

Based on the nanosilica XRD analysis results, the sample crystal size was calculated based on the analysis of the Scherrer method with an average crystal size of around 23.09 nm.

3.2. Analysis of Mechanical Properties of LDPE Thermoplastics

In testing the mechanical properties of LDPE thermoplastics, results of tensile strength, elongation at break, and modulus of elasticity will be obtained. LDPE thermoplastic is mixed with rice husk ash nanosilica filler with variations in composition (100/0, 98/2, 96/4, 94/6, and 92/8 % by weight).

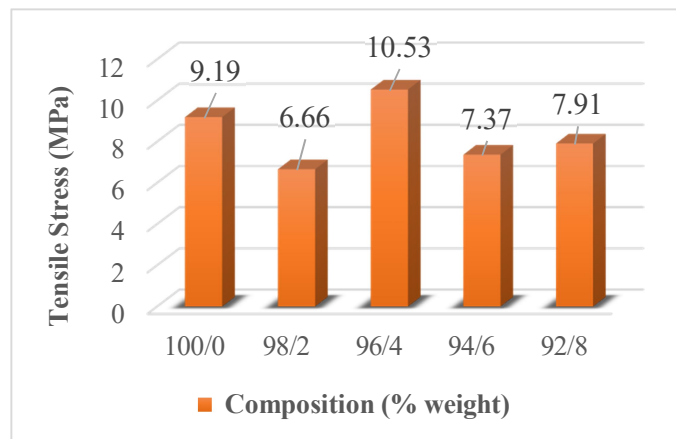


Figure 2. Histogram of tensile strength values vs. composition.

Based on the results of the tensile strength test (Figure 2), the results of the tensile strength ranged from 6.6 to 10.53 MPa. Meanwhile, the standard tensile strength value for LDPE-type plastic is 9.86 MPa [9]. The tensile strength value that meets the standards for LDPE-type plastics is in the composition of LDPE/ rice husk silica nanosilica (96/4% by weight).

The results of this study show a superior tensile strength value compared to the study [10], which uses a mixture of natural zeolite and boiler ash as a thermoplastic filler material LDPE. The tensile strength value obtained in this study reached 9.16 MPa. This is because the natural zeolite in the study received an average size of around 44.46 nm, while in this study, the average size of rice husk ash nanosilica was around 23.09 nm. The increase in tensile strength of LDPE is affected by the interaction between rice husk ash nanosilica and LDPE matrix. The smaller nanosilica particle size results in a more homogeneous mixture, thus increasing the tensile strength of LDPE. This is supported by research [11], which shows that the smaller the particle size and the more evenly distributed phase dispersion, the higher the tensile strength of the mixture produced.

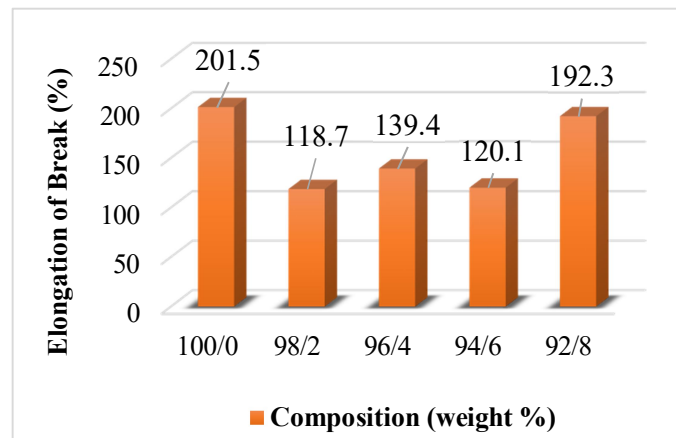


Figure 3. Histogram of elongation values at break vs composition.

As seen in Figure 3, the highest elongation value for LDPE thermoplastic, found at a composition of 100% LDPE, is 201.5%. This elongation value meets the typical range for thermoplastic LDPE, which is characterized by a tensile strength of no less than 10 MPa and an elongation at break of at least 200% [9]. The results of the elongation test at break decreased when rice husk silica nanosilica was added. It was seen that when rice husk silica nanosilica was added to the LDPE matrix, there was a decrease in the elongation value of break in all samples to which rice husk ash nanosilica filler was added.

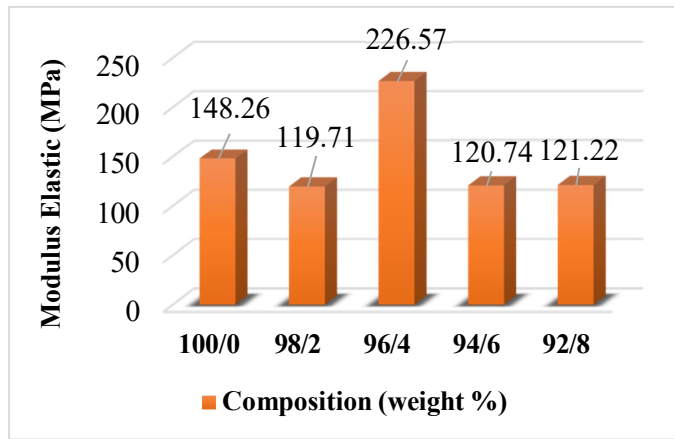


Figure 4. Histogram of modulus of elasticity vs. composition.

Based on the results of the elastic modulus test (Figure 4), it can be seen that the elastic modulus value varies from 119.71 to 226.5 MPa. The highest value of the data was in sample C with LDPE/rice husk ash nanosilica composition (96/4% by weight) of 226.5 MPa. The Elastic Modulus standard of LDPE, according to [12] ranges from 22.0 – 42.1 (psi) or equivalent to 151.7 – 290.3 MPa. So, it can be concluded that only one sample satisfies the composition (96/4 weight%).

The elastic modulus value of LDPE with the addition of fillers in this study was higher than LDPE without fillers (pure LDPE). This is because the silica content in rice husk ash nanosilica can increase hardness and improve physical properties in LDPE [13]. However, the more the composition of rice husk ash nanosilica is added, the more the value of the young modulus decreases. This is directly proportional to the decrease in the sample's tensile strength, which also decreases.

Based on the research conducted [10], the value of the elastic modulus of the largest LDPE was obtained, which was 165.47 MPa. This value is smaller than the modulus of elasticity in this study. This is because the study used a mixture of natural zeolite and boiler ash with an average size of natural zeolite of 44.46 nm and boiler ash of 100.7 nm. The average size is larger than the rice husk silica nanosilica filler used in this study, thus improving the mechanical properties of LDPE at the modulus of elasticity. The smaller size of the filler used as a thermoplastic filler material can increase the stress transfer between the thermoplastic matrix and the reinforcing particles, resulting in a more robust and ductile material [14].

3.3. LDPE Thermoplastic XRF Analysis

Table 3. Elemental content in the composition (96/4 % by weight).

Element	Concentration (% wt)	Line Name	Peak Position (keV)
The	43.47%	Si KA1	1.74
Cl	2.04%	Cl KA1	2.622
K	1.63%	K KA1	3.314
Ca	0.83%	Ca KA1	3.692
P	0.35%	P KA1	2.014
Fe	0.57%	Fe KA1	6.404
Al	0.07%	Al KA1	1.487
Zn	0.07%	Zn KA1	8.639
Mn	0.00%	Mn KA1	5.899
S	0.00%	S KA1	2.307
Mg	0.00%	Mg KA1	1.254
Na	0.00%	Na KA1	1.04

Table 4. Compound content in composition (96/4 % by weight)

Element	Concentration (% wt)	Line Name	Peak Position (keV)
SiO ₂	92.99%	Si KA1	1.74
Cl	2.04%	Cl KA1	2.622
K ₂ O	1.97%	K KA1	3.314
CaO	1.16%	Ca KA1	3.692
P ₂ O ₅	0.81%	P KA1	2.014
Fe ₂ O ₃	0.81%	Fe KA1	6.404
Al ₂ O ₃	0.13%	Al KA1	1.487
ZnO	0.09%	Zn KA1	8.639
MnO	0.00%	Mn KA1	5.899
SO ₃	0.00%	S KA1	2.307
MgO	0.00%	Mg KA1	1.254
Na ₂ O	0.00%	Na KA1	1.04

Based on the results of testing with XRF (Table 3 and Table 4), it was obtained that there were 92.99% SiO compounds on composition (96/4 weight%). This shows that the nanosilica synthesized using the coprecipitation method was successfully carried out, with the most element obtained being Si element at 43.47%. The results obtained show that the silica that has been successfully synthesized has high purity, although there are still other compounds such as Cl, K₂O, CaO, and other compounds with relatively low elements. High-purity silica can be caused by its homogeneous composition, which produces more accurate values in XRF tests [15].

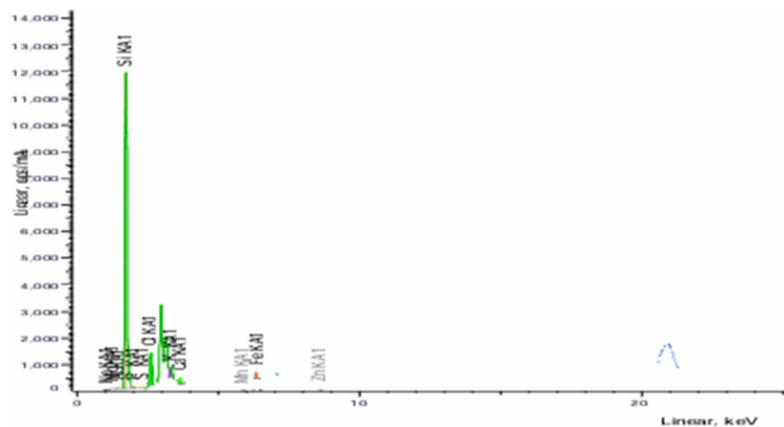


Figure 5. Graph of compound content and elemental content in composition (96/4 weight %).

The size of the crystals from rice husk silica nanosilica produced by the coprecipitation method significantly affected many Si elements detected in XRF. This is in accordance with research conducted by [16] which shows that the smaller the size of the nanosilica crystalline, the higher the amount of Si element detected in the XRF test. This is because the small size of the crystals increases the specific surface area, allowing more Si atoms to be exposed to X-rays.

3.4. Scanning Electron Microscopy (SEM) Analysis

SEM tests are carried out in the sample with the best mechanical property value to see the morphology of LDPE thermoplastics. Namely in composition (100/0, 96/4, and 92/8 weight%).

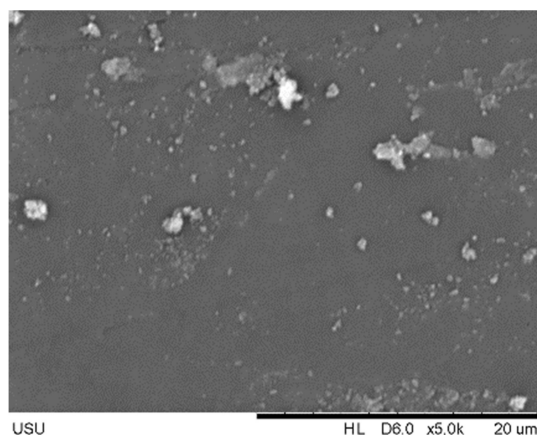


Figure 6. SEM test on composition (100/0 weight%) 5000X magnification.

Figure 6 shows the results of the SEM test on the composition (100/0 % weight) with a magnification of 5000X; it can be seen that there are irregularities in the arrangement of plastic particles and the microstructure of LDPE which looks a little rough, which is caused by the melting process of plastic (granular) using a low reflux temperature so that LDPE is not homogeneously dissolved. There are particles that clump together when printing is carried out. Plastic particles that have clumped together are seen to be non-uniform in size. This agglomeration causes the density of LDPE to be lower due to the presence of particles that do not bind to each other and causes a decrease in the impact strength value or impact due to the agglomeration of nanoparticles of SiO_2 particles in the polymer matrix. As a result, the polymer matrix does not bind SiO_2 perfectly [16].

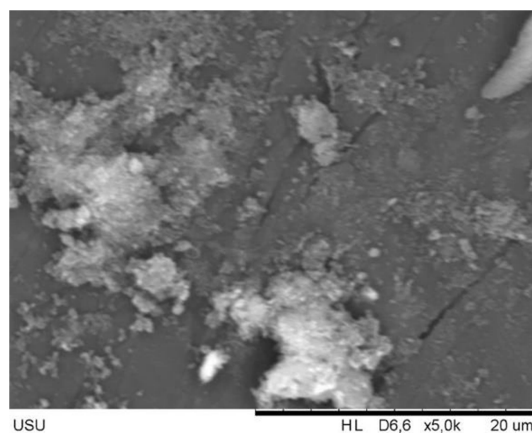


Figure 7. SEM test on composition (96/4 weight%) 5000X magnification.

Figure 7 shows that in the composition of LDPE/Nanosilica rice husk silica (96/4% by weight) with a magnification of 5000X, the nanosilica distribution is uneven. This is due to the mixing between LDPE powder and rice husk silica nanosilica, which produces agglomerates formed due to the nanosilica's non-uniform size. So, it tends to form clumps in the form of large particles or agglomerations [17].

Agglomerates formed on rice husk silica nanosilica were also identified to be scattered but with a distribution that was still random and had gaps. According to research [18], the shape of crystals/particles that are not evenly distributed is caused by the heterogeneous nucleation process due to the presence of foreign particles.

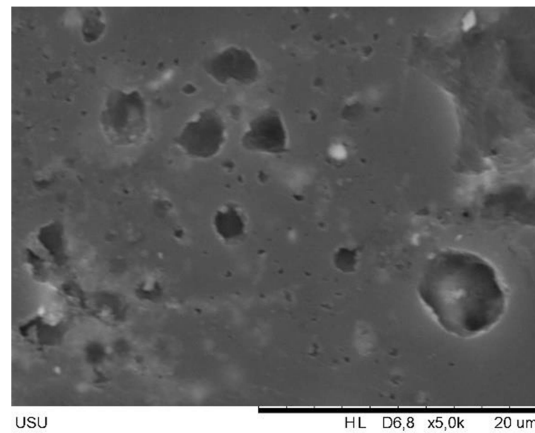


Figure 8. SEM test on composition (92/8 weight%) 5000X magnification.

Figure 8 shows the SEM test results on the composition (92/8weight%) with a magnification of 5000X and large pores in the sample. Meanwhile, the microstructure of RICE HUSK SILICA nanosilica can be seen to be dispersed into the LDPE matrix even though there are still agglomerates of RICE HUSK SILICA nanosilica added. The pores formed in the LDPE matrix are caused by the increase in the addition of RICE HUSK SILICA nanosilica to the LDPE matrix, which causes large agglomeration. According to [19] in his research, silica generally easily forms agglomerates in its application. For the formation of a composite with thermoplastic polymers, silica is difficult to disperse well into the matrix because of the viscosity of the polymer. The higher the viscosity of a polymer, the more difficult it is for silica to be dispersed. The formation of pores is in line with the results of LDPE tensile strength, which is decreasing because large pores also form large cavities

4. Conclusion

The optimum composition of low-density polyethylene (LDPE) thermoplastic material with rice husk ash nanosilica filler synthesized using the coprecipitation method is found in the composition (96/4% by weight). Because of the composition (96/4 weight%), the optimal mechanical test value was obtained over LDPE without filler: tensile strength of 10.53 MPa and elastic modulus of 226.57 MPa. The mechanical properties (tensile test, elongation at break, and modulus of elasticity) of thermoplastic material low-density polyethylene (LDPE) with rice husk ash nanosilica filler synthesized using the coprecipitation method were obtained through the tests carried out, namely the highest tensile strength value in the composition of LDPE/rice husk nanosilica (96/4% by weight) of 10.53 MPa, the highest elongation value at break in the composition of LDPE/ rice husk silica nanosilica (100/0% by weight) of 201.5% and the highest modulus of elasticity value in the composition of LDPE/rice husk silica nanosilica (96/4% by weight) is 226.57 MPa.

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