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# Research and Development of Natural Vegetable Insulating Oil Based on *Jatropha curcas* Seed Oil

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**Abstract:** *Jatropha curcas* is a natural non-food resource with high oil-content seeds, that has attracted worldwide attention as it is an ideal renewable resource for the production of biofuels. With the increasing use of vegetable insulating oil in related industries, it is valuable to develop the vegetable insulating oils from *Jatropha curcas* seed oil. This study explores how to use *Jatropha curcas* seed oil to prepare high-quality natural vegetable insulating oil. A six-step process is first established according to the optimization results of alkali refining, activated clay treatment and alumina treatment of *Jatropha curcas* seed oil, combined with cold treatment, water washing and high temperature decompression treatment. Physicochemical and electrical performance tests show that most of the properties of the prepared vegetable insulating oil are significantly improved compared with the original seed oil, and meet the standard requirements for vegetable insulating oil, especially with no sulfur corrosion, a breakdown voltage of 72 kV and an acid value (KOH, potassium hydroxide) of 0.012 mg/g.

**Keywords:** vegetable insulating oil; *jatropha curcas*; alkali refining; electrical properties

## 1. Introduction

Most of the insulating oils currently used in the power industry are mineral-based oils. The use of mineral insulating oils has a long history, its cooling performance is good, and the cost is low [1–3]. However, mineral insulating oils have a low fire point, poor biodegradability, while its petroleum source is facing depletion [4,5]. Besides, corrosive sulfur in mineral insulating oils has been reported to be the main culprit inducing failures in shunt reactors and power transformers. If not processed in time, this will affect the safety of power operations as the insulating oil is continuously degraded during operation [6]. Moreover, once a leak, fire, or other similar accident occurs, the environment will be seriously polluted [7]. Therefore, the Institute of Electrical and Electronics Engineers (IEEE) has released its "Guide for the Reclamation of Insulating Oil and Criteria for Its Use" (IEEE Std 637-1985, superseded by IEEE Std C57.637-2015). In China, waste transformer oil has been included in the "National Hazardous Waste List" since 2016. Therefore, it is important to develop safer and cleaner insulating oil replacement products.

Studies have indicated that vegetable insulating oil can be used as a substitute for mineral insulating oil in transformers and oil-filled cables [8]. Vegetable oils have excellent electrical properties such as flash points greater than 300 °C, which significantly improves the safety level of the power grid, biodegradation rates of more than 95%–97%, and relative dielectric constants about 1.5 times that of mineral insulating oils [9]. Therefore, as an environmentally friendly product, vegetable insulation oils are increasingly used in related industries [4]. Moreover, the vegetable oils used to prepare insulating oils are renewable since they are produced continuously by oil plants.

However, vegetable oils are generally not a direct substitute for mineral insulating oils. Parameters such as breakdown voltage, acid value, and dielectric loss need to be improved [10]. At present, vegetable insulation oils are prepared from soybean oil, rapeseed oil, palm oil, Camellia Oil and esters, and so on [9,11,12].

*Jatropha curcas* L. (Euphorbiaceae) is a multipurpose small tree with a high seed oil content (34.4% on average) [13–15]. It has been considered worldwide as an ideal renewable natural resource for the production of biodiesel and bio jet fuel [16–18]. Moreover, the seed oil has long been used for making soap, lamp oil, paraffin, and in the cosmetics industry [19,20]. Compared with other oil plants that can be used to make vegetable insulation oils (such as soybean, rapeseed, sunflower, and sesame), it can grow on barren soils and in areas with limited annual rainfall [21]. However, there have been few reports on the use of *Jatropha curcas* seed oil as a raw material to prepare industrial insulating oils. Therefore, in this study we developed up-to-standard vegetable insulating oils from *Jatropha curcas* seed oil by simple methods to improve the comprehensive utilization and economic competitiveness of *Jatropha curcas* resources.

The seed oil of *Jatropha curcas* contains a number of fatty acids, including oleic acid, linoleic acid, and palmitic acid However, its acid value can reach 8–28 mg KOH/g [22,23], while the ASTM (American Society of Testing Materials) vegetable insulating oil standard (ASTM D6871-2003) stipulates that the acid value should be equal to, or less than, 0.06 mg KOH/g and the National Standard for Transformer Oil of China (GB2536-90) standard requires the insulating oil acid value to be 0.03 mg KOH/g or less. Therefore, decreasing the acid value is one of the key issues to be solved in the preparation of vegetable insulating oil from *Jatropha curcas* seed oil. Studies have shown that the procedure to refine the vegetable oil-based dielectric liquid consists of three steps in order to reduce the acid value: alkaline refinement, bleaching and decompression distillation [11,24]. Moreover, crude vegetable oils extracted from oil seeds generally have a dark color and contain solid constituents such as proteins and fibers [7]. Therefore, the main focus of this study was to reduce the acid value and to explore a novel method to improve the quality of *Jatropha curcas* seed oil to meet the standard requirements for vegetable insulating oil.

## 2. Materials and Methods

### *2.1. Reagents and Instruments*

Preparation of *Jatropha* seed oil: The fresh and mature *Jatropha curcas* seeds were collected from Ningnan County, Sichuan Province, China (27°07′ N, 102°77′ E) and pressed with a common squeezer and left to precipitate more than 15 days. The resulting supernatant oil was used as the crude *Jatropha curcas* oil (CJCO) for subsequent experiments.

The KOH used was of analytical grade. Activated clay and aluminum oxide were purchased from Shenzhen Zhenbang Environmental Technology Co., Ltd. (Guangdong, China). Alkali refining, activated clay treatment, alumina treatment and vegetable insulating oil preparation were carried out through a reactor capable of stirring, heating and depressurization, which was equipped with a heater and a vacuum pump, purchased from Shanghai Qiuzuo Science Instrument Co., Ltd., Shanghai, China.

### *2.2. Experimental Methods*

#### 2.2.1. Alkali Refining

KOH treatment was used to reduce the acid value of CJCO by alkali refining, and the treatment amount and time were optimized using 300 g CJCO per treatment. The amount of KOH used was 0%, 0.1%, 0.5%, 1%, 3%, and 5% (w/w) of the quantity of seed oil. By referring to related research [25,26], the alkali refining process of this study was designed as follows. A KOH solution (20% w/v) in deionized water was added to the CJCO, stirred at room temperature for 30 min, then centrifuged at 6500× *g* for 10 min at room temperature. The oil was collected and washed in a beaker by adding

an equal volume of deionized water, stirred for 3 min, allowed to stand for 30 min, after which the oil was collected. The washing was repeated 2 times. The acidity of the oil in this study was assayed by the method of the Chinese National Standard GB 5009.229-2016 and the optimal KOH concentration was determined based on the acidity values. Subsequently, the optimal alkali refining time was determined by measuring the acidity at different reaction times based the optimal KOH amount. The reaction times tested were 5 min, 10 min, 20 min, 30 min, 60 min, and 90 min. Each test setting of KOH amount and alkali refining time was repeated 3 times and the average value was taken for the parameter optimization.

#### 2.2.2. Activated Clay Treatment

Activated clay treatment was carried out based on the procedures described by Dulger and Gecgel [27] with appropriate modifications. Active clay was added to 150 g CJCO or alkali treated CJCO, then moved into the reaction kettle and stirred for 45 min at 75 °C under reduced pressure. Subsequently the mixtures were collected, centrifuged for 10 min at a speed of 6500× *g* at room temperature. The supernatant oil was collected, and the acid value was measured. Different amounts of active clay were used to determine the optimal amount according to the acid value. Three replicates were set for each treatment, and the average value was used to optimize the amount of clay.

#### 2.2.3. Aluminum Oxide Treatment

Aluminum oxide treatment was performed according to the modified method of Gil et al. [28]. Different mass ratios of aluminum oxide were added to 150 g CJCO or alkali or clay treated CJCO and stirred for 3 h. Then the mixtures were centrifuged at 6500× *g* for 10 min at room temperature, the oil was collected, and the optimal amount of aluminum oxide was determined by measuring the acid value of the oils. Three repeats were set for each process, and the average value was used to optimize the amount of alumina.

#### 2.2.4. Preparation of *Jatropha Curcas* Natural Vegetable Insulating Oil (JNIO)

According to the experimentally obtained optimal parameters of KOH, activated clay, and aluminum oxide treatments, JNIO was prepared as outlined in Figure 1. The yield of every step of the process was investigated. Three copies of JNIO were prepared for related parameters and physical and chemical properties detection, and the average value was used for analysis.

#### 2.2.5. Test of Physicochemical and Electrical Properties of JNIO

The relevant indicators of plant insulating oil prepared from *Jatropha curcas* seed oil were tested in accordance with the Chinese Standard DL/T 1360-2014 "Quality Criteria of Soybean Plant Transformer Oils". This standard refers to ASTM D5222-2008 "Standard Specification for High Fire-Point Mineral Electrical Insulating Oils", ASTM D6871-2003 "Standard Specification for Natural (Vegetable Oil) Ester Fluids Used in Electrical Apparatus" and IEEE C57.147—2008 "Guide for Acceptance and Maintenance of Natural Ester Fluids in Transformers", formulated in accordance with the actual situation of oil products. The ASTM D6871-2003 standard was developed as ASTM D6871-17. Thus, the standard specifications between DL/T 1360-2014 with ASTM D6871-17 referred in this study were similar.

#### 2.2.6. The Detection of Fatty Acids

Fatty acids in oils were assayed by Qingdao Sci-tech Innovation Quality Testing Co., Ltd., Qingdao, China using a Thermo Trace 1310 gas chromatograph mass spectrometer (see Table S1 for details).

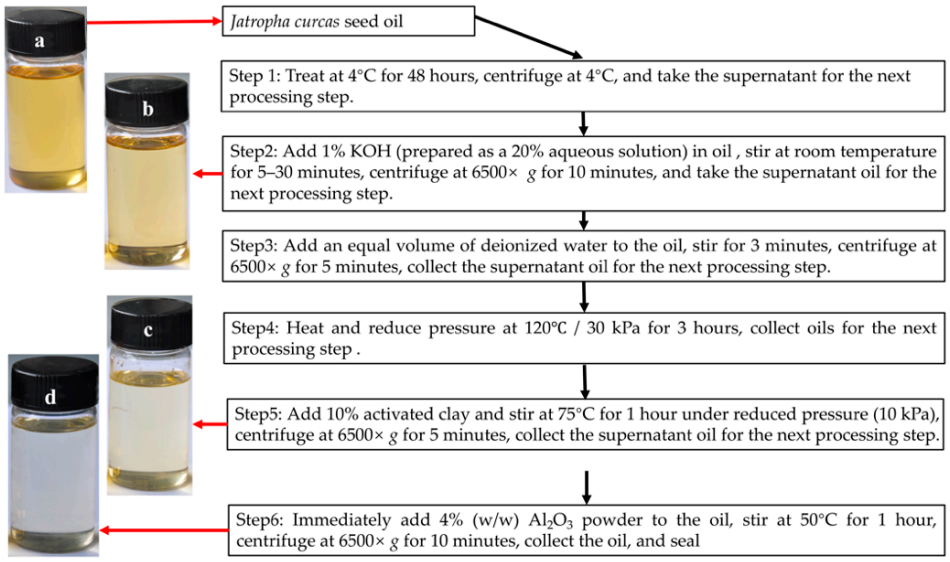


**Figure 1.** The six-step process of de-acidifying JNIO (show the specific process). a: crude Jatropha curcas oil (CJCO); b: oil following alkaline refinement; c: oil following activated clay treatment; d: Jatropha curcas natural vegetable insulating oil (JNIO). Red arrow indicates the corresponding processed oil; black arrow indicates the processing progress. Steps one to six of this procedure are: cold treatment, alkaline refining, water wash, high-temperature decompression treatment, clay treatment, $Al_2O_3$ treatment.

## 3. Results

### 3.1. Effective Decreasing of CJCO Acidity

The average acid value of *Jatropha curcas* seed oil used in this study was 9342 mg KOH/g. Therefore, according to the requirements of the acid value of the vegetable insulating oil standard, *Jatropha curcas* seed oil is a high acid oil. Thus, this study first explored the effects of KOH, activated white clay, and aluminum oxide on reducing the acid value of *Jatropha curcas* seed oil. The results show that KOH treatment led to a significant reduction in the acid value of *Jatropha curcas* seed oil. The effect of KOH treatment was greatest while activated clay and aluminum oxide had a limited effect on reducing the acid value Figure 2.

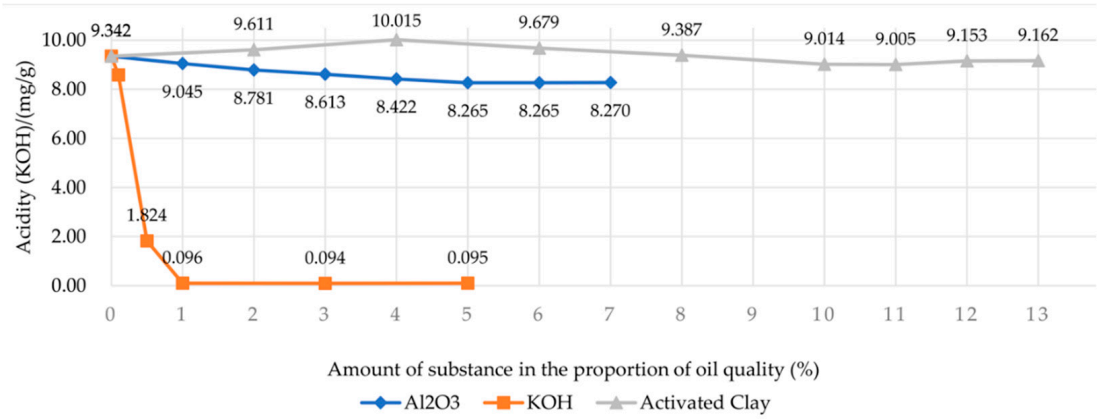


**Figure 2.** Effects of three different treatments on the acidity value of *Jatropha curcas* seed oil.

Studies have shown that the amount of alkaline used and the temperature during Alkali refining influence the quality of the vegetable oil [24]. Our results show, that when the amount of KOH used increased from 0.1% to 1% (w/w) of the CJCO mass, the acid value of *Jatropha curcas* seed oil decreased.

As the amount of KOH used reached 1% (w/w) of the CJCO mass, the acid value fell to its lowest value of 0.096 mg KOH/g. Adding greater amounts of KOH (2%–5% w/w) did not lead to a further reduction in the acid value (Figure 2). Thus, the optimal amount of KOH was 1% (w/w) of the oil mass, which led to a reduction in the acid value of the CJCO from 9.342 mg KOH/g to 0.096 mg KOH/g. Next, we tested the effect of adding activated clay on the acidity value of the oil. When the amount of activated clay used increased from 0%–4% (w/w) of the CJCO mass, the acid value increased as well. As the amount of activated clay used rose above 4% (w/w), the acid value decreased. The acid value reached the lowest value of 9005 mg/g as 10% (w/w) activated clay was used Figure 2, representing a decrease of 0.337 mg/g. We also tested the effect of adding aluminum oxide. The acid value of CJCO decreased as the amount of aluminum oxide used increased. When the amount of aluminum oxide reached 5% (w/w) of the CJCO mass, the acid value reached its lowest value of 8265 mg/g, i.e., a decrease of 1.077 mg/g (Figure 2). In summary, activated clay and aluminum oxide reduced the acid value of *Jatropha curcas* seed oil relatively weakly, i.e., by less than 1.1 mg/g.

According to the above results, CJCO was treated using 1% (w/w) KOH for different refining times after which the acid values were measured. The results show that the acid value decreased with extended reaction times. The acid values decreased to a minimum of 0.096 mg KOH/g, when the reaction time reached 30 min, after which the acid values tended to stabilize (Figure 3). Therefore, in subsequent experiments, the alkaline refining reaction times of CJCO was set to 30 min.

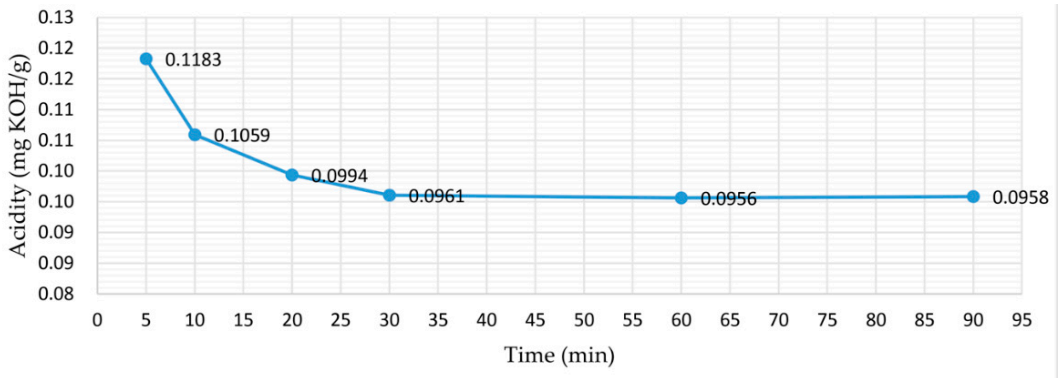


**Figure 3.** Effect of different alkali refining times on the acid value of CJCO. CJCO, crude *Jatropha curcas* oil.

Alkaline refining of vegetable oils is usually performed between 50–90 °C [29,30]. Therefore, we compared the effect of alkaline refining (using 1% (w/w) KOH) at room temperature vs. at 65 °C on the acid value of CJCO. The results showed that the acid value of CJCO treated at 65 °C reached 0.095 mg KOH/g, which was not significantly different from the acid value (0.096 mg KOH/g) treated at room temperature.

However, alkaline refining at room temperature resulted in a better separation of the soap base from the oil than when the procedure was carried out at 65 °C (Figure 4). When the alkaline refining reaction was done at room temperature, the oil and soap base could be separated by centrifugation at 6500× *g* for 5 min resulting in a clear, light yellow oil with improved color compared to CJCO (Figure 4a,b).

However, when *Jatropha* seed oil was subjected to alkaline refining at 65 °C, and then centrifuged at 12,000× *g* for 30 min at room temperature immediately or after 1 h, the oil and soap base were still mixed together and became cloudy (Figure 4c). That is, after CJCO was subjected to alkaline refining at 65 °C, even if the centrifugation speed and time were increased, the separation of the oil and soap base was poor. Therefore, for *Jatropha curcas* seed oil, alkaline refining at room temperature not only greatly reduced the acid value of the oil, but also resulted in better separation of the oil and soap base than when the procedure was done at 65 °C.

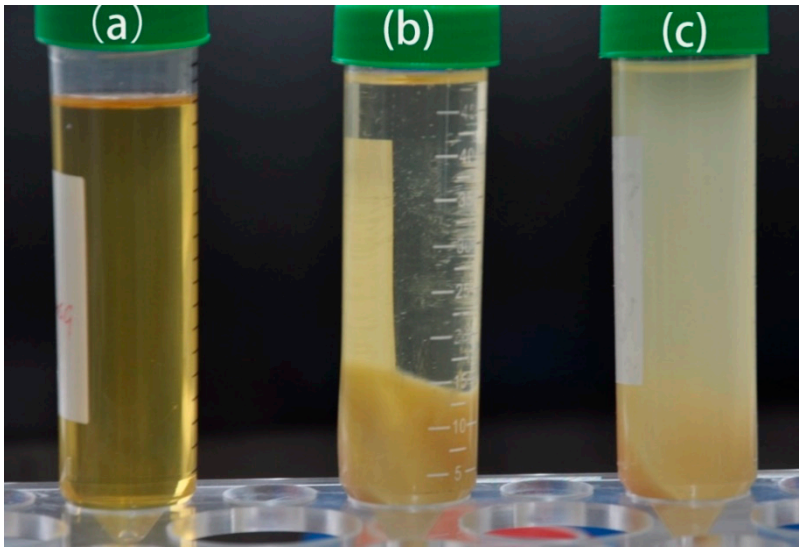


**Figure 4.** Separation of soap base and oil after alkaline refining at room temperature vs. 65 °C. (**a**) *Jatropha curcas* seed oil; (**b**) after KOH (1% w/w) treatment at room temperature; (**c**) after KOH (1% w/w) treatment at 65 °C.

### 3.2. Effect of Activated Clay on Improving the Acidity for Preparing JNIO

In this study, the acid value of CJCO was reduced to 0.096 mg KOH/g after deacidification by alkali refining. This value did not meet the requirements for the acid value in ASTM D6871-17 and GB2536-90, which are 0.06 mg KOH g and 0.03 mg KOH/g, respectively. Therefore, we used treatment with activated clay to further reduce the acid value and to improve the color of the alkali-refined oil.

The alkali-refined seed oil was treated with 8%–12% active clay. Treatment with 8% (w/w) active clay led to an increase in the acid value as shown in Figure 5. However, treatment with 9%–12% (w/w) of active clay resulted in a marked decrease in acid value. A minimum value of 0.043 mg KOH/g was obtained using 10% (w/w) active clay. At this point, the acid value had reached the ASTM D6871-17 standard but had not yet reached the GB2536-90 standard and thus, needed further processing. Therefore, in the subsequent experiments, the amount of active clay used was 10% (w/w) of the oil mass. To further reduce the acid value of the oil and moisture content, aluminum oxide treatment was subsequently selected.

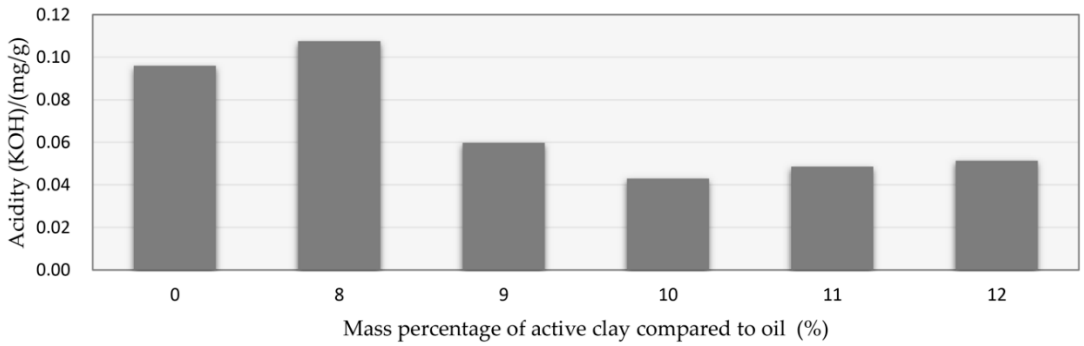


**Figure 5.** Effect of treatment with different amounts of active clay on the *Jatropha curcas* seed oil acid value. Oils had been pre-treated with KOH (1% w/w).

### 3.3. Effect of Basic Aluminum Oxide Treatment on Lowering the Acidity of JNIO

Alkali and activated clay-treated CJCO was subjected to different amounts (w/w) of basic aluminum oxide at 50 °C for 1 h. The results showed (Figure 6) that when increasing amounts of the aluminum oxide were added, the acid value of the oil showed a downward trend. When the amount of aluminum

oxide used was 4% (w/w) of the oil mass, the acid value dropped to 0.0116 mg KOH/ g. Further increases in the amount of aluminum oxide added did not lead to significant changes in the acid value (Figure 6). In addition, there was no significant difference in the acid value between 1 h of aluminum oxide treatment and 2 h of treatment (data not shown). Therefore, the optimal amount of aluminum oxide was considered to be 4% (w/w) of the oil mass and optimal treatment time was 1 h.

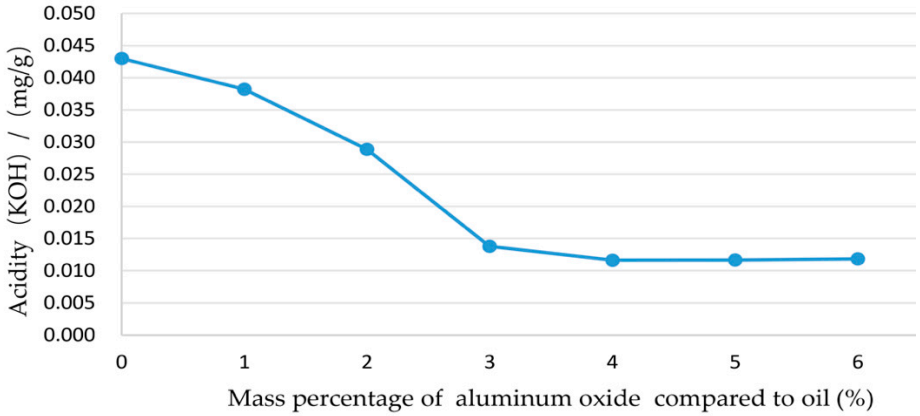


**Figure 6.** The effect of alkaline aluminum oxide treatment on the acid value of *Jatropha curcas* seed oil. Seed oils had been previously treated with KOH (1%, w/w) and active clay (10%, w/w).

### *3.4. Process Design for Preparing JNIO*

Based on the preferred parameters obtained from the above experiments, a six-step process, as shown in Figure 1, was designed to prepare JNIO. According to this process, natural vegetable insulating oils can be produced that meet industrial standards. The color of CJCO gradually improved throughout the main steps of alkaline refining, active clay and aluminum oxide treatments (Figure 1).

Table 1 evaluates the output efficiency for each of the six steps using 500 g of CJCO as the starting material for the preparation of JNIO. Except for the "water wash" step (87.9%), the output efficiency of each of the other steps was 95% or greater. The overall output efficiency of this vegetable insulating oil production process was 75% (Table 1).

**Table 1.** Stepwise yield during the process of preparing vegetable insulating oil from *Jatropha curcas* seed oil.

| Step | Initial Oil Mass (g) | Collected Oil Mass (g) | Output Efficiency [1] |
|---|---|---|---|
| Cold treatment | 500 | 480 | 96% |
| KOH treatment | 480 | 455 | 95% |
| Water wash | 455 | 400 | 87.9% |
| High-temperature decompression | 400 | 395 | 98.8% |
| Clay treatment | 395 | 380 | 96.2% |
| $Al_2O_3$ treatment | 380 | 375 | 98.7% |
| Total | 500 | 375 | 75% |

[1] Output efficiency= 100% × (Collected oil mass/Initial oil mass).

### *3.5. Physicochemical and Electrical Properties of JNIO*

The physical, chemical, and electrical properties of three replicate samples of CJCO and JNIO were tested according to the relevant parameters of the standard DL/T 1360-2014. Variance analysis by Excel software showed that CJCO and JNIO had extremely significant differences in water content, dissipation factor, dielectric breakdown voltage, flash point, ignition point, pour point, and total acid content, while the differences in relative density and viscosity were not significant (Tables S2 and S3). Overall, compared with CJCO, JNIO's relevant parameters for vegetable insulating oils were greatly

improved, and all tested parameters of JNIO met ASTM D6871-17 and DL/T 1360-2014 standards (Table 2). The JNIO prepared according to the process outlined in Figure 1 was clear and transparent, free of impurities and corrosive sulfur. In addition, the water content of JNIO was 376 ppm lower than that of CJCO, and the dissipation factor was significantly reduced, from 0.67% in CJCO to 0.1% in JNIO. The breakdown voltage of JNIO reached 72 kV, which was 16.6 kV greater than that of CJCO. The acid value of JNIO was 0.012 mg KOH/g, which was significantly lower than the acid value of CJCO (9432 mg KOH/g) and was five times smaller than the standard maximum value. The flash point had increased from 236 °C for CJCO to 295 °C for JNIO, while the fire point had slightly improved as well. Compared with CJCO, the pour point of JNIO had greatly improved from −2 °C to −10 °C. JNIO had the same viscosity as CJCO, which was significantly better than the standard requirements for vegetable insulating oils (Table 2). In summary, using *Jatropha curcas* seed oil as the raw material, a high-quality vegetable insulating oil can be prepared by the process designed in this study.

**Table 2.** Physicochemical Properties of CJCO, JNIO and Specifications for Natural Vegetable Insulating Oil.

| Property | CJCO | JNIO | DL/T 1360-2014 [1] | | ASTM D6871-17 [2] | |
|---|---|---|---|---|---|---|
| | | | Method | Specification | Method | Specification |
| Visual examination | Yellow | Colorless clear and bright | Visual Examination | Bright and Clear | D1524 | Bright and Clear |
| Water content, ppm | 569 | 193 | GB/T 7600-2014 | ≤200 | D1533 | ≤200 |
| Dissipation factor, % | 0.67 | 0.1 | GB/T 5654-2007 | ≤0.5(90 °C) | D924 | ≤4(at 100 °C) |
| Dielectric breakdown voltage, kV | 55.4 | 72 | GB/T 507-2002 | ≥35 | D877 | ≥35 |
| Flash point, °C | 236 | 295 | GB/T 3536-2008 | >250 | D92 | ≥275 |
| Fire point, °C | 308 | 320 | GB/T 3536-2008 | >300 | D92 | ≥300 |
| Viscosity at 40 °C, mm2/s | 32.9 | 32.83 | GB/T 265-1988 | ≤50 | D445 or D88 | ≤50 |
| Viscosity at 100 °C, mm2/s | 7.377 | 7.456 | GB/T 265-1988 | ≤15 | D445 or D88 | ≤15 |
| Pour point, °C | −2.0 | −10.0 | GB/T 3535-2006 | ≤−10 | D97 | ≤−10 |
| Relative density, g/cm3 | 0.915 | 0.915 | GB/T 1885-1998 | ≤1.0 | D1298 | ≤0.96 |
| Corrosive sulfur | not corrosive | not corrosive | GB/T 25961 | not corrosive | D1275 | not corrosive |
| Total acid content, mg KOH/g | 9.342 | 0.012 | GB 5009.229-2016 | ≤ 0.06 | D974 | ≤0.06 |

[1] DL/T 1360-2014: Quality criteria of soybean plant transformer oils, [2] ASTM D6871-17: Standard Specification for Natural (Vegetable Oil) Ester Fluids Used in Electrical Apparatus.

Compared with some reported vegetable insulating oil products [9,11,12], the overall performance index of JNIO can meet the requirements of the ASTM standards, and it was better in visual appearance, breakdown voltage, acidity, dissipation factor and viscosity (Table 3). For example, JNIO was clear and bright while the others were usually light yellow or green. The acidity, dissipation factor and viscosity of JNIO were the lowest among these products (Table 3).

**Table 3.** Statistics of types and performance for vegetable insulation oil.

| Property | JNIO | Rapeseed Oil | Sunflower Oil | Soybean Oil | Palm Oil | Camellia Oil | Cargill FR3 [1] |
|---|---|---|---|---|---|---|---|
| Visual examination | clear and bright | | | | | Light yellow | Light green |
| Water content, ppm | 193 | | < 80 | < 80 | | | |
| Relative density, g/cm$^3$ | 0.915 | 0.9 | 0.919 | 0.923 | 0.90 | 0.9 | 0.92 |
| Acidity, mg KOH/g | 0.012 | 0.03 | 0.02 | 0.02 | | 0.03 | 0.04 |
| Dielectric breakdown voltage, kV | 72 | 73 | 38–45 | 51 | 75 | | |
| AC breakdown voltage, kV | | | | | | 70 | 56 |
| Dissipation factor at 90 °C, % | 0.1 | 0.75 | | | | 0.88 | |
| Dissipation factor at 100 °C, % | | | 0.59 | 1.85 | | | 0.89 |
| Flash point, °C | 295 | 325 | <330 | 326 | >220 | 322 | 316 |
| Fire point, °C | 320 | | <360 | 362 | >220 | | |
| Viscosity at 100 °C, mm$^2$/s | 7.456 | | 10 | | | | |
| Viscosity at 40 °C, mm$^2$/s | 32.83 | 43 | 41.4–45 | 33.8 | 48–50 | 39.9–28 | 34.1 |
| Pour point °C | −10.0 | −18 | −12 to −25 | −21 | 15 | | −21 |

[1] Cargill FR3 is a natural ester derived from renewable vegetable oil made by Cargill Incorporated.

### 3.6. Fatty Acid Composition of CJCO and JNIO

The pour point of CJCO has been tested to be −2 °C (Table 2), which did not meet the requirements of −10 °C for vegetable insulation oil standards. For *Jatropha curcas* seed oil, the fatty acid composition mainly consisted of unsaturated fatty acids, with saturated fatty acids comprising about 22% [13,23], which was consistent with the CJCO analysis results in this study (Table 4). Since the freezing point of unsaturated fatty acids is lower than the freezing point of saturated fatty acids, the low-temperature fluidity of the oil can be improved by reducing the content of saturated fatty acids. Therefore, our process included the CJCO to be incubated at 4 °C for 48 h, followed by a centrifugation step to remove the precipitated saturated fatty acids (Figure S1) present in the CJCO.

The relative content of different fatty acids in CJCO and JNIO was analyzed by gas chromatography mass spectrometry. The results showed that the proportion of most saturated fatty acids decreased, and the proportion of most unsaturated fatty acids increased, with the largest increase being oleic acid. Compared with CJCO, the total saturated fatty acid content had reduced from 22.2096 g per 100 g to 20.4753 g per 100 g in JNIO, a reduction of 1.7343 g, while total unsaturated fatty acid content increased from 73.42 g per 100 g in CJCO to 75.1572 g per 100 g in JNIO, an increase of 1.7372 g, of which oleic acid (C18: 1) increased by 2.0408 g, accounting for 2% of the total oil volume (Table 4, Figure S2). In brief, treatment of CJCO according to the process established in this study increased the unsaturated fatty acid content and decreased the saturated fatty acid content in the resulting JNIO. This might be the main factor that caused the pour point of JNIO to be lower than that of CJCO.

**Table 4.** Fatty acid composition of CJCO, JNIO.

| Fatty Acid Types | CJCO Content (g/100g) | JNIO Content (g/100g) | JNIO–CJCO Content (g/100g) |
|---|---|---|---|
| C14:0 | 0.0625 | 0.0527 | −0.0098 |
| C15:0 | 0.0000 | 0.0128 | 0.0128 |
| C16:0 | 14.3300 | 13.3658 | −0.9642 |
| C16:1 | 0.7105 | 0.6995 | −0.0110 |
| C17:0 | 0.0997 | 0.0872 | −0.0125 |
| C18:0 | 7.3554 | 6.6594 | −0.6960 |
| C18:1 | 39.0613 | 41.1021 | 2.0408 |
| C18:2 | 33.2898 | 33.0683 | −0.2215 |
| C20:0 | 0.2426 | 0.2210 | −0.0216 |
| C20:1 | 0.2534 | 0.1738 | −0.0796 |
| C18:3 | 0.0000 | 0.0615 | 0.0615 |
| C22:0 | 0.0540 | 0.0406 | −0.0134 |
| C23:0 | 0.0371 | 0.0219 | −0.0152 |
| C22:2 | 0.0377 | 0.0000 | −0.0377 |
| C24:0 | 0.0283 | 0.0139 | −0.0144 |
| C20:5n3 | 0.0673 | 0.0520 | −0.0153 |
| Saturated fatty acids | 22.2096 | 20.4753 | −1.7343 |
| Unsaturated fatty acids | 73.4200 | 75.1572 | 1.7372 |

## 4. Discussion

This study found that alkali treatment had the greatest effect on reducing the acid value of *Jatropha curcas* seed oil, while activated clay and aluminum oxide treatment displayed relatively weak effects on the reduction in acid value. In the alkaline refining process of this study, KOH was used because the refined by-product soap base could be used to make potassium soap. However, NaOH could reduce the acid value of *Jatropha curcas* seed oil as effectively as KOH (data not shown).

Traditionally, the method of reducing the acid value of vegetable oils by alkali refining is to stir the reaction at 50 °C~90 °C [29–32]. However, in this study, the alkaline refining process of *Jatropha curcas* seed oil was performed at room temperature, which requires less energy and is simpler to operate than the high-temperature alkaline refining process. Moreover, this study shows that the oil can be better separated from the soap base when alkaline refining takes place at room temperature, and there is no

significant advantage in deacidification by heat treatment. Therefore, for the preparation of vegetable insulation oil from some seed oils, it is preferred to reduce the acid value by performing the alkaline refining at room temperature.

We found, that alkaline refining for 5 min at room temperature reduced the acid value of *Jatropha curcas* seed oil from 9432 to 0.1183 mg KOH/g, while 30 min of alkaline refining, reduced the acid value to 0.096 mg KOH/g (Figure 3). Since there is no significant difference in the decrease in acid value between the two treatment times it could be considered to reduce the reaction time during step two to 5 min.

Based on the vegetable insulating oil prepared in this study, a better vegetable insulating oil can be developed according to actual needs. For example, the pour point of the vegetable insulation oil prepared in this study was −10 °C, which just meets the requirements of ASTM D6871-17 and DL/T 1360–2014 standards. However, this parameter could be improved by increasing the content of unsaturated oils, by increasing the number of branched fatty acids, or by adding anti-condensing agents.

Moisture in the insulating oil is considered to be "enemy number one" for transformer insulation [33]. The moisture in the insulating oil can increase the conductivity and dissipation factor and reduces the electrical strength of the transformer oil, which greatly affects the electrical properties, the life expectancy, and the load capacity of a transformer [9]. Maintaining low water concentration in the insulating oil is an important factor in achieving low dielectric loss, a high dielectric breakdown strength, and a low degradation rate [34].

However, the ester oils absorb many times more moisture than mineral oils because the moisture molecules are easily combined with the hydrophilic groups of the ester oil molecules [11]. Therefore, for preparing vegetable insulating oil, finding a method to effectively decrease the moisture content is very important. Although the moisture content of JNIO (193 ppm) complies with the requirements of ASTM D6871-17 and GB2536-90 (≤200 ppm), it may still be possible to reduce the moisture content even further. In the process of the preparation of JNIO, activated clay, aluminum oxide, and reduced pressure heating treatments all contributed to decrease the water content of the oil. Previous research has shown that by vacuum drying for 120 h, the water content in the vegetable oil was reduced to about 50 ppm, and that the extended drying time did not change the electrical properties [35]. Therefore, for *Jatropha curcas* seed oil, considering that the reduced pressure heat treatment time in this research process was 3 h, extending the drying time to reduce the moisture content may be a good option. In addition, using molecular sieve 3A to reduce the moisture content of JNIO may also be effective, because some studies have shown a greatly reduced water content of Jatropha methyl ester oil (from 1159.78 ppm to 6491 ppm) upon the addition of molecular sieve 3A [21].

The properties of the related *Jatropha curcas* methyl ester oil (JMEO) as a vegetable insulating oil have been studied [21]. The main differences between JNIO and JMEO are their preparation processes, and the physicochemical properties including flash point, pour point, and kinematic viscosity. The preparation process of JNIO is simpler than that of JMEO, and it avoids the esterification process catalyzed by potassium hydroxide (KOH) for preparing JMEO. In addition, JNIO's flash point (295 °C) and pour point (−10 °C) are better than those of JMEO (191 °C and 0 °C, respectively), and in fact, JMEO does not meet the ASTM D6871-17 standard requirements for these two parameters (>275 °C and ≤−10 °C, respectively). On the other hand, the water content (6491 ppm) and kinematic viscosity (1045 $mm^2/s$ at 40 °C) of JMEO were better than JNIO (193 ppm, 3283 $mm^2/s$ at 40 °C, respectively).

## 5. Conclusions

A high-quality natural vegetable insulation oil can be made from *Jatropha curcas* seed oil by the six-step process designed in this study. The vegetable insulation oil met all standard requirements for vegetable insulating oils. The processes were simple, efficient, and cost-effective. A cold treatment was included to reduce the saturated fatty acid content, improving the flash point. A combination of alkali refining, active clay, and aluminum oxide treatments contributed to a low acid value and improved color of the vegetable oils. In particular, the optimization of the alkali refining greatly reduced the

acid value by gentle, low energy room temperature processing. The active clay and aluminum oxide treatments, together with the high-temperature decompression treatment, caused a decrease in the moisture content of the oil. Comprehensively, all parameters of the *Jatropha curcas* seed oil for the vegetable insulation oil were improved by this process. Moreover, the natural vegetable insulating oil made in this study can also be used as raw material for the preparation of improved plant insulating oils and other specialized oil products. Therefore, this study promotes the utilization of *Jatropha curcas* as a non-food resource.

**Supplementary Materials:** The following are available online at http://www.mdpi.com/1996-1073/13/17/4319/s1, Figure S1: The *Jatropha curcas* seed oil treated at 4 °C for 48 h, Figure S2: Mass spectrum of fatty acid analysis in CJCO and JNIO, Table S1: Detection processes of fatty acids in CJCO and JNIO, Table S2: Descriptive Statistics of CJCO and JNIO properties, Table S3: ANOVA of differences in physical and chemical properties between CJCO and JNIO.

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