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(54) **VEGETABLE OIL, PREPARATION METHOD AND USE THEREOF**

(57) The present invention provides a vegetable insulating oil, preparation method and use thereof, and belongs to the technical field of liguid insulation. The vegetable insulating oil provided by the present invention includes the following components: 20%-90% by weight of refined rapeseed (RDB) insulating oil and 10%-80% by weight of mediumchain trimethylolpropane tri-ester (MTT) insulating oil. By using RDB and MTT insulating oils as raw materials in the present invention, the RDB insulating oil is mixed with different proportions of MTT insulating oil. Because the MTT insulating oil has prominent physicochemical properties and oxidation stability, the vegetable insulating oil prepared is guaranteed to feature high oxidation stability and low dielectric loss on the premise of excellent physicochemical properties and environmental protection performance.

VEGETABLE OIL, PREPARATION METHOD AND USE THEREOF

TECHNICAL FIELD

The present invention relates to the technical field of liquid insulation, and in particular to a vegetable insulating oil, preparation method and use thereof.

BACKGROUND

Vegetable insulating oil is easily-obtainable and fully-biodegradable insulating oil consisting essentially of triglyceride. Since 1990s, researchers began to use oilseed (e.g., rapeseed and soybean) to prepare novel vegetable oils, and these natural vegetable oils have a breakdown voltage at power frequency of >70 kV, higher than a breakdown voltage of mineral oil. Researchers have conducted experiments of dielectric strength, dielectric properties, viscosity, and thermal conductivity on vegetable oils, and it is believed that vegetable oils are entirely possible to be used as insulating oils instead of mineral oils.

Vegetable insulating oils have been used preliminarily, but natural vegetable insulating oils have low oxidation stability, and synthetic vegetable insulating oils have a large dielectric loss. These are not enough to limit a widely use thereof further.

SUMMARY

In view of this, the present invention provides a vegetable insulating oil, preparation method and use thereof. The vegetable insulating oil provided by the present invention features high oxidation stability and low dielectric loss.

To achieve the above objective, the present invention provides the following technical solutions.

The present invention provides a vegetable insulating oil, including the following components:

20%-90% by weight of refined rapeseed (RDB) insulating oil

and 10%-80% by weight of mediumchain trimethylolpropane tri-ester (MTT) insulating oil.

Preferably, the MTT insulating oil is prepared by esterification of trimethylolpropane (TMP), a mediumchain saturated fatty acid, and a catalyst.

Preferably, a molar ratio of the TMP to the mediumchain saturated fatty acid is 1:(3.0-3.8).

The present invention further provides a preparation method of the vegetable insulating oil according to the above technical solution, including the following steps:

mixing the MTT insulating oil with the RDB insulating oil to obtain an insulating oil mixture; and

degassing and deodorizing the insulating oil mixture to obtain the vegetable insulating oil.

Preferably, the dehydration and the degassing are conducted under vacuum with a vacuum degree of ≤-0.1 MPa.

Preferably, the dehydration and the degassing are conducted at 98-100°C.

The present invention further provides use of the vegetable insulating oil according to the above technical solution or a vegetable insulating oil prepared by the preparation method according to the above technical solution in distribution transformers.

The vegetable insulating oil provided by the present invention includes the following components: 20%-90% by weight of RDB insulating oil and 10%-80% by weight of MTT insulating oil. By using RDB and MTT insulating oils as raw materials in the present invention, the RDB insulating oil is mixed with different proportions of MTT insulating oil. Because the MTT insulating oil has prominent physicochemical properties and oxidation stability, the vegetable insulating oil prepared is guaranteed to feature high oxidation stability and low dielectric loss on the premise of excellent physicochemical properties and environmental protection performance. Results of examples indicate that: the vegetable insulating oil prepared by the present invention has an acid number of 0.03 mg $KOH \cdot g^{-1}$, dielectric loss factor is 0.5%-0.8% at 90°C,

breakdown voltage at power frequency is 64.1-68.4 kV, relative dielectric constant is 2.848-3.008 at 90°C, and oxidation induction time is 201-317 min.

DETAILED DESCRIPTION

The present invention provides a vegetable insulating oil, including the following components:

10%-80% by weight of MTT insulating oil and 20%-90% by weight of RDB insulating oil.

In the present invention, unless otherwise specified, all raw materials used are commercially available products conventional in the art.

The present invention provides a vegetable insulating oil, and the vegetable insulating oil includes 20% by weight to 90% by weight, further preferably 40% by weight to 90% by weight, further more preferably 80% by weight to 90% by weight, and most preferably 90% by weight of RDB insulating oil. In the present invention, the RDB insulating oil is preferably purchased from Henan EPRI Electric Power Technology Co., Ltd.

Based on the mass percentage of the RDB insulating oil, the vegetable insulating oil includes 10% by weight to 80% by weight, further preferably 10% by weight to 60% by weight, further more preferably 10% by weight to 20% by weight, and most preferably 10% by weight of MTT insulating oil.

In the present invention, a preparation method of the MTT insulating oil preferably includes the following steps:

Under a protective gas atmosphere, mixing, heating, and esterifying trimethylolpropane (TMP), a mediumchain saturated fatty acid, a catalyst, and a water-carrying agent to obtain esterification products;

under vacuum, separating, water-washing, and drying the esterification products successively, mixing the esterification products with an antioxidant and a metal passivator, followed by dehydrating and degassing, to obtain an original MTT insulating oil; and

alkali refining, water-washing, degassing, and deodorizing the original MTT insulating oil successively to obtain the MTT

insulating oil.

In the present invention, under a protective gas atmosphere, TMP, the mediumchain saturated fatty acid, the catalyst, and the water-carrying agent are mixed, heated, and esterified to obtain esterification products.

In the present invention, a molar ratio of the TMP to the mediumchain saturated fatty acid is preferably 1:(3.0-3.8), and more preferably 1:3.2. In the present invention, a number of carbon atoms of the mediumchain saturated fatty acid is preferably C6-C10; content of a mediumchain saturated fatty acid with a number of carbon atoms of >8 is preferably lower than 40% of the content of the mediumchain saturated fatty acid with a number of carbon atoms of 6 to 10. In the present invention, the catalyst is preferably stannic chloride. An amount of the catalyst is not particularly limited in the present invention and adjusted according to the practical conditions. In the present invention, the water-carrying agent is preferably xylene. An amount of the water-carrying agent is not particularly limited in the present invention and adjusted according to the practical conditions.

In the present invention, a mixing method is preferably as follows: heating and mixing the mediumchain saturated fatty acid with the TMP, the catalyst, and the water-carrying agent to conduct esterification. In the present invention, the heating is preferably conducted at 80-120°C, and more preferably 90°C. Heating rate at which is heated to the heating temperature is not particularly limited in the present invention, as long as the heating rate used is well known to those skilled in the art. In the present invention, the esterification is preferably conducted at 120-160°C; the esterification preferably lasts for 5 h. eating rate at which is heated to the esterification temperature is not particularly limited in the present invention, as long as the heating rate used is well known to those skilled in the art.

After the esterification products are obtained, under vacuum, the esterification products are separated, water-washed, and dried successively, mixed with the

antioxidant and the metal passivator, dehydrated and degassed to obtain the original MTT insulating oil.

In the present invention, the antioxidant is preferably a mixture of 6-*O*-palmitoyl-*L*-ascorbic acid, 2,6-di-tert-butyl-4-methylphenol, and tocopherol; a mass ratio of 6-*O*-palmitoyl-*L*-ascorbic acid: 2,6-di-tert-butyl-4-methylphenol: tocopherol in the mixture is preferably (1-6):(1-8):(1-11). An amount of the antioxidant is not particularly limited in the present invention and adjusted according to the practical conditions. Type of the metal passivator is not particularly limited in the present invention, as long as the type of the metal passivator used is well known to those skilled in the art. An amount of the metal passivator is not particularly limited in the present invention and adjusted according to the practical conditions.

In the present invention, the separation preferably includes solid-liquid separation, simple distillation, and vacuum distillation successively. In the present invention, preferably, a catalyst is obtained by the solid-liquid separation; a water-carrying agent is obtained by the simple distillation; an unreacted mediumchain saturated fatty acid is obtained by the vacuum distillation. In the present invention, the solid-liquid separation is preferably conducted by centrifugation or standing and precipitation. In the present invention, the simple distillation is preferably conducted at 60-100°C; a pressure of the simple distillation is preferably 100-600 mmHg. Specific operations of the simple distillation are not particularly limited in the present invention, as long as the simple distillation used is well known to those skilled in the art. In the present invention, the vacuum distillation is preferably conducted at 150-160°C; the vacuum distillation preferably has a vacuum degree of 2-10 mmHg. Specific operations of the vacuum distillation are not particularly limited in the present invention, as long as the vacuum distillation used is well known to those skilled in the art.

In the present invention, the water-washing is preferably conducted at 50-80°C; the water-washing preferably lasts for

0.5-3 h. In the present invention, the drying is preferably conducted by molecular sieve adsorption; a type of the molecular sieve is preferably a 3A molecular sieve. In the present invention, the mixing is preferably conducted under vacuum, and the vacuum preferably has a vacuum degree of 1-2 mmHg. In the present invention, the mixing is preferably conducted at 40-70°C; the mixing preferably lasts for 2-4 h. In the present invention, the mixing is preferably conducted by stirring; the stirring rate is not particularly limited in the present invention, as long as the stirring rate used is well known to those skilled in the art. Specific operations of the degassing and dehydrating are not particularly limited in the present invention, as long as the degassing and dehydrating used are well known to those skilled in the art.

In the present invention, after the original MTT insulating oil is obtained, the original MTT insulating oil is alkali refined, water-washed, degassed, and deodorized successively to obtain the MTT insulating oil.

In the present invention, the alkali refining preferably includes the following steps: mixing potassium hydroxide (KOH) solution with the original MTT insulating oil, followed by liquid-liquid separation, to obtain an alkali refined MTT insulating oil. In the present invention, alkali charge for the alkali refining preferably includes theoretical alkali quantity and excess alkali quantity; the theoretical alkali quantity is preferably 0.714-0.72 times a product of a mass of the original MTT insulating oil and an acid number thereof, which can be used to neutralize other small molecular acids and free fatty acids and is determined by the acid number of the original MTT insulating oil; the excess alkali quantity is preferably 0.002-0.003 times the mass of the original MTT insulating oil, which can meet technical requirements. In the present invention, the KOH solution preferably has a mass fraction of 2%, and a mass of the KOH solution is preferably a half of a sum of the theoretical alkali quantity and the excess alkali quantity. In the present invention, the mixing is preferably conducted by stirring, and the stirring is

preferably conducted at 65-70°C, and more preferably 65°C; the stirring is preferably achieved by stirring for 10-15 min at 100-110 r/min, followed by 30-40 min at 50-60 r/min. The liquid-liquid separation is not particularly limited in the present invention, as long as the liquid-liquid separation used is well known to those skilled in the art. A supernatant is separated by the liquid-liquid separation, i.e., the alkali refined MTT insulating oil.

In the present invention, the water-washing is achieved by washing with ultrapure water. In the present invention, the water-washing method preferably includes the following steps: mixing the alkali refined MTT insulating oil with the ultrapure water under stirring, followed by liquid-liquid separation, to obtain a water-washed MTT insulating oil. In the present invention, a temperature of the alkali refined MTT insulating oil is preferably 85-90°C, and more preferably 85°C. In the present invention, the stirring rate is preferably 50-60 r/min, and more preferably 50 r/min. In the present invention, a temperature of the ultrapure water is preferably 95-100°C, and more preferably 95°C. In the present invention, the mixing is preferably conducted by stirring, and the stirring rate is preferably 100-110 r/min, and more preferably 100 r/min; the stirring preferably lasts for 100-110 min, and more preferably 100 min. The liquid-liquid separation is not particularly limited in the present invention, as long as the liquid-liquid separation used is well known to those skilled in the art. A supernatant is separated by the liquid-liquid separation, i.e., the water-washed MTT insulating oil. In the present invention, a mass of the ultrapure water is preferably 0.15-0.18 times a mass of the alkali refined MTT insulating oil.

In the present invention, the degassing and deodorizing are preferably conducted in a vacuum drying oven. In the present invention, the degassing and deodorizing are preferably achieved by a high temperature method. In the present invention, the high temperature method is preferably achieved by conducting a first heating on the water-washed MTT

insulating oil, followed by a second heating after evacuating, to obtain the MTT insulating oil. In the present invention, a temperature of the first heating is preferably 95-98°C, and more preferably 98°C; the first heating preferably lasts for 30-40 min, and more preferably 30 min. The present invention preferably conducts the first heating until bubbles are slowly produced in the water-washed MTT insulating oil, followed by evacuating. When the vacuum degree ranges from -0.1 to -0.2 MPa during evacuating, the present invention preferably stops evacuating and holds for 3-4 h; after no bubble is produced in the water-washed MTT insulating oil, the second heating is conducted. In the present invention, a temperature of the second heating is preferably 180-200°C, and more preferably 200°C; the second heating time is not particularly limited in the present invention, as long as the second heating is stopped until no bubble is produced in the water-washed MTT insulating oil. Heating rate at which is heated to the temperature of the second heating is not particularly limited in the present invention, as long as the heating rate used is well known to those skilled in the art.

In the present invention, the original MTT insulating oil is alkali refined, water-washed, degassed, and deodorized to remove a plurality of impurities therefrom effectively, e.g., unreacted alcohols, free fatty acids, and catalyst residues. The presence of these impurities influences physicochemical properties and electrical characteristics of synthetic esters and particularly a normal operation of a transformer due to excessively high acid number and dielectric loss. In the present invention, the original MTT insulating oil has an acid number of 0.15 mg $KOH \cdot g^{-1}$ and a dielectric dissipation factor of 2.7 at 90°C before refining; after refining, the MTT insulating oil has an acid number of 0.03 mg $KOH \cdot g^{-1}$ and a dielectric dissipation factor of 1.6 at 90°C, effectively reducing the acid number and dielectric dissipation factor of the original MTT insulating oil.

The present invention further provides a preparation method of the vegetable insulating oil according to the above

technical solution, including the following steps:

mixing the MTT insulating oil with the RDB insulating oil to obtain an insulating oil mixture; and

dehydrating and degassing the insulating oil mixture to obtain the vegetable insulating oil.

In the present invention, the MTT insulating oil is mixed with the RDB insulating oil to obtain the insulating oil mixture.

In the present invention, the mixing is preferably conducted by magnetic stirring; the stirring rate is preferably 100-300 r/min, and more preferably 150 r/min; the stirring preferably lasts for 60-90 min. The mixing method used in the present invention ensures that raw materials do not splash during stirring. In the present invention, stoppering is done during mixing, preventing external impurities from entering into the insulating oil mixture.

In the present invention, after a vegetable insulating oil mixture is obtained, the vegetable insulating oil mixture is dehydrated and degassed to obtain the vegetable insulating oil. In the present invention, the dehydrating and degassing are preferably conducted in a vacuum drying oven; the dehydrating and degassing are preferably conducted at 98-100°C; the dehydrating and degassing preferably has a vacuum degree of ≤-0.1 MPa; the dehydrating and degassing preferably last for 2-4 days.

In the present invention, after dehydrating and degassing, a dehydrated and degassed vegetable insulating oil mixture is cooled to obtain the vegetable insulating oil. In the present invention, the cooling is preferably conducted under vacuum. In the present invention, the cooling is preferably achieved by furnace cooling. In the present invention, preferably, the vegetable insulating oil mixture is cooled to room temperature, removed, and stored in a sealed manner.

Preferably, a moisture of the vegetable insulating oil is determined in the present invention; a method for determining the moisture is preferably Karl Fischer Coulomb titration. If the moisture is below 50 mg/L in the vegetable insulating oil,

the vegetable insulating oil is deemed to be eligible; otherwise, the vegetable insulating oil is required to vacuum dry again to reduce the moisture.

The present invention further provides use of the vegetable insulating oil according to the above technical solution or a vegetable insulating oil prepared by the preparation method according to the above technical solution in distribution transformers.

The use is not particularly limited in the present invention, as long as the use is well known to those skilled in the art.

The vegetable insulating oil and the preparation method thereof provided by the present invention will be described in detail below in conjunction with examples, but should not be construed as limiting the scope of the invention.

Example 1

(a) First, 2 wt% KOH solution was weighed with a microsyringe and charged into original MTT insulating oil, while stirring for 10 min at 100 r/min at 65°C, followed by 30 min at 50 r/min; after standing in a separating funnel for 30 min, a supernatant was separated to obtain an alkali refined MTT insulating oil;

the alkali refined MTT insulating oil was heated to 85°C and stirred at 50 r/min, and ultrapure water was heated to 95°C; once the temperature met the requirement, stirring speed was accelerated to 100 r/min, while hot water was poured into the alkali refined MTT insulating oil, and stirred for 10 min at 100 r/min; after standing in a separating funnel for 60 min and observing an obvious layering phenomenon, a supernatant was separated to obtain a water-washed MTT insulating oil;

the water-washed MTT insulating oil was placed in a vacuum drying oven, heated to 98°C and held for 30 min until bubbles were produced slowly; a vacuum pump was turned on; the vacuum pump was turned off until a vacuum gauge reached -0.1 MPa; the state was held for 3 h until no bubble was produced; the temperature rose to 200°C and was held at 200°C until no obvious bubble was produced, so as to obtain an MTT insulating oil.

(b) An iodine flask was placed on an electronic balance,

and MTT and RDB insulating oils were weighed and prepared in the electronic balance; the MTT insulating oil had a mass percentage of 10%.

(c) A magnetic stirrer was placed in the iodine flask and stirred for 60 min at slow speed (150 r/min) to mixing two different insulating oils fully; during stirring, the oil did not splash; during stirring, the flask should be well stoppered for fear of entrance of external impurities into the mixed oil.

(d) After unstoppering, the iodine flask was placed in the vacuum drying oven, and dehydrated and degassed for two days at -0.1 MPa and 98°C; after vacuum drying, a heater of the vacuum drying oven was powered off and cooled to room temperature under vacuum; a vegetable insulating oil was prepared, removed timely and sealed.

(e) A Karl-Fischer Coulomb titrator was used to determine the moisture of the vegetable insulating oil after vacuum drying and check if the moisture thereof was at a low level (<50 mg/L). In case of excessively high moisture, the vegetable insulating oil would be needed to vacuum-dry again to reduce the moisture.

Performance tests were conducted on the vegetable insulating oil with eligible moisture. Acid number was determined in accordance with national standard GB 264-1983. Dielectric dissipation factor and relative dielectric constant were determined in accordance with national standard GB/T 5654-2007. Breakdown voltage at power frequency was measured in accordance with national standard GB/T 507-2002. Oxidation induction time was determined in accordance with national standard SH/T 0719-2002. Specific test results are shown in Table 1.

Example 2

The example had the same preparation conditions as those in Example 1, and the only difference was that the MTT insulating oil had a mass percentage of 20%.

Performance tests were conducted on the vegetable insulating oil with eligible moisture. Testing methods and standards were the same as those in Example 1. Test results

are shown in Table 1.

Example 3

The example had the same preparation conditions as those in Example 1, and the only difference was that the MTT insulating oil had a mass percentage of 40%.

Performance tests were conducted on the vegetable insulating oil with eligible moisture. Testing methods and standards were the same as those in Example 1. Test results are shown in Table 1.

Example 4

The example had the same preparation conditions as those in Example 1, and the only difference was that the MTT insulating oil had a mass percentage of 60%.

Performance tests were conducted on the vegetable insulating oil with eligible moisture. Testing methods and standards were the same as those in Example 1. Test results are shown in Table 1.

Example 5

The comparative example had the same preparation conditions as those in Example 1, and the only difference was that the MTT insulating oil had a mass percentage of 80%.

Performance tests were conducted on the vegetable insulating oil with eligible moisture. Testing methods and standards were the same as those in Example 1. Test results are shown in Table 1.

Comparative Example 1

The comparative example had the same preparation conditions as those in Example 1, and the only difference was that the MTT insulating oil had a mass percentage of 0%.

Performance tests were conducted on the vegetable insulating oil with eligible moisture. Testing methods and standards were the same as those in Example 1. Test results are shown in Table 1.

Comparative Example 2

The comparative example had the same preparation conditions as those in Example 1, and the only difference was that the MTT insulating oil had a mass percentage of 100%.

Performance tests were conducted on the vegetable insulating oil with eligible moisture. Testing methods and standards were the same as those in Example 1. Test results are shown in Table 1.

Comparative Example 3

Commercially available FR3 fluid (Cargill, Inc.) was taken as a comparative example to conduct performance tests. Testing methods and standards were the same as those in Example 1. Test results are shown in Table 1.

Table 1 Results of performance tests for vegetable insulating oils prepared in Examples 1 to 4 and Comparative Examples 1 and 2

Value / Performance parameter	Example 1	Example 2	Example 3	Example 4	Example 5	Comparative Example 1	Comparative Example 2	Comparative Example 3
Acid number/(mg KOH·g^{-1})	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Dielectric dissipation factor (90°C)/%	0.5	0.5	0.7	0.8	0.8	0.4	1.6	0.5
Breakdown voltage at power frequency/(kV)	65.9	68.4	66.7	65.1	64.1	64.4	68.3	68.8
Relative dielectric constant (90°C)	2.848	2.880	2.915	2.960	3.008	2.820	3.055	2.832
Oxidation induction time/(min)	218	216	201	244	317	190	>500	140

According to the above experimental results, different proportions of mixed vegetable insulating oils have an acid number of 0.03 mg $KOH \cdot g^{-1}$ and meet a requirement of national standard GB2536-90 that the acid number is not greater than 0.03 mg $KOH \cdot g^{-1}$. When the MTT insulating oil has a low mass percentage, the dielectric dissipation factor rises slowly; when the MTT insulating oil has a mass percentage of 10%, the MTT insulating oil meets a requirement of national standard GB2536-90 that the dielectric dissipation factor for transformer oils is not greater than 0.5%. The relative dielectric constant is linearly related to the mass percentage of the MTT insulating oil largely; when the MTT insulating oil has a mass percentage of higher than 10%, the relative dielectric constant of the vegetable insulating oil is far higher than that of mineral insulating oil (2.000), which advantageously prolongs the service life of insulating paper. For the vegetable insulating oil, average of the breakdown voltage at power frequency basically ranges between 65 and 70 kV, which is far greater than the minimum of the breakdown voltage at power frequency (35 kV) required in national standard GB2536-90. FR3 fluid (Cargill, Inc.) is one of the most commercially available vegetable insulating oils, with an oxidation induction time of 140 min, whereas the vegetable insulating oil has longer oxidation induction time than the FR3 fluid.

The foregoing descriptions is merely preferred examples of the present invention, it should be noted that various modifications and variations can be made by those skilled in the art without departing from the principles of the present invention and are within the scope of the invention.

CONCLUSIES

1. Plantaardige isolerende olie, omvattende de volgende componenten:

20-90 gew.% isolerende geraffineerde koolzaad(RDB)-olie en 10-80 gew.% isolerende olie van trimethylolpropaantri-ester met medium keten (MTT).

2. Plantaardige isolerende olie volgens conclusie 1, waarbij de isolerende MTT-olie bereid wordt door het veresteren van trimethylolpropaan (TMP), een verzadigd vetzuur met medium keten, en een katalysator.

3. Plantaardige isolerende olie volgens conclusie 2, waarbij een molaire verhouding van het TMP tot het verzadigde vetzuur met medium keten 1: (3,0-3,8) is.

4. Werkwijze voor het vervaardigen van de plantaardige isolerende olie volgens één van de conclusies 1 tot 3, omvattende de volgende stappen:

het mengen van de isolerende MTT-olie met isolerende RBD-olie om een mengsel van isolerende olie te krijgen; en

het dehydrateren en ontgassen van het mengsel van isolerende olie om de plantaardige isolerende olie te verkrijgen.

5. Werkwijze voor het vervaardigen van de plantaardige isolerende olie volgens conclusie 4, waarbij het dehydrateren en het ontgassen uitgevoerd worden onder vacuüm met een vacuümgraad van $\leq -0,1$ MPa.

6. Werkwijze voor het vervaardigen van de plantaardige isolerende olie volgens conclusie 4 of 5, waarbij het dehydrateren en het ontgassen uitgevoerd worden bij 98-100 °C.

7. Gebruik van de plantaardige isolerende olie volgens één van de conclusies 1 tot 3 of een plantaardige isolerende olie bereid door middel van de vervaardigingswerkwijze volgens één van de conclusies 4 tot 6 in distributietransformatoren.

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