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Synthesis of Alkyd Resin Through Alcoholysis – Polyesterification Process from Used Cooking Oil Pretreatment Using Alkaline Activated Natural Bayah Zeolite

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ARTICLE HISTORY	ABSTRACT
Received 29 November 2022 Received in revised form 8 December 2022 Accepted 8 December 2022 Available online 9 December 2022	Waste cooking oil can be treated by an adsorption process using Bayah natural zeolite as raw material for alkyd resin synthesis. This study aimed to determine the optimum conditions for the equivalent ratio of OH:COOH and the temperature of the alkyd resin synthesis. The research was carried out by preparing used cooking oil and Bayah natural zeolite (BNZ), adsorption process, and alkyd resin synthesis through the alcoholysis-polyesterification process. The optimum addition of Bayah natural zeolite- activated NaOH 0.75 N in pretreatment waste cooking oil is 30% weight in terms of color, density, viscosity, and functional group characteristics of the resulting alkyd resin. The characteristics of the alkyd resin product at optimum conditions obtained light brown color with an OH: COOH equivalent ratio of 1.4, and the alcoholysis and esterification temperature at 190 oC with a density value is 1.112 g/cm3, FFA value is 0.1528%, viscosity value is 4808 cP. The ester functional group at wavelengths 1267 cm-1 and 1722 cm-1, the 1H NMR spectrum of the ester group at the peak of 3.5 ppm, and a functional group of 7.6 ppm.
	Keywords : Alcoholysis-Polyesterification, Alkyd resin, Waste Cooking oil, Zeolite

1. INTRODUCTION

Used cooking oil is defined as oil that has been used for frying. Used cooking oil derived from vegetable oil waste can be used as a raw material for synthesizing alkyd resins. Oils derived from plants or vegetable oils are used as modified alkyd resin substituents because they are considered safer, non-toxic, and biodegradable by having properties as alkyd resins similar to petroleum derivatives (Haryono et al., 2005)..

Alkyd resin is an additive that functions as a binder in paint, coating, plastic, adhesive, and other industries. Alkyd resins are widely used in the anti-corrosion coating industry. Alkyd-based coatings are renowned for their good corrosion protection, high gloss, and ease of application, even on poor surfaces (Gan & Tan, 2001).

Processing efforts are rife by reusing used cooking oil into various products. There are several innovative applications for using used cooking oil as raw materials, including plasticizers, synthesis gas, and biosolvents for pollutants (Mannu et al., 2019). In addition, oil is explored more deeply as an energy source using pyrolysis, hydrogen gas production, and transesterification (biodiesel) (Panadare & Rathod, 2015). Used cooking oil can also be used as a component of fermentation media and raw material for products with economic value, including grease preparation, biolubricant production, polyurethane products, animal feed, alkyd resin manufacture, and soap manufacture. The oil refining process is carried out by mixing the oil with a small amount of adsorbent. The adsorbents commonly used in the oil bleaching process consist of bleaching earth, bleaching carbon, and fiber. The working mechanism of the adsorption process is by absorbing colloidal suspension and the results of oil degradation (e.g., peroxide) on the surface of the adsorbent (Kurniawan & Saputra, 2011).

The use of zeolite has been carried out (Silvianti et al., 2018) to process used cooking oil into raw materials for making alkyd resins, but the zeolite used is not Bayah's natural zeolite which is activated by bases. Using activated zeolite can increase the absorption of zeolite and remove impurities on the surface. The application of technology in this study is based on the processing used cooking oil into alkyd resins by utilizing alkalineactivated natural zeolite adsorbents to reduce the value of free fatty acids and produce different quality oils as raw materials for making alkyd.

Alkyd resins are polyester compounds formulated from modified unsaturated fatty acids, which are compositions of natural oils with dibasic acids and polyols (Heriyanto et al., 2013). The properties and proportions of these components control the properties of the resin. Esterification is the resulting reaction between a carboxylic acid and an alcohol. The acid used can be a mono-base or more, and the alcohol used can be a mono-hydroxy alcohol or more. If a dibasic acid or more is used in esterification, and the alcohol used is dihydroxy alcohol or more, a polyester compound with a high molecular weight is obtained. Making alkyd resins is a polyesterification reaction between fatty acids (corn oil), polyols (glycerol), and dibasic acids (dibasic acid, phthalic anhydride) (Heriyanto et al., 2013).

The process of making alkyd resin occurs through the esterification process. The initial process, namely the formation of monoglycerides, results from mixing vegetable oil and polyols heated at high temperatures with a catalyst such as lead, sodium, calcium, or zinc to form monoglycerides. The process of forming monoglycerides is also known as alcoholysis. The alcoholysis process is carried out at high temperatures so that the reaction speed will be greater (Elliot, 1993). At the esterification stage, the alkyd resin is a condensation reaction; water is a by-product. Esterification is an equilibrium reaction, so water must be separated. There are two techniques for separating water, namely by the fusion process by adding inert gas, usually nitrogen or carbon dioxide. This phenomenon causes the reaction to avoid interference with oxygen,

CH ₂ COOR	Сн₂он	CH ₇ COOR
CHCOOR + 2	снон <u></u>	3 CHOH
CH ₂ COOR	сн₅он	CH ₂ OH
triglyceride (eg. vegetable oil)	glycerol	monoglycerides

Fig. 1. Alcoholysis Reaction

and water vapor is continuously separated from the process. At the same time, the azeotropic process involves the addition of a hydrocarbon solvent, usually xylene or toluene, to the reaction mixture. A watersolvent azeotropic condition is formed at a certain temperature, which condenses externally. Water and solvent are separated, the solvent will return to the process, and the water will come out as waste or a residual process (Elliott, 1993).

The esterification process is controlled by increasing the viscosity and decreasing the acid number. After reaching the process temperature, samples are sampled from the batch at specified intervals. Measurement of the viscosity of samples containing solvent. The acid number is determined by the titration method with a KOH-alcohol solution (Elliott, 1993). The reseach studied purpose the research studied about the alkyd resins formed when polyhydric alcohols react with polybasic and monobasic acids.



Fig. 2. Polyesterification Reaction

In the reaction, the equilibrium mixture contains triglycerides, diglycerides, and glycerin. Monoglycerides can directly react with phthalic anhydride to get the final product as alkyd resin (polyester). Phthalic anhydride is the most common component found in alkyd resins. The anhydride ring opens to form a monoester at 160 oC. The acid group on the phthalic monoester begins to react at 180 oC (Elliott, 1993)

2. METHODS

2.1 Pretreatment of Waste Cooking Oil

The cooking oil from domestic waste was filtered with filter paper. Meanwhile, the Zeolite Alam Bayah (ZAB) adsorbent, which was activated using 750 ml of 0,75 N NaOH solution, was dried using an oven at 110 °C and calcinated at 400 °C for 2 hours. Then, activated zeolite was added to the oil with a percentage of 0, 15, and 30% weight, heated at 80 °C for 1 hour and filtered again using filter paper to separate the waste zeolite from the oil.

2.2 Alkyd Resin Synthesis

The pretreated oil is mixed with a glycerol and calcium oxide (CaO) catalyst. Then the mixture is heated at 190 °C for 2 hours using a heating mantle. A small number of samples is examined for solubility in methanol as an indicator for monoglyceride formation. The process temperature was lowered to 180 °C. Then, phthalic anhydride was added and dissolved in 10 grams of xylene at temperatures 170, 180, and 190 °C. The process was stopped when the alkyd resin was homogeneous, then the alkyd resin that had formed was cooled to room temperature. The alkyd resin was analyzed for density, viscosity, and FFA and characterized by FT-IR and ^{1H} NMR. Table 1. Shows the synthesis of resin formulations.

Weight (W)					
Ratio	Ratio	Ratio 1.4			
15	18	21			
21.51	18.58	15.77			
12.99	12.92	12.73			
0.5	0.5	0.5			
50 gram					
	Ratio 1.2 15 21.51 12.99	Ratio Ratio 1.2 1.3 15 18 21.51 18.58 12.99 12.92 0.5 0.5			

Table 1. Alkyd Resin Synthesis Formulation

2.3 Analysis and Characterization

2.3.1 Density

Measurement of sample density by measuring the weight of a liquid with a known volume. The determination procedure is to enter the test liquid into the pycnometer up to the test limit. Then weigh the mass of the tool filled with liquid and subtract it from the mass of the tool without liquid so that the mass of the liquid is obtained.

2.3.2 Free Fatty Acids

A total of 10 grams of sample was weighed and put into a 250 ml Erlenmeyer flask. 50 ml of hot-neutral alcohol and 2 ml of phenolphthalein indicator (PP) were added to the sample and titrated using 0.1 N NaOH until a color change from colorless to pink did not disappear for 30 seconds.

2.3.3 Viscosity

Alkyd resin was put into a 100 ml plastic bottle. Then the viscosity was measured using a Brookfield viscometer by installing a spindle and putting it in a bottle containing the sample. The test was carried out at room temperature and pressure with spindle no. 4 special viscous liquid.

2.3.4 FT-IR

Changes in the character of the adsorbent can be determined by testing the FT-IR functional groups using FT IR Spectroscopy-Shimadzu. The functional groups of FT-IR zeolite can be determined by detecting the vibrational absorption of Si-O-Al, Si-O, TiO4, and Si-O-Si at long certain waves.

2.3.5 ¹H NMR

Alkyd resin is put into a 100 ml plastic bottle. 1H NMR testing was carried out to determine the vibration of hydrogen protons in a functional group using 1H NMR Bruker Avance 500 MHz

3. RESULTS AND DISCUSSION

3.1 Alkyd Resin Synthesis Process

In this research, the main raw materials for making alkyd resins are used-cooking oil, glycerol, and phthalic anhydrous. The oil added to the raw material formulation is also included in the short oil because the oil content used is based on the calculation results of 30%, 36%, and 42%. Other raw materials in the manufacture of alkyd resins are catalysts and solvents. The catalysts used include those having elements of alkali, alkaline earth, and alkali metals. In the research of the alkyd resin synthesis process using calcium oxide. This catalyst is used because it is easy to obtain, has high purity, and is a low price compared to other catalysts. In this research, the alkyd resin synthesis process uses xylene solvent. Xylene is one of the ingredients included in organic solvents. The solvent is used and added during the synthesis process to form an azeotrope with water vapor and does not interfere with the synthesis process.

The synthesis method in this study consisted of two stages. In the first step, the oil is reacted with glycerol and calcium oxide catalyst to produce monoglycerides. The presence of these monoglycerides is identified by checking the solubility of the mixture in alcohol. The first stage is complete when the solubility of the mixture in alcohol has occurred. This phenomenon indicates that monoglycerides have been formed. This process is called the alcoholysis process. Then it is reacted with phthalic anhydride to form alkyd resin. During the synthesis process, a magnetic stirrer performed agitation or stirring at a speed of 490 rpm. At this stage, the polyesterification reaction occurs, and the resulting alkyd resin product has the physical characteristics of a transparent brown viscous liquid.

The resulting alkyd resin product shows similar characteristics to (Islam et al., 2014), which produces a bright brown alkyd resin product with a bright yellow palm oil base ingredient using a calcium oxide catalyst. The research conducted (by Paparingan et al., 2019) states that the resulting color changes due to temperature-sensitive compounds that change color due to damage caused by oxidation reactions.

Alkyd resin synthesis is based on used cooking oil at an alcoholysis temperature of 190 oC and polyesterification of 190 oC. Compared to research conducted by (Silvianti et al., 2018), the polyesterification temperature was only carried out at 180 oC, while research (by Javanudin et al., 2011) showed that the best temperature for polyesterification was 190 oC from the synthesis of alkyd resins from oil corn. The characteristic of alkyd resin is that it has a brownish metallic color when exposed to light. The results of alkyd resin with the pretreatment process were better in terms of color quality compared to alkyd resin without pretreatment. which had a darker alkyd resin color. The initial raw material used-cooking oil causes the situation without pretreatment contains compounds from food, such as proteins and pigments, which are damaged during heating. The dark color is due to the presence of oxidative damage. Oxidation reactions occur between oxygen and the double bond of the triglyceride/oil. Compounds resulting from this oxidation reaction result in a darker color of the alkyd resin, while used cooking oil resulting from pretreatment is lighter because pretreatment of used cooking oil using activated zeolite absorbs compounds resulting from oxidation in the oil such as peroxide compounds, as well as compounds resulting from hydrolysis, namely free fatty acids..

3.2 Effect of Temperature and OH:COOH Equivalent Ratio on Alkyd Resin Density

The effect of temperature and the OH:COOH equivalent ratio on the density of alkyd resin is presented in Figure 3.





The results in Figure 3 show that the difference in the OH:COOH equivalent ratio and temperature does not have much effect on density because the difference in density is less than 1%, so the change in density is not significant. Compared with research on the synthesis of alkyd resin from palm oil conducted by Islam in 2014, the density of the resulting alkyd resin has a value of around 0.914-0.950 g/cm3.

The results shown in Figure 4 show a comparison of the densities of alkyd resins with oil raw materials without pretreatment and pretreatment using 15% zeolite and 30% zeolite, alkyd resin formulations with variations in the OH:COOH equivalent ratio showed an

increase in yield density which was not significantly different due to differences in density less than 1%...





When compared with the densities of the raw oil used, which were 0.8976 g/ml, 0.8984 g/ml, and 0.8988 g/ml, respectively, for oil without pretreatment, pretreatment oil with 15% zeolite and 30% zeolite, showed that after being processed into alkyd resin there are differences that can be caused by changes in molecular weight or compound concentration during the synthesis process. The increase in molecular density (density) occurs because the number of molecules increases because the polymer chains are getting longer. These situations happened due to the polymerization reaction rate, which increases with increasing temperature. By the theory of reaction rate increasing temperature speeds up the reaction rate because increasing temperature causes more particle movement fast. The resulting movement will cause the kinetic energy of the particles to increase so that more effective collisions occur. In addition, in the process of using an agitator to improve the mixing of substances. The more the particles move and collide, the more products are produced.

Temperature is directly proportional to the degree of polymerization. The higher the temperature, the greater the degree of polymerization (Jayanudin & Rochmadi, 2011). This phenomenon happens because, at higher temperatures, the speed of the esterification reaction is greater, so the reaction conversion will also be greater. In the manufacture of polymers, the molecular density or density is affected by the number of compound molecules in the mixture. The more polymers or esters formed will affect the product's viscosity..

3.3 Effect of Temperature and OH:COOH Equivalent Ratio on Alkyd Resin FFA

The effect of temperature and the OH:COOH equivalent ratio on FFA is shown in Figure 5.



Fig. 6. Graph of Alkyd Resin Product FFA to Ratio OH:COOH Equivalents of Crude Oil Without Pretreatment Process

The research results in Figure 5 show a decrease in FFA when the equivalent ratio of OH:COOH and temperature increases. The situation occurs due to an increase in triglyceride reactants which will increase the conversion of carboxyl groups so that the content of the COOH group in the alkyd resin product is getting less and causing the FFA value to decrease.

These results are consistent with research conducted by (Nurandini et al., 2018) which shows that the greater the OH:COOH equivalent ratio, the lower the FFA value in alkyd resin. Furthermore, a graph of alkyd resin products to the equivalent ratio of OH:COOH with raw oil without pretreatment and with pretreatment (15% Zeolite and 30% Zeolite) is presented in Figure 6.

The research results in Figure 6. show a comparison of alkyd resin products using raw oil without pretreatment and with pretreatment. The content of FFA in alkyd resin products will relatively decrease with increasing OH:COOH equivalent ratio. Alkyd resin products from oil without pretreatment showed the highest FFA value compared to alkyd resin products pretreated using zeolite. The FFA value of alkyd resin from used cooking oil with 30% zeolite pretreatment showed the lowest result. The smaller the FFA value, the better the quality of the alkyd resin due to the low free fatty acids, which indicate oil damage.

3.4 Effect of Temperature and OH:COOH Equivalent Ratio on Alkyd Resin Viscosity

Viscosity testing is carried out to determine the viscosity of a liquid. The lower the viscosity, the easier it is for a liquid to flow, or the frictional force in the liquid is low, so it is easier to flow. Viscosity testing in this study was carried out using the Brookfield method to determine the fluid's viscosity, and the results are presented in Figure 7.



Fig. 5. Graph of Alkyd Resin Product FFA to Different Ratios of Oil Raw Materials Without Pretreatment and Pretreatment (15% Zeolite and 30% Zeolite)

Figure 7 shows that the greater the temperature and the equivalent ratio of OH:COOH, the greater the viscosity value.



Fig. 7. Graph of Viscosity to Increase Ratio OH:COOH Equivalents of Raw Materials Oil Without Pretreatment

Thus, the more molecules in the resin, the higher the molecular density and the thicker the resin becomes. This study's final alkyd resin viscosity value was greater than the commercial alkyd resin value of 1220 cPa and the synthesized alkyd resin (Uzoh et al., 2018) of 4000 cPa. In this study, alkyd resin was synthesized for up to 4 hours. According to research (Uzoh et al., 2018), during the synthesis process, the viscosity of the alkyd resin increases with increasing time. The longer the synthesis time, the thicker the alkyd resin produced.

Test results for the viscosity of alkyd resin products to the equivalent ratio of OH:COOH with raw oil without pretreatment and with pretreatment.

Zeolite 15% and Zeolite 30% are presented in Figure 8. Figure 8 shows that the viscosity value increases with the increase in the reactants' OH:COOH equivalent ratio, whereas the smaller the FFA value of the oil used, the greater the viscosity value. Alkyd resin from used cooking oil pretreatment using 30% zeolite showed the highest viscosity value while cooking oil without pretreatment had the lowest viscosity value.

The small number of triglycerides causes the low can converted into alkvd resins viscositv because triglycerides undergo hydrolysis. In contrast, the low FFA in oil raw materials affects good oil quality because of low hydrolysis, so the amount of triglycerides converted to alkyd resins is greater and causes an increase in viscosity.

According to (Elliot, 1993) and (Nurandini et al., 2018), the increased viscosity of alkyd resin can also be influenced by the amount of the main oil reactant, which reacts with glycerol and forms monoglycerides, so more monoglycerides will polymerize into esters, in this case, the ratio the OH:COOH equivalent of the reactant affects the polymer concentration.



(a) Oil Raw Materials Without Pretreatment



1500

cm

1000

500



(a) Pretreatment Oil Raw Material with 30% Zeolite

Fig. 9. Graph of Wavelength and Vibration Absorption Peak Functional Groups in Alkyd Resin OH:COOH Equivalent

0

4 P-1(2)

3000

2000

Ratio 1.4

The increase in alkyd resin viscosity can also be influenced by the amount of the main reactant of oil, which reacts with glycerol and forms monoglycerides, so more monoglycerides polymerize into esters, in this case, the equivalent ratio of OH:COOH reactants affects the concentration of the polymer produced.

The viscosity of alkyd resins increases with increasing temperature in the polymerization process. This phenomenon occurs because the higher the temperature, the faster the reaction will increase, so the degree of polymerization will increase. Then, the greater the degree of polymerization, the greater the resulting



Fig. 8. Graph of Alkyd Resin Product Viscosity to Equivalent Ratio of OH:COOH with Oil Raw Materials Without Pretreatment and Pretreatment (15% Zeolite and 30% Zeolite)

polymer product, causing the viscosity of the polymer to increase. This evidence is also supported by research that has been conducted by and (Heriyanto et al., 2013).



Fig. 10. ¹HNMR Alkyd Resin Raw Material Oil Pretreatment with Zeolite on 190 °C

3.5 Effect of Oil Pretreatment on Alkyd Resin Characteristics

Characterization of the content of product compounds in alkyd resins can be determined by analyzing the functional groups of these compounds using FT IR. Tests in this study were carried out on polyesterified alkyd resin products at a temperature of 190 °C with an OH:COOH equivalent ratio of 1.4.

This test was carried out to find out the differences in the products produced qualitatively, the test results of which are presented in Figure 9.

Figure 9. shows a graph of the results of the FT IR test. The graph displays the absorption wavelength spectrum of the molecule, which can be seen in table 2.

	Aikyu Kesilis			
	Alkyd resin Compound Wavelength (cm ⁻¹)			
Functional groups	(a)	(b)	(c)	
OH alcohol	3450.01	3440.73	-	
Aromatic -CH	742.46	742.46	742.46	
C-C	985.44	985.44	985.44	
0=C-O-C ester	1120.43	1120.43	1120.43	
C-O-C ester	1278.57	1278.57	1267.00	
C=C aromatic phthalate	1598.69	1598.69	1598.69	
C=O ester triglycerides	1722.11	1722.11	1722.11	
-CH ₂	2852.19	2852.19	2852.19	

 Table 2. Characterization of Compound Functional Groups in Alkyd Resins

The FT IR functional group analysis results showed the highest absorbance of the C-O-C ester groups from the triglyceride and C=O ester functional groups. The more ester compounds contained, the greater the ester functional groups experienced vibration, thus causing greater absorbance in the FT IR spectrum. The presence of an ester functional group indicates an ester Alkyd resin compound contained in the resin resulting from the reaction of alcoholysis and polyesterification. Apart from that, there are differences in the results of the study. Alkyd resin products from oil without pre-treatment and using 15% zeolite pre-treatment have absorption at a wavelength of 3450 and 3440 cm-1, which shows the vibration of the alcohol functional group, while Alkyd resin from oil using 30% zeolite pre-treatment does not show absorption at the functional group wavelength alcohol, this is due to the alkyd resin without pretreatment. With 15% pre-treatment, there is still glycerol residue, while the alkyd resin from 30% zeolite pretreatment does not have glycerol residue, or glycerol residue is so low that the FT-IR instrument cannot read it. Alkyd resins with no residual glycerol indicate that glycerol has been converted to esters during the synthesis process. The glycerol content in alkyd resins can come from hydrolyzed oil or alcoholysis reactions, which produce low monoglycerides. In Table 3, there are no carboxylic acid functional groups. Carboxylic acid functional groups indicate the presence of free fatty acid compounds. The situation occurs due to the alkyd resin's low value of free fatty acids. The better the oil processing with zeolite, the better the quality of the alkyd resin. The phenomenon appears due to the smaller impurities or compounds that can damage the Alkyd resin product.

Based on the comparison in Figure 8 (a), (b), and (c), the characteristics of the alkyd resin produced from processing used cooking oil using 30% zeolite (P-2) has the highest vibrational absorbance of the functional group, which is equal to 2.25. ¹HNMR test results for alkyd resin raw materials for oil pre-treatment with 30% zeolite Temperature 190 °C

Information for the proton densities of the different sections is shown in Figure 10, with the ¹H NMR spectra of the respective resins shown in Table 3.

Table 3. Data 1H NMR Alkyd Resin				
	^{1H} NMR Alkyd Resin (ppm)			
Functional groups	(a)	(b)	(c)	
CH ₃	0.85	0.85	0.85	
COCH2CH2(C <u>H</u> 2)n	1.3	1.3	1.3	
COCH ₂ C <u>H</u> ₂ (CH ₂) _n	1.5	1.6	1.6	
COC <u>H</u> 2CH2(CH2)n	2.3	2.3	2.1	
C-C <u>H</u> (O)-C	3.5	3.5	3.6	
ArCOOCH2CH(O)C	4.4	4.4	4.4	
CH=C <u>H</u>	5	5	5.4	
Ar(<i>m</i> -)	7.6	7.6	7.6	

Based on the data in Table 4, ^{1H} NMR Alkyd resin was obtained according to research conducted by Islam et al. in 2014, which indicated the presence of protons in unsaturated, aromatic, and ester hydrocarbon functional groups at wavelengths of 5-5.4 ppm, 4 .4 ppm and 7.6 ppm respectively. According to Islam et al., in 2014, the proton for the terminal methyl group of fatty acids was confirmed with a peak of around 0.87-1.02 ppm. The adjacent peak in the 1.25-1.30 ppm range is due to protons from all the $-CH_2$ in the chain. The peak of 3.5 ppm is due to the methylene glycerol proton. The peak at 4.2 - 4.4 ppm is due to the methylene proton of the glycerol molecule and at 5.25 ppm to 5.4 ppm is due to unsaturated carbon. The peak appears at 6.25-6.85 ppm for -CH present in the glycerol molecule. The phenomenon exhibited is due to a phthalic anhydride group, which produces a deshielding effect. Protons for aromatic rings can be described with peaks at 6.8–7.5 ppm.

4. CONCLUSION

Alkyd resins can be synthesized from used cooking oil without processing and by processing using alkaline activated natural zeolite. The best addition of 0.75 N NaOH-activated zeolite in processing used cooking oil into raw material for Alkyd resin is 30% with color, density, viscosity, and functional group characteristics. The OH:COOH equivalent ratio and the most effective temperature during the synthesis process were 1.4 mgek/mgek with a polyesterification temperature of 190 °C, and the characteristics of the Alkyd resin were shown by the absorption of the ester functional group at wavelengths of 1598.69 and 1722.11 cm⁻¹.

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