



## ANALYSIS OF MECHANICAL PROPERTIES OF NATURAL RUBBER COMPONENTS WITH A MIXTURE OF ABKS-TiO<sub>2</sub> WITH PEG-6000

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### ABSTRACT

Research has been conducted on the mixing of Oil Palm Boiler Ash (ABKS) and Titanium Dioxide (TiO<sub>2</sub>) as a filler for rubber compounds to improve the mechanical properties of rubber compounds which include tensile strength, elongation at break, modulus of elasticity, hardness and swelling tests. The filler used to make rubber compounds is ABKS nanoparticles and TiO<sub>2</sub> with variations in composition (0, 2, 4, 6, 8) % by weight. The manufacture of rubber compounds is done by grinding SIR-20 rubber and then mixing it with chemicals, ABKS nanoparticles and TiO<sub>2</sub> in an open mill. After that, the rubber was moulded with a size of 15 cm long x 15 cm wide x 0.18 cm thick at 45° C according to SNI 18 - 6478 - 2000 standard. The best composition of the mechanical properties of rubber compounds using ABKS-TiO<sub>2</sub> fillers for tensile tests obtained tensile strength values at 6% filler composition which is 2.3 Mpa. Elongation at break with the composition of 4, 6 and 8% filler material is 90%. The hardness test obtained the hardness value in the composition of 8% filler material is 57 Shore A. As a result of the compound swelling test, the development value of the 4% filler composition compound was obtained, which experienced a development of 98.73% for 72 hours.

**Keywords:** Palm Oil Boiler Ash, Rubber Compound, Hardness, Tensile Test, Swelling

### INTRODUCTION

The development of science and technology in the production of composite materials is becoming increasingly interesting to study. Composites are materials made from two or more substances, such as a mixture of rubber and rubber, rubber and plastic, or plastic with plastic. The use of a mixture of two or more materials can enhance the properties of the resulting products (Mayasari, 2019). The production and consumption of natural rubber are far below the use of synthetic rubber; however, in reality, natural rubber cannot be

replaced by synthetic rubber. The advantages of natural rubber are difficult to match by synthetic rubber. Nevertheless, synthetic rubber has advantages such as resistance to various chemicals and tends to maintain a stable price (Anti, 2019).

Natural rubber compounds with various filler materials include research (Sidebang, 2018) which aims to determine the mechanical property values of rubber compounds. The characteristics testing of the rubber compound includes hardness, tensile strength, and elongation at break using variations of palm oil

boiler ash (ABKS) nanoparticles filler (0%, 2%, 4%, 6%, 8%) by weight. The analysis results in this study show that the mechanical properties of hardness and tensile strength increase, while elongation at break decreases with the increasing variations of palm oil boiler ash filler. Research on ABKS as a filler has been conducted (Tambunan, 2017), which found that ABKS filler can enhance the hardness, tensile strength, elastic modulus, and tear strength of rubber compounds. However, it has the disadvantage of decreased elongation at break and less stable mechanical properties.

Based on the research by Harahap and Nurdin Bukit (2019), the process of producing ABKS nanoparticles using the coprecipitation method and the ball mill method resulted in ABKS nanoparticles with a length of 56.31 nm. In the study by Siringo-ringo (2021), the addition of PEG-6000 to Empty Palm Fruit Bunch Ash (ATKKS) was varied (1:3), (1:4), and (1:5) to determine the best size produced with the addition of PEG-6000, resulting in sizes of 16.31 nm for the (1:3) variation, 50.57 nm for the (1:4) variation, and 46.08 nm for the (1:5) variation. The best/smallest size was obtained for the (1:3) variation.

Titanium dioxide (TiO<sub>2</sub>) is a semiconductor material used externally in various applications such as catalysts, solar cells, and cosmetics (Ahsan, 2022). TiO<sub>2</sub> is an oxide mineral with many advantages, including good optical properties, excellent photocatalytic activity, superhydrophilicity, environmental friendliness, and high mechanical stability (Akifah, 2017). According to research by Nasrudin (2019), the high mechanical stability of TiO<sub>2</sub> can enhance the mechanical properties of rubber compounds by increasing elongation at break and hardness of the rubber compound. The photocatalytic properties of TiO<sub>2</sub> can also be used to make rubber cleaner and whiter. However, the use of TiO<sub>2</sub> as a filler reduces the tear strength of rubber compounds.

In the studies by Tambunan (2017) and Sidebang (2018), the use of ABKS as a filler can improve the hardness, tensile strength, elastic modulus, and tear strength of rubber

compounds. However, there is a decrease in the mechanical property of elongation at break, resulting in compounds with darker colors and less stable mechanical properties. In the research by Nasrudin (2019), TiO<sub>2</sub> was used as a filler in rubber compounds to enhance the mechanical properties of elongation at break and hardness while also making the rubber cleaner and whiter. However, there was a decrease in the mechanical property of tear strength in the rubber compound. Based on the challenges in trying to develop and produce quality mechanical properties, the researchers are interested in conducting research related to rubber composite nanocomposites with ABKS and TiO<sub>2</sub> fillers.

## RESEARCH METHOD

The research process for the processing and testing of samples was conducted at the Physics Laboratory of Medan State University and the Rubber Laboratory in Bogor in the production of rubber compounds.

The equipment used includes a furnace, XRD, SEM, durometer, rheometer, and the materials are ABKS, SIR 20 rubber, TiCl<sub>4</sub>, PEG-6000, wax, ZnO, sulfur, IPPD, TMTD, and MBTS.

### Production of ABKS nanoparticles using the coprecipitation method

1. ABKS nanoparticles are weighed using a digital scale, amounting to 20 grams.
2. ABKS is placed into a beaker and then dissolved with 40 ml of 2M HCl. It is then stirred and heated with a magnetic stirrer at a temperature of 70°C for 1 hour.
3. PEG-6000 is added to the solution at a ratio of (1:3) and then stirred using a magnetic stirrer at a temperature of 70°C for 1 hour.
4. 2.5 M NaOH is added to the mixture of PEG-6000 with the ABKS solution while stirring using a magnetic stirrer.
5. it is dried in an oven at a temperature of 70°C for 4 hours.

### Production of TiO<sub>2</sub> nanoparticles using the sol-gel method

1. The materials are weighed using a digital scale.
2. Measure 20 ml of TiCl<sub>4</sub>, 100 ml of NH<sub>4</sub>OH, and 100 ml of distilled water using a graduated cylinder.
3. The TiCl<sub>4</sub> solution is mixed with distilled water in a beaker and stirred for 2 hours using a magnetic stirrer.
4. The NH<sub>4</sub>OH solution is gradually added to the solution while stirring until the solution turns white over a period of 4 hours.
5. The precipitate is dried in an oven at a temperature of 70°C for 24 hours and then calcined at a temperature of 900°C for a duration of 2 hours.

### Production of rubber compounds

1. Weigh the rubber compound materials, which include SIR-20 rubber, zinc oxide (ZnO), stearic acid, ABKS, carbon black, wax, IPPD, TMTD, and MBTS, using a digital scale, while varying the ABKS filler and TiO<sub>2</sub> filler.
2. After the materials are weighed, SIR-20 rubber is placed into the roll mill machine and then milled until the rubber is completely dense.
3. During the rubber milling process, the materials are gradually added one by one into the rheometer.

Table 1. The composition of the compound formula mixture in phr (per hundred rubber) with ABKS PEG-6000 and TiO<sub>2</sub> PEG-6000 fillers

No	Bahan	Formula kompon karet (phr)				
		S <sub>0</sub>	S <sub>1</sub>	S <sub>2</sub>	S <sub>3</sub>	S <sub>4</sub>
1	Karet SIR 20	100	98	96	94	92
2	Wax	1,5	1,5	1,5	1,5	1,5
3	Filler 50/50 ABKS PEG-6000 TiO <sub>2</sub> PEG-6000	0	1	2	3	4
		0	1	2	3	4
4	ZnO	5	5	5	5	5
5	SA	2	2	2	2	2
6	Sulfur	3	3	3	3	3
7	IPPD	2	2	2	2	2
8	TMTD	1,5	1,5	1,5	1,5	1,5
9	MBTS	2,5	2,5	2,5	2,5	2,5

Table 2. The composition of the compound formula mixture in grams

No	Bahan	Formula kompon karet (gram)				
		S <sub>0</sub>	S <sub>1</sub>	S <sub>2</sub>	S <sub>3</sub>	S <sub>4</sub>
1	Karet SIR 20	130	127,4	124,8	122,2	119,6
2	Wax	1,95	1,95	1,95	1,95	1,95
3	Filler 50/50 ABKS PEG-6000 TiO <sub>2</sub> PEG-6000	0	1,3	2,6	3,9	5,2
		0	1,3	2,6	3,9	5,2
4	ZnO	6,5	6,5	6,5	6,5	6,5
5	SA	2,6	2,6	2,6	2,6	2,6
6	Sulfur	3,9	3,9	3,9	3,9	3,9
7	IPPD	2,6	2,6	2,6	2,6	2,6
8	TMTD	1,95	1,95	1,95	1,95	1,95
9	MBTS	3,25	3,25	3,25	3,25	3,25

## RESULT AND DUSCUSSION

### XRD analysis of ABKS nanoparticles

Pengujian XRD (X-Ray Diffraction) dilakukan untuk mendapatkan pola difraksi, struktur kristalin dan ukuran partikel dari nanopartikel abu boiler kelapa sawit. XRD yang digunakan adalah Philips tipe JEOL -3530 Shimadzu dengan panjang gelombang Cu-Kα1 = 1.540560 Å = 0.15406 nm. Ukuran kristal sampel dihitung berdasarkan metode Scherrer dari pola difraksi sinar X. Dari plot difraksi XRD, ukuran butir diperoleh dengan menghitung lebar penuh pada tinggi setengah (FWHM) dari puncak difraksi menggunakan metode Scherrer. FWHM diubah menjadi radian dengan mengalikan π/180.

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

The analysis results from Match software version 3 show the highest diffraction pattern peaks at 2θ: 23.24°, 18.97°, and 26.62°, with the maximum peak occurring at an angle of 2θ = 23.24° and a dhkl spacing of [1 0 1] at 13.0629 Å. The X-ray diffraction pattern of ABKS nanoparticles exhibits a quartz (SiO<sub>2</sub>) phase and trigonal crystal structure with crystal plane orientations of [1 0 1] and [2 0 0], and lattice parameters of a = b = 18.36000 Å and c = 5.14700 Å, with a density of 1.96200 g/cm<sup>3</sup>. Using the Scherrer equation, the particle size of ABKS after the ballmilling and coprecipitation methods was determined to

have an average particle size of 19.00539 nm. The peak intensity analysis results are shown in Figure 1.

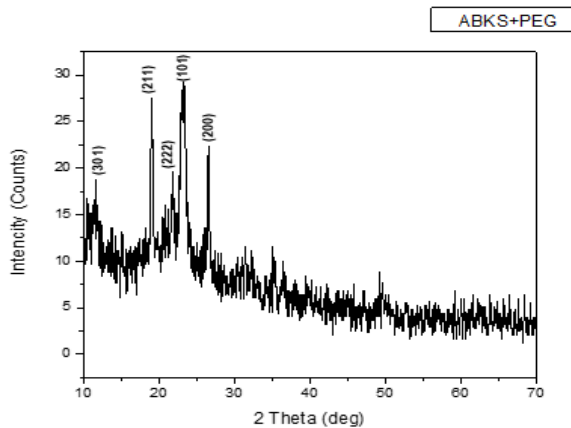


Figure 1. XRD diffraction pattern of ABKS nanoparticles

In the image above, the highest peaks can be observed at  $2\theta = 25.46^\circ$ ,  $48.25^\circ$ , and  $27.52^\circ$ , with the maximum peak at an angle of  $2\theta = 25.46^\circ$  and  $d_{hkl}$  of [1 1 0] with a spacing of 3.2456 Å. The X-ray diffraction pattern of TiO<sub>2</sub> nanoparticles shows a rutile (TiO<sub>2</sub>) phase. The X-ray diffraction pattern of TiO<sub>2</sub> nanoparticles has a tetragonal system with lattice parameters  $a = 4.59000$  Å,  $c = 2.96000$  Å, and a density of 4.25400 g/cm<sup>3</sup>. The particle size of TiO<sub>2</sub> with PEG-6000 obtained from XRD using the Debye-Scherrer method is 20.70 nm.

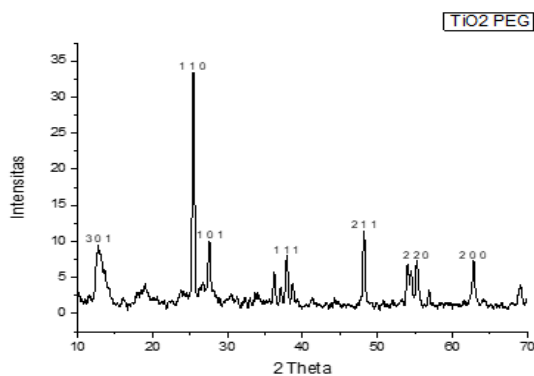


Figure 2. XRD Diffraction Pattern of TiO<sub>2</sub> Nanoparticles

### Mechanical Property Analysis of Natural Rubber Compound Using ABKS-TiO<sub>2</sub> Nanoparticle Filler

#### 1. Tensile strength

The tensile strength of the rubber compound with a 6% mixture of ABKS-TiO<sub>2</sub> nanoparticle filler increased compared to the

tensile strength of the rubber compound without filler (0% ABKS-TiO<sub>2</sub>). The tensile strength values at 0%, 2%, 4%, and 8% are 1.8 MPa, 1.9 MPa, 2.2 MPa, and 2.0 MPa, respectively. The addition of 6% ABKS-TiO<sub>2</sub> nanoparticles enhances tensile strength by increasing covalent and hydrogen bonding with the OH groups and oxygen from the carboxyl groups, which in turn strengthens the interaction between the filler and the natural rubber matrix (Ginting, 2015). However, the tensile strength decreases when 8% ABKS-TiO<sub>2</sub> filler is added. This is due to the increasing silica content, which leads to a reduction in tensile strength (Tambunan, 2017).

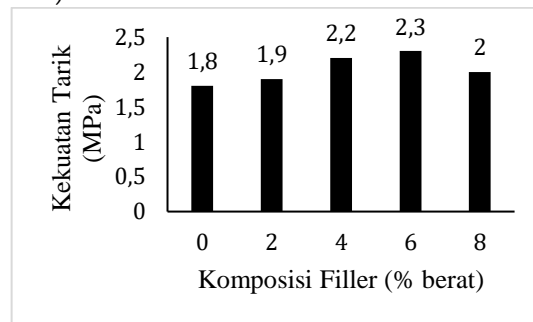


Figure 3. The relationship between the tensile strength of rubber compound nanocomposites and the variation in % weight of the ABKS-TiO<sub>2</sub> nanoparticle filler

#### 2. Elongation at break

The elongation at break value is inversely related to the tensile strength (Nanda, 2014). The results of the study indicate that the addition of more than 4% of the ABKS-TiO<sub>2</sub> nanoparticle filler can increase the elongation at break value of the rubber compound compared to the addition of less than 2% filler and no filler at all.

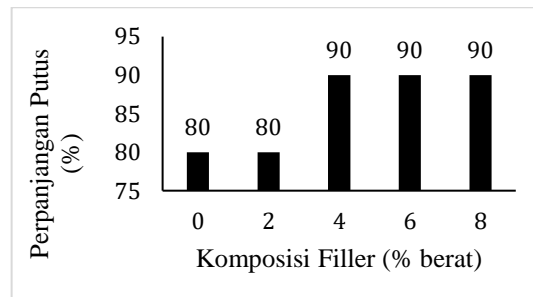


Figure 4. The relationship between elongation at break and the composition of ABKS-TiO<sub>2</sub> nanoparticles

#### 3. Elastic modulus

The 100% modulus of elasticity increased when ABKS-TiO<sub>2</sub> filler was added at 2%, 4%, and 6%, but there was a decrease when the filler was increased to 8%. The highest 100% modulus of elasticity was observed in the rubber compound with 6% ABKS-TiO<sub>2</sub> filler, which was 25 MPa. In the rubber compound with ABKS-TiO<sub>2</sub> filler, an 8% variation in filler composition resulted in a decrease to the same value as the rubber compound without filler, which is 22 MPa. This is due to the presence of silica and oxide minerals, which reduce the density of cross-linking and lead to a decrease in elongation at break, resulting in an increased modulus of elasticity, consistent with the findings of Tambunan (2017).

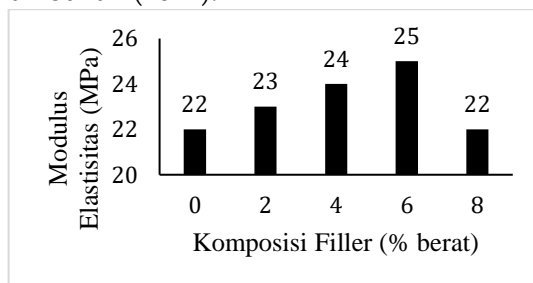


Figure 5. The relationship between modulus and the composition of ABKS-TiO<sub>2</sub> nanoparticles

#### 4. Hardness

The hardness value of the rubber compound with filler increased compared to the hardness value of the rubber compound without filler. The highest hardness value was found in sample 5, which was the rubber compound with 8% ABKS-TiO<sub>2</sub> nanoparticle filler, measuring 57 Shore A. The lowest hardness values were in sample 1 (the rubber compound without filler) and sample 2 (the rubber compound with 2% ABKS-TiO<sub>2</sub> nanoparticle filler), both measuring 53 Shore A.

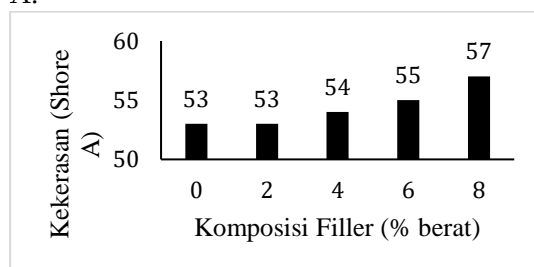


Figure 6. The relationship between hardness and the composition of ABKS-TiO<sub>2</sub> nanoparticles

#### 5. Swelling

The swelling value of the rubber compound without filler is better compared to the rubber compound with filler. The highest swelling value is found in sample 1, which is the rubber compound without filler, with a swelling value of 99.80%. The lowest swelling value is in sample 4, which contains 4% filler, with a swelling value of 98.73%.

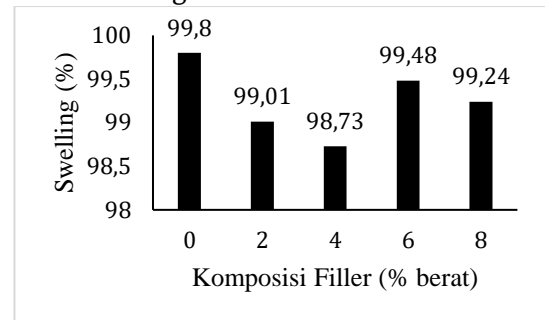


Figure 7. The relationship between swelling and the composition of ABKS-TiO<sub>2</sub> nanoparticles

### CONCLUSION AND SUGGESTION

1. The characterization results of XRD and SEM for ABKS nanoparticles produced by ball milling and coprecipitation show that palm oil boiler ash particles have a particle size of 19.00 nm with a quartz crystal shape and trigonal crystal structure. The XRD and SEM characterization of TiO<sub>2</sub> nanoparticles conducted using the ball milling and sol-gel methods indicates that TiO<sub>2</sub> can be classified as nanoparticles due to its particle size of 20.70 nm with a rutile crystal type and tetragonal crystal structure.
2. The mechanical properties of the rubber compound with variations in the composition of ABKS-TiO<sub>2</sub> filler (0%, 2%, 4%, 6%, 8%) resulted in the best mechanical properties, with the highest tensile strength at a 6% filler composition of 2.3 MPa, elongation at break at filler compositions of 4%, 6%, and 8% of 90%, elastic modulus at a 6% filler composition of 2 MPa, hardness at an 8% filler composition of 57 Shore A, and swelling at a 4% filler composition of 98.73%. From the research results, it can be concluded that the influence of ABKS-TiO<sub>2</sub> filler on

the mechanical properties of natural rubber compounds can enhance the hardness, tensile strength, elongation at break, elastic modulus, and swelling of the rubber compound. According to SNI 16-2623-2002, it is concluded that the rubber compound can be used for the production of rubber gloves.

TiO<sub>2</sub> and ABKS nanoparticles should be coated before conducting SEM and XRD tests to achieve more significant results.

## REFERENCE

- Ahsan, S., Diniyah, R., Firmana, M.F. (2022). "Sifat Mekanik dan Termal PLA dengan Filler TiO<sub>2</sub> dan ZnO." *Jurnal Teknologi dan Manajemen*, 20(1), 27-32
- Akifah, N., Subaer, Muris. (2017). "Pengaruh Penambahan Nano - TiO<sub>2</sub> terhadap Sifat Mekanik dan Karakterisasi Mikro Komposit sebagai Material Self Cleaning." *Jurnal Sains dan Pendidikan Fisika*, 13(1), 282 – 286
- Anti, J. (2019) 'Pengaruh Campuran Abu Tandan Kosong Kelapa Sawit (Atkks) Dan Carbon Black Terhadap Sifat Mekanik Kompon Karet', *Jurnal Einstein*, 7, pp. 22–29.
- Bukit, N., (2011), Pengolahan Zeolit Alam Sebagai Bahan Pengisi Nano Komposit Polipropilena dan Karet Alam SIR-20 dengan Kompatibeliser Anhidrida Maleat-Grafted-Polipropilena, Disertasi, FMIPA, USU, Medan.
- Bukit, N., Frida, E., Ginting, E.M. (2016), Nanopartikel Fe<sub>3</sub>O<sub>4</sub> sebagai Bahan Pengisi Nanokomposit Termoplastik HDPE. Medan : Unimed Press
- Bukit, N., Ginting, E.M., Hutagalung, E.A., Sidebang, E., Frida, E., Bukit, B.F. (2019). "Preparation and Characterization of Oil Palm Ash from Boiler to Nanoparticle." *Reviews on Advanced Materials Science*, 58, 195-200
- Bukit, N., Ginting, E.M., Frida, E., Bukit, B.F. (2021). "Physical Analysis of TiO<sub>2</sub> and Bentonite Nanocomposite as Absorbent Materials." *Reviews on Advanced Materials Science*, 60, 912-920
- Frida E. Bukit, N., Ginting, E.V., Bukit, B.F. (2019) The effect of carbon black composition in natural rubber compound, *Case Studies in Thermal Engineering*, 16 100566
- Ginting, E. M., Wirjosentono, ., Bukit, N., dan Agusnar, H., (2015), Pengolahan dan Karakterisasi Abu Boiler Kelapa Sawit Sebagai Bahan Pengisi Termoplastik HDPE, *majalah polimer indonesia*, 18(1) :26-32.
- Ginting, E.M., Padang, M.M. (2016). "Analisis Sifat Mekanis dan Struktur Nanokomposit Abu Sekam Padi sebagai Filler Termoplastik HDPE." *Jurnal Einstein*, 4(2), 42 – 46
- Ginting, E.M., Bukit, N. (2016). Sifat Mekanis Nanokomposit Termoplastik HDPE dengan Beberapa Bahan Pengisi, Medan : Unimed Press
- Ginting, E.M., Bukit, N., Frida, E., Bukit, B.F. (2020). "Microstructure and Thermal Properties of Natural Rubber Compound with Palm Oil Boilers Ash for Nanoparticle Filler." *Case Studies in Thermal Engineering*, 17, 100575
- Harahap, L. R. (2019) 'Pengaruh Campuran Abu Boiler Kelapa Sawit (ABKS) dan Carbon Black Terhadap Sifat Mekanik Kompon Karet', *Jurnal Einstein*, 7, pp. 30–36.
- Mayasari, H. E. (2019) 'Pengaruh Jenis Bahan Pengisi Terhadap Karakteristik Pematangan Kompon Kloroprena / Karet Alam', *Jurnal Teknologi Proses Dan Inovasi Industri*, 4(October). doi: 10.36048/jtpii.v4i1.5154.
- Nanda, H N., Bahrudin., dan Fadli, Ahmad., (2014), Pengaruh Maleated Natural Rubber Terhadap Morfologi dan Sifat Thermoset Rubber Dengan Filler Abu Sawit – Carbon Black, *JOM FTEKNIK*, 1(2) : 1-13.
- Sidebang, E. (2018) 'Analisis Sifat Mekanik Kompon Karet', *Jurnal Einstein*, 6, pp. 45–50.
- Siringo-ringo, M.J. (2021). Pengaruh Campuran Nanopartikel Abu Tandan Kosong

Kelapa Sawit (ATKKS) dan PEG-6000 terhadap Termoplastik LDPE. Skripsi, Matematika dan Ilmu Pengetahuan Alam, Universitas Negeri Medan, Medan

Tambunan, N.M. (2017). Pembuatan dan Karakterisasi Kompon Karet Dengan Filler Nanopartikel Abu Boiler Kelapa Sawit dan Carbon Black. Skripsi, Matematika dan Ilmu Pengetahuan Alam, Universitas Negeri Medan, Medan