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Development of insulation oil based on Palm Oil Mill Effluent with nano silica



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Abstract

Various studies and research have been conducted to find alternatives to liquid insulation. One that is considered the most potential is vegetable oil since it has various advantages, including non-toxic, biodegradable, renewable waste products due to reactions in the form of CO2 and water, high flash points, and better thermal characteristics. In this study, Palm Oil Mill Effluent (POME) was used as the raw material for insulation oil with the addition of an additive in the form of nano-silica, which improves the quality of the insulation oil. As for determining the feasibility of insulation oil, characteristic tests were carried out in the form of density, viscosity, moisture content, acid number, pour point, flash point, and breakdown voltage. Based on the results of the tests, it was obtained that the lowest density in pure oil was 0.8757 g / cm³, the lowest viscosity in oil with the addition of 0.13 wt% nano-silica was 4.0248 cSt, and the lowest acid number in pure oil was 0.5797 mgKOH / g. It was also discovered that the pour point value is the same for each sample, the moisture content is 0.05%, the flashpoint is > 104 °C, and the breakdown voltage is \geq 60 kV for each sample. The data show that the insulation oil made from POME has the potential to be used as an alternative to insulation oil.

Keywords:

Guidance; Insulation oil; Nano silica; POME; Vegetable oil;

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INTRODUCTION

Transformers are essential in an electrical system, especially in the transmission and distribution system of the electric power The transformer increases system. and decreases the voltage with the same working frequency in the electrical system. The essential parts of the transformer consist of a core made of plated iron, primary and secondary windings, and insulation. One of the critical factors affecting the transformer's reliability is the insulation system [1].

The transformer has two types of insulation: solid and liquid. Solid insulation lies in the conductor of the coil in the form of mica and paper. Solid insulation is utilised to protect the windings against other windings; thus, flashover does not occur. Liquid insulation in the form of oil is used as a separator between voltage conductors and as a coolant when the transformer is overheated. Insulation oil cools the system by transferring heat to the fins of the transformer. Insulation oil can even extinguish arcs if a spark occurs in the transformer windings [2, 3, 4]. The oil most widely used as liquid insulation is mineral oil. Mineral oil has a reasonable heat transfer rate and can improve its properties after a breakdown [3].

The Ministry of Energy and Mineral Resources of the Republic of Indonesia stated that the petroleum reserves in Indonesia from 2009 to 2016 have consistently decreased [5].

The effort to reduce the use of mineral oil is to find alternatives to liquid insulation using vegetable oils. Moreover, vegetable oil has several advantages compared to mineral oil, namely non-toxic waste products due to reactions in the form of CO2 and water, biodegradable, high flash point, better thermal characteristics, and renewable [6]. Vegetable oils that have been widely researched as alternatives to liquid insulation are virgin coconut oil (VCO), palm oil (CPO), RBDPO, castor oil, and corn oil (Oleum Maydis). One of the alternative sources of transformer oil insulation that many researchers have not seen is Palm Oil Mill Effluent (POME). POME is palm oil wastewater that still contains many dissolved solids, carbohydrates, protein, nitrogen (N), lipids, minerals and other nutrients [7].

The wastewater derived from the palm oil industry might be used after processing [8, 9, 10, 11]. The cooling pond wastewater contains oil and fat of approximately 6000 mg/L and should be extracted [12]. The oil can be separated from cooling pond wastewater by the liquid-liquid extraction method to recover oil and use it as a raw material for making biodiesel through the esterification and transesterification processes [13]. The oil obtained can be further processed to become raw material for transformer insulation oil [14]. Insulation oil is produced from the transesterification process with vegetable oil or animal fat as raw material that is reacted with alcohol compounds such as methanol. The raw material contains triglyceride chains, which can be simplified into methyl esters monoglyceride chains with a catalyst. These methyl ester compounds are known as pure biodiesel or commonly known as Fatty Acid Methyl Esters (FAME). The chemical structure of FAME can be illustrated in Figure 1.



Figure 1. Chemical structure of biodiesel [15]

According to Kusumawardani et al. [14]. POME processing using NaOH and KOH catalysts produced an alternative transformer insulation oil that meets several characteristics of insulation oil. Furthermore, to improve the insulation ability of the catalysed oil, a nanomaterial was added, namely, silica treated by saline, which can dissolve in oil [16, 17, 18, 19]. The results of processing POME using a KOH catalyst with added silica treated by saline are better than processing using NaOH, which is also added silica treated by saline.

It is similar to Wang et al. [1], that adding nano-silica to insulation oil can improve the quality of insulation oil. However, to obtain an insulation oil that complies with the standard characteristics of liquid insulation, as shown in Table 1 and Table 2, intensive research must be conducted to get the most appropriate variety of nanofiller types and ratios in processing POME.

Table 1. Insulation Oil Specifications IEC 60296:2020 (International Electrotechnical Commission, 2020)

No.	Insulation Oil Properties	Unit	Standards				
1.	Density	g/cm ³	≤ 0.879				
2.	Viscosity (40°C)	cSt	≤ 12				
3.	Flash Point	°C	≥ 135				
4.	Pour Point	°C	≤ -30				
5.	Acidity	mgKOH/g	≤ 0.01				
6.	Sulfur Corrosion	-	Non-corrosive				
7.	Breakdown Voltage	kV/2.5 mm	≥ 30				
8.	Dielectric Leakage Factor	-	≤ 0.005				

Table 2. Characteristics And Standard Testing Methods for New Insulation Oil

	Standard				
Properties	ASTM	Other Standards	Limits		
Breakdown Voltage, Mushroom Electrodes (gap = 2,5 mm, R = 36 mm), new insulation oil, kV	-	IEC 60156:2018	≥ 30		
Viscosity, cSt max, 40 °C *20 °C	D - 445	*SPLN 49- 1- 1982	12 *40		
Flash Point, °C, min	D – 92	*IEC 60296	145 *135		
Pour Point, °C, min Density et	D – 97	*ISO 3016	-30 *-40		
Density at Temperature 20 °C, g/ <i>cm</i> 3, max	D – 1298	ISO 3675	0.895		
Water content, ppm, max	D – 153	IEC 733	35		

MATERIAL AND METHOD

The research was conducted at the Laboratory of Energy Engineering and Waste Management, Laboratory of Chemical Separation and Purification Engineering, Department of Chemical Engineering, Universitas Sriwijaya. The breakdown voltage test was carried out at the PT. PLN (Persero) Laboratory at Keramasan Generator Control Unit in Palembang. Testing density, viscosity, moisture content, flash point, and pour point was conducted at the Akamigas Polytechnic Laboratory in Palembang.

The equipment used in this study included an oil sample bottle, 250 ml Erlenmeyer, 100 ml beaker, 500 ml beaker, funnel, burette for calculating acid numbers, 500 ml separating funnel, 1000 ml round bottom boiling flask, 500 ml flat bottom boiling flask, 1000 ml flat boiling flask, separator funnel for the extraction process, magnetic stirrer, three-neck boiling flask, twoneck boiling flask, mantle heater, condenser, scale, oven, automatic oil test set for breakdown test, hoses, pumps and buckets for the process distillation, esterification, of and transesterification.

Materials used in laboratory analysis include samples from the Cooling Pond for insulation oil as raw material, N-Hexan for the extraction process, methanol for esterification and transesterification processes, chloroform, ethanol, and dry potassium hydrogen phthalate. PP indicator was utilised to analyse the acid number values, HCl for the esterification process, KOH for the transesterification process, and nano-silica to improve the quality of insulation oil.

Extraction Process

There were several steps in the extraction process. Prepare 500 ml of palm oil waste liquid and 500 ml of N-Hexane solvent (1:1 ratio)). Mixturing the waste sample and N-Hexane solvent stir for one hour using a magnetic stirrer at 50 rpm. When the stirring process was completed, the waste sample and the solvent mixture were put into a separating flask. The oil and solvent were separated at the top of the oil, and at the bottom, there was water and dirt.

Distillation

This distillation process was carried out to separate the solvent from the oil and water content. The condenser was connected to a boiling flask filled with a mixture of oil and solvent. Firstly, set the temperature of the heater to $105 \,^{\circ}$ C – the boiling point of N-Hexane solvent is 68 °C. The boiling ball was put into the flask to obtain even heating and accelerate the heating process for a complete evaporation process. The

N-Hexane solvent that had been evaporated was collected with Erlenmeyer. The oil from the waste was left in the boiling flask.

Measurement of Acid Levels

This process was to determine the fatty acid content in the oil. The steps were initiated by inserting 0.5 g oil to 250 ml Erlenmeyer. Furthermore, ethanol and chloroform were mixed with as much as 50 ml each with a ratio of 1:1. Indicator PP was dropped while stirring for neutralisation. The titration process used a burette. The alcoholic KOH solution was put into the burette to be titrated while stirring until it turned pink. The pink colour should last for 15 seconds. Record the volume of titrant needed (v/ml). Standardisation of KOH liquid by using 100 mg of dry potassium hydrogen phthalate (KHC8H4O4) and added with 0.5 ml of PP indicator then titrated with alcoholic KOH until pink. To determine the acid number (1) was applied.

acid level =
$$\frac{KOH \times N KOH \times 56,1}{sample \ weight} ml/g$$
(1)

Esterification

This process aims to reduce the free fatty acids in the oil. A 500 ml distilled oil was put into a three-neck flask and then on a magnetic stirrer. The three-neck flask was connected to a condenser (reflux condenser circuit) and then heated on a hot plate until it reached a temperature of 65 °C. The magnetic agitation speed of the stirrer was adjusted; thus, the heat transfer within it was faster. In a different place, 2.5 ml/1000 ml of HCl oil catalyst was mixed with methanol and then stirred evenly. After the temperature of 65 °C was reached, the mixture of HCI and methanol was slowly mixed into a threeneck flask. The three-neck flask was quickly closed with a cork until the batch reaction conditions were obtained. The mixture was refluxed while stirring using a stirrer. The reaction temperature was kept constant at 65 °C for 60 min, and the stirrer's magnetic agitation speed was adjusted. After 60 min, the reaction product in the three-neck flask was put into the separating flask. Then, it was left to stand for ± 60 min. After the settling process was complete, the water and methyl ester products were separated into different containers. The water product and the top layer formed were weighed on the analytical balance. The resulting lower layer (methyl ester) was put in a glass beaker for reuse in the transesterification process.

Transesterification

This process was carried out for esterified oils to meet several requirements for insulation oil, namely low viscosity, high flash point, and low pour point. The results of the esterification stage (lower layer) were inserted into a three-neck flask, and then put on a magnetic stirrer. The three-neck flask was connected to a condenser (reflux condenser circuit). Then, it was heated on a hot plate until it reached 65 °C for ± 60 min. The magnetic agitation speed of the stirrer was adjusted; thus, quicker heat transfer was obtained. In a different place, KOH catalysts of as much as 1.5% and 3% of oil weight were mixed with methanol and then stirred evenly. After a temperature of 65 °C was reached, a mixture of sodium hydroxide and methanol was slowly mixed into a three-neck flask. Then, the threeneck flask was closed tightly with a cork until the batch reaction conditions were obtained. The mixture was refluxed while stirring using a stirrer. The reaction temperature was kept constant at 65 °C for 60 min, and the magnetic agitation speed of the stirrer was adjusted. After 60 min, the reaction product in the three-neck flask was put into the separating funnel. Then, let stand for ± 60 min.

After settling, the crude methyl ester and glycerol products were separated into different containers. Crude methyl esters and glycerol formed were weighed on an analytical balance. The crude methyl ester was put back into the separating funnel to carry out the washing process. Clean water was heated to 55 °C on the hotplate. After heating, the water was put into a separating funnel and left until two layers were formed (the water layer at the bottom). The washing water in the lower layer was removed, and the upper part oil was washed. This washing was carried out until the washing water became clear. The washing product in the form of methyl ester was removed into a breaker glass container and heated at a temperature of 110 °C until all water was evaporated while stirring. After ± 60 min, the washing product was cooled to room temperature. After cooling, the methyl esters were weighed.

Addition of Nano-silica

In blending the oil from POME processing with nano-silica, 400 ml of POME oil was carried out by adding silica treated with saline. The percentages of weight used were 0.03%, 0.08%, and 0.13%. Samples were blended using a magnetic stirrer for ± 30 min.

After the samples were well blended, they were heated in an oven for 24 h at a temperature of 110 $^{\circ}$ C to reduce the water content in the nanofluid.

Nanofluid Testing

Several tests were carried out to determine the characteristics of nanofluid. The test was the breakdown stress, density, viscosity, moisture content, acid level, pour point, and flash point.

The breakdown voltage test method used a mushroom electrode with a diameter of 25 mm under IEC 60156:2018. The sequence of testing for insulation oil is as follows. The oil sample to be tested was first filtered to remove solid particles, then heated to a temperature of 50 °C to remove moisture contained in the oil. After reaching the temperature, the oil was cooled to room temperature of about 26-30 °C. In the cooling process, the oil was covered with filter paper. The succeeding process involved pouring the oil into a dry, clean test vessel. Cautiously, the oil was poured into the test vessel to avoid the formation of gas bubbles. The oil should be 20.0 mm above the top of the electrodes or 40.0 mm from the axis of the electrodes. Then, the oil rested for about 10 minutes to detach any gas bubbles formed after pouring the oil into the test vessel.

HV Testing Equipment was turned on, and the start button was pressed; the voltage would increase automatically to 2.0 kV/sec (autotest). If the reset button was red, the value on the measuring instrument screen was the breakdown voltage. After the breakdown, the oil in the test vessel was stirred automatically with a thin rod to remove gas bubbles that arose during the breakdown. Then after the breakdown voltage had been recorded, the failure/reset button was pressed, and the test was repeated after 2 (two) minutes. The test was repeated up to six times, and then the breakdown voltages of the six tests were averaged.

RESULTS AND DISCUSSION

In this study, 1.6 L of POME-based insulation oil was produced, divided into four samples, i.e. pure oil and three other oils with the addition of nano silica with concentrations of 0.03 wt%, 0.8 wt% and 0.13 wt%, respectively. Data on the overall characteristics of the POME-based insulation oil are shown in Table 3.

Table 3. Testing Data								
	Oil		Std.		Nano silica weighted			
No	Characteristics	Unit	Test Method	Insulation Oil	0 %	0,03 %	0,08 %	0,13 %
1.	Density	g/cm ³	ASTM D – 1298	Max. 0.879	0.8757	0.8757	0.8759	0.8759
2.	Viscosity	cSt	ASTM D - 95	Max. 12	4.3267	4.1925	4.0919	4.0248
3.	Water Content	% Vol	ASTM D – 153	Max. 0.01	< 0.05	< 0.05	< 0.05	< 0.05
4.	Pour Point	°C	ASTM D – 97	Min30	14	14	14	14
5.	Flash Point	°C	ASTM D – 93	Min. 145	> 104	> 104	> 104	> 104
6.	Acid Level	mgKOH/g	ASTM D-974	Max. 0.06	0.5797	0.7106	0.7667	0.9163
7.	Breakdown Voltage	kV	IEC 60156:2018	Min. 30	60	60	60	60

Table 3.	Testing	Data
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Addition Effect of of Nano Silica Concentration on Insulation Oil Density

The density of each sample was almost the same, with a difference of 0.0002 g/cm3. Samples with 0.08% and 0.13% nano silica had a higher density of 0.8759 g/cm3 than samples without nano silica of 0.8757 g / cm3. These show that the addition of nano silica is minimal in affecting the density of the insulation oil. The density of the four samples complied with the standard, below 0.879 g/cm3. A graph of the percentage of nano silica concentration on density can be seen in Figure 2.

In previous research [14] pure oil density was 0.876 g/cm³. The density in this study was lower than in previous studies-the lower the density, the better the quality of the insulation oil. The density of the insulator oil is affected by heating, resulting in reduced bonds between molecules and reduced oil density; thus, the density value increases.

Effect of Addition of Nano silica **Concentration on Viscosity of Insulation Oil**

With the addition of nano silica, the viscosity of the sample decreased. The lowest viscosity was in the sample with the addition of 0.13% nano silica, 4.0248 cSt. It shows that adding nano silica can reduce the viscosity of insulation oil. The more nano silica, the lower the viscosity of the insulation oil. The four samples complied with the insulation oil standard because their viscosity was below 0.879 cSt. Figure 3. shows the Percentage of nano silica concentration on viscosity.

The viscosity of pure oil was 2.09 cSt in previous studies [14]. However, the viscosity in this study was higher than in the [14], the lower the viscosity, the better the quality of the insulation oil. The viscosity of the insulation oil is influenced by the size and molecular weight, pressure, temperature, and cohesion force between similar molecules [20]. Extraction, esterification, and transesterification proses have been carried out to reduce the viscosity.



Figure 2. Percentage of nano silica concentration to density



Figure 3. Percentage of nano silica concentration to viscosity

Effect Addition of Nano Silica of **Concentration on Water Content of Insulation** Oil

The water content in all samples was the same, < 0.05% Vol, while the standard water content is < 0.01% Vol. With a water content of < 0.05% Vol. it indicates the insulation oil has the potential to meet the standard. The effect of adding nano silica cannot be analysed since there is no definite value. The exact value was not obtained because the ability of the test equipment at the Laboratory had a minimum limit of 0.05% Vol. It is necessary to do a test with a

capability lowest limit below 0.01% Vol. However, based on theory, the addition of nano silica would reduce the water content since nano silica binds water in the oil [21]. The percentage of nano silica concentration in water content is shown in Figure 4.

In previous research, samples with nano silica decreased their water content from 0.02% Vol to 0.005% Vol [14].

Effect of Addition of Nano Silica Concentration on Water Content of Insulation Oil

The test results found that the flashpoint of all samples was> 104 ° C. It shows that the insulation oil could comply with the standards above 145 °C. The effect of adding nano silica to the flashpoint cannot be analysed because there is no definite value. The ability of Laboratory test equipment has a maximum limit of 104 °C, and it is necessary to conduct the test by utilising a tool with a higher limit above 145 °C. In Fig. 5, the percentage of nano silica concentration to the flashpoint.



Figure 4. Percentage of nano silica concentration in water content.



Figure 5. Percentage of Nano Silica concentration to the flash point

Previous studies found that the flashpoint of pure oil was 166 °C [14]. The greater the flashpoint of insulator oil, the better the quality of the insulation oil. In this research, efforts have been made to increase the flashpoint through the transesterification process.

Effect of Addition of Nano Silica Concentration on Acidity Level of Isolator Oil

The increase in acidity level was linear with the addition of nano silica. The lowest acid level was in the pure oil sample, 0.5797 mgKOH / g, and the highest was in the oil sample with 0.13% nano silica, namely 0.9163 mgKOH / g. The four samples had not complied with the standard, which must be below 0.06 mgKOH/g. Figure 6 shows the percentage of nano silica concentration at the acid level. In previous studies, samples with the addition of nano silica decreased from 0.5 mgKOH/g to 0.005 mgKOH/g [14].

The acid level of the insulation oil did not comply with the standard could be due to excessive retention duration, which was approximately four months - the Laboratory was closed. It caused fatty acids at room temperature to break down due to hydrolysis or oxidation to hydrocarbons, alkanes, or ketones, as well as small amounts of epoxy and alcohol. The process of forming this peroxide was accelerated by light, an acidic atmosphere, humidity, and a catalyst. The oxidation process occurs when an interaction occurs between a certain amount of oxygen, oil, and fat [22][23]. In this research, an attempt has been made to reduce the number of acids through the esterification process. The oil's initial acid level was 18 mgKOH/g before the esterification and transesterification processes.

Effect of Addition of Nano silica Concentration on Pour Point of Insulation Oil

The pour point for all samples was the same at 14 °C. Adding nano silica did not change the oil's pour point, and then the pour point did not comply with the standard, below -30 °C.



Figure 6. Percentage of Nano Silica to acidity level

In the previous study, it was found that the pour point of pure oil was 12.8 °C [3]. The pour point in this study was higher than—the lower the pour point, the better the quality of the insulation oil. One of the causes of the high pour point of insulation oil was the high content of saturated fatty acid methyl esters in vegetable oils thus was easy to crystallise and become a gel at low temperatures [24]. Waste-derived oil has a higher saturated fatty acid methyl ester than oil made from palm oil fruit. The percentage of nano silica concentration to the pour point can be seen in Figure 7.

Esterification and transesterification processes have been carried out to reduce saturated fatty acids. Moreover, further treatment is needed to reduce saturated fatty acids. The insulation oil in distribution transformers in Indonesia with continuous loading had a temperature of about 30 °C and a winding temperature of 98 °C [25]. It shows that POME-based insulation oil has the potential to be applied to transformers in Indonesia.



Figure 7. Percentage of Nano Silica concentration to pour point



Figure 8. Percentage concentration of Nano Silica against the breakdown voltage

Effect of Addition of Nano silica Concentration on Breakdown Voltage of Insulation Oil

All samples had the same breakdown voltage value of \geq 60 kV. It indicates that the insulation oil complied with the standard of \geq 30 kV. Figure 8. shows the percentage concentration of nano silica against the breakdown voltage.

Adding nano silica to plant-based insulation oil increased the breakdown voltage [21]. The higher the breakdown voltage, the better the quality of the insulation oil. The breakdown strength of insulation oil is subjected to electric field intensity, as shown in (2).

$$E = -\nabla V_b \tag{2}$$

Where E (V/m) is the electric field intensity, Vb (V) is the applied voltage, and the $-\nabla$ operator is as shown in Eq. 3.

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$$\nabla \equiv a_x \frac{\partial}{\partial x} + a_y \frac{\partial}{\partial y} + a_z \frac{\partial}{\partial z}$$
(3)

Equation (2) can be solved using a numerical method. The finite element method was used to obtain the potential and electric field distribution. At first, a model based on IEC 60156:2018 was developed as shown in Figure 9 (a). Furthermore, the problem type was defined as planar with a depth and solver precision of 1.0 mm and 10-8 respectively. The created mesh had 9496 nodes and the permittivity of the POME Nano Oil was defined as 2.25. High voltage and low voltage electrodes used copper materials whereas the distance between electrodes was 2.5 mm. In Fig. 9 (a), the simulation model and created mesh is shown. Subsequently, the electric field density of 30 kV and 60 kV input voltage (V_{in}) are shown in Figure 9 (b) and 9 (c) respectively. To observe the electric field density in the centre of the model, a horizontal line was drawn from the centre edge of both electrodes high voltage and low voltage. The horizontal line crossed the gap between the electrodes. In Figure 10, the difference can be observed.

From Figure 10 can be analysed that the breakdown strength is affected by electron emission. In POME nano silica insulation oil, electron emission from the cathode required higher energy to be ionised. Insulation oil breakdown strength is also influenced by viscosity and moisture content.







Figure 10. Electric field difference between 30 kV and 60 kV.

Lower viscosity leads to a higher breakdown strength of transformer oil since low viscosity oil is hard to be contaminated by foreign contaminants, which can form a conductive bridge on the oil [26][27]. Correspondingly, the more water content in the insulation oil, the lower the breakdown strength because the value of the water breakdown voltage is lower than that of the insulation oil [21].

A liquid insulation oil should possess good insulation characteristics. From conducted investigation, POME nano silica insulation oil has complied with IEC 60296 for oil density, viscosity, and breakdown strength. However, water content and the flash point are still uncertain since the requirement is to utilise equipment with better precision. Regarding the acidity level and pour point, further study must be conducted.

The acidity level can be improved by conducting an esterification process. In this study, the esterification used an HCL catalyst, and the process was carried out only once. However, the one-time esterification process is insufficient; it should be repeated until it obtains an acidity level that complies with the standard.

Then the pour point can be reduced using an isomerisation process. This process aims to give a branch at a straight carbon chain by contacting the hydrocarbon under pressure with proper catalyst assistance. The isomerisation reaction will change the molecule structure affecting insulation oil characteristics such as pour point, octane number, etc.

Regarding the breakdown voltage, even though it has exceeded the IEC standard, further testing is still required to obtain a specific value of breakdown strength. A higher high-voltage generator is required to conduct breakdown voltage tests.

CONCLUSION

An investigation on the potential of POMEbased insulation oil has been conducted. Seven characteristics have been tested with the results of three characteristics comply, two require more precession instruments, and two require further study. The results of this study can be utilised as a reference for further research regarding alternative raw materials for liquid insulation. As an alternative raw material for liquid insulation, POME-based insulation oil can reduce environmental pollution.

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