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Green Refining of Waste Lubricating Oil: A China Perspective

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Presently, many regeneration processes of waste lubricating oil, such as catalytic hydrogenation, are available. However, some of these processes are highly costly and not suitable for Chinese economic conditions, and some may produce contaminated impurities such as acid slag, which cannot meet environmental protection requirements. This study aims to develop a green process for the regeneration of waste lubricating oil into a base oil, which should meet the requirements of green chemistry, have the characteristics of simple operation, low cost, less pollution and high recovery rate, and turn wastes into renewable resources. The new process developed via this research has three stages. First, mechanical and large particle impurities in the waste lubricating oil were removed by pretreatment. Second, most of the colloid and asphaltene were removed by thermal extraction and sedimentation. Finally, the activated bleaching earth was used to further purify the waste lubricating oil. The performance evaluation of the finally obtained lubricating base oil conformed to the standard of the HVI-100 lubricating oil. The total recovery rate of the process was about 63.5%.

Keywords: Waste lubricating oil; Green refining; Pretreatment; Thermal extraction and sedimentation; Response surface methodology

1. Introduction

1.1 Overview

The development of the lubricant industry is directly linked to the development of the transportation industry and has a greater impact on the ups and downs of the macro economy. From 2000 to 2010, along with the rapid development of China's economy, the scale of the lubricant market has continued to grow rapidly. In 2010, the consumption of lubricating oil reached 8.5-8.8 million tons/year [1]. Since 2013, China's economic development has entered a steady growth phase. Many industries including the lubricant industry have been affected and showed a gradual slowdown in development. The lubricant industry in China has been continuously eliminating outdated products and switching to high-end products with the extended oil-changing period.

In 2017, the domestic demand for automotive lubricants was 3.572 million tons, accounting for 53% of the total lubricant market, and the share of industrial ones was reduced to 28% [2]. Lubricant or motor oil is a consumer product for all vehicles, which

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needs to be replaced every 5000-7000 km or 3000-5000 miles. Correspondingly, the amount of waste lubricating oil produced in China is proportional to this market demand (Figure 1) [3].

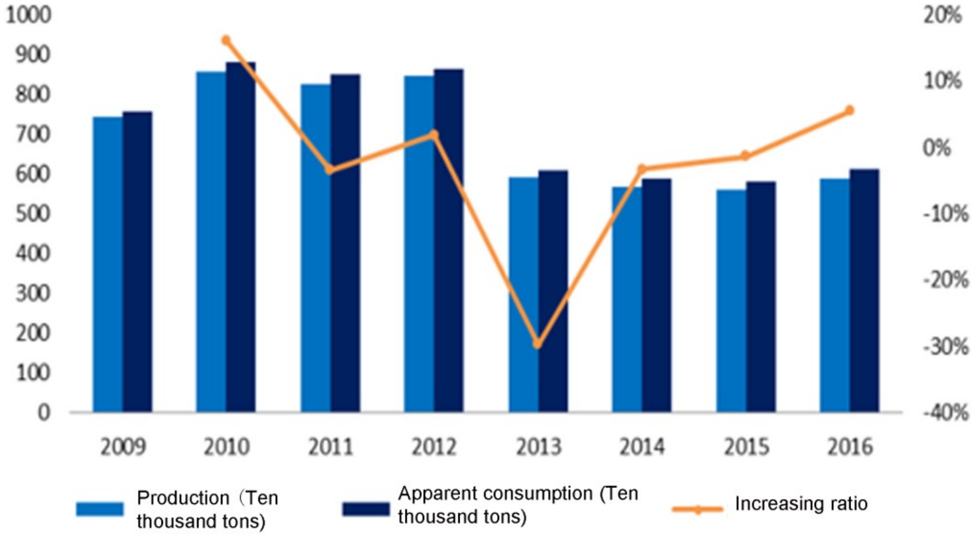

Figure 1. Lubricant production and apparent consumption (Adapted from http://www.chyxx.com/industry/201708/555496.html, Public Domain)

During the use, some hydrocarbons in the lubricating oil will react with oxygen at high temperature and pressure to produce peroxides, carboxylic acids, alcohols, ketones, asphaltenes, gums, etc. The color of the oil becomes darker [4]. The produced acids cause the increased acidity of the lubricating oil, which can corrode easily the mechanical parts, resulting in the reduced service life. Meanwhile, incomplete combustion in the engine produces carbon residue, and mutual friction between mechanical parts tends to generate minute metal powder particles. Dust, moisture, and soil in the environment are also components of impurities in the lubricating oil. These impurities may form deposits on the surface of mechanical parts, which are liable to cause engine blockage and reduce its service life [5]. When the metamorphic portion reaches 5-10%, the lubricating oil must be disposed of as waste lubricating oil.

Waste lubricating oil is mainly composed of base oil, asphaltene, colloids, oxides, carbon residue, sludge, moisture, and other components [6]. The main oxides in waste lubricating oils are carboxylic acids, carboxylates, and aldehydes [7]. Metals such as Ni, Sn, Cr, and Fe are produced by corrosion of the engine, while metals like Ag, B, Ba, Ca, Cd, Co, Hg, Mg, Mo, P, Pb, Sb, Se, Ti, Zn, etc. are derived from the additives [8]. Waste lubricating oil is mainly processed in the following ways [9-12]:

(1) Directly discarded as wastes;
(2) Catalytic cracking;
(3) Used as a fuel after removing heavy metals;
(4) Raw materials for road oiling;
(5) Regeneration into a lubricating base oil

Among them, the regeneration of waste lubricating oil into a base oil has better economics and reflects the concept of green chemistry. As early as the beginning of the 20th century, the recycling process of waste lubricating oil was commercialized on a large scale in the United States and Germany. China has carried out research on the regeneration process of waste lubricating oil since the 1940s. So far, most of China's manufacturers are still using the sulfuric acid-bleaching earth process, which was mainly used in the early 1960s by other countries. Currently, petroleum has become an increasingly important resource, and environmental protection became an unavoidable issue. Many countries have raised the standard for the recycling of waste lubricating oil and issued the policies to encourage the recycling.

1.2 Regeneration Processes of Waste Lubricating Oil

1.2.1 Sulfuric Acid - Bleaching Earth Process

The sulfuric acid-bleaching earth process (Figure 2) utilizes the principle that properties of oil and water are incompatible, and the boiling point of water is lower than that of waste oil. [13, 14]. The water is first removed by heating. Then, concentrated sulfuric acid is used to oxidize organic matters, which is removed by washing with alkali. The active bleaching earth absorbs impurities such as pigments. Final filtration could remove solid residues.

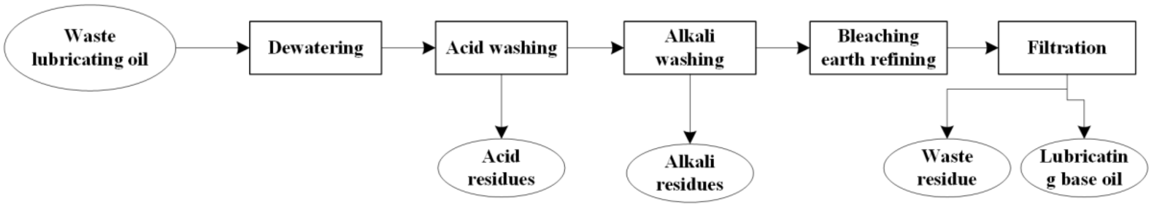

Figure 2. Sulfuric acid- bleaching earth process

1.2.2 Hydrotreating

The hydrotreating process has advantages including the high oil yield, simple operation and well-established process, but its cost is relatively high [15]. It is widely used abroad. Under certain conditions of catalysis and pressure, impurity elements such as N, O, S, etc. in the waste oil are removed as their hydrides such as NH_3, H_2O, H_2S, etc [16]. The olefin and aromatic hydrocarbons can be partially hydrogenated to improve the quality of the oil.

1.2.3 Molecular Distillation

The mass transfer and heat transfer of the device for molecular distillation is very efficient, and the material requirements are relatively demanding. It is necessary to remove the pollutants such as mechanical impurities in the waste lubricating oil before further purification (Figure 3). The quality of the lubricating oil obtained by the molecular distillation process [4, 17] satisfies the national standard of the lubricating base oil. But the distillation process has higher requirements on the environment and requires a higher degree of vacuum. The size of the reactor is fixed and has certain limitations.

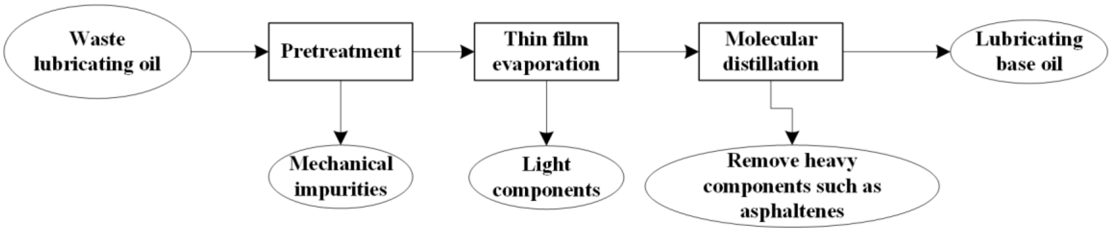

Figure 3. Molecular distillation process

1.2.4 Solvent Refining

Solvent refining is one of the commonly used methods for refining petroleum products. Figure 4 shows the solvent refining process flow chart. It utilizes different solubility of components of the lubricating oil in a solvent to remove impurities and non-ideal component [18, 19].

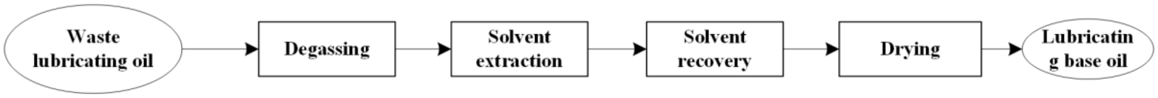

Figure 4. Solvent refining process

The degassing process removes oxygen dissolved in the oil to prevent the solvent from being slowly oxidized. After solvent extraction, two layers are formed. The lower layer is a mixture of solvent and impurities, and the upper layer mainly contains refined oil. The energy consumption of the solvent recovery part accounts for 80% of the process. The solvent in the upper layer is removed in the evaporation stripper, and then dewatered to obtain the lubricating base oil.

1.2.4 Membrane Separation

Recently, membrane separation process [20] has been proposed as new mean to regenerate waste lubricating oil. Because of its high cost, related studies remain at the research stage. Figure 5 is a flow chart of the membrane separation process.

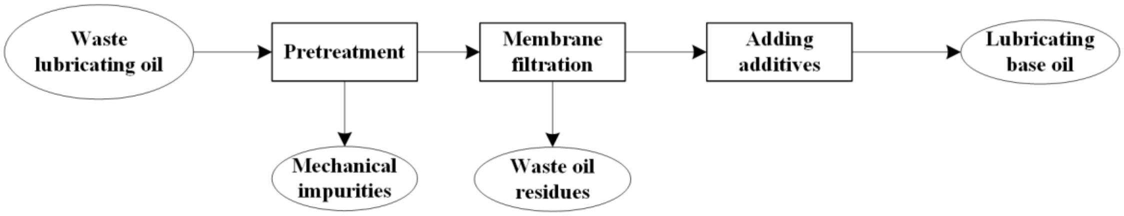

Figure 5. Membrane separation process

1.3 The Choice of Waste Lubricating Oil Regeneration Process in China

The hydrofining process is used by many developed countries for waste lubricant treatment. However, the investment in hydrotreating process is relatively high. For the time being, there is no waste lubricating oil recycling plant in China that uses hydrotreating process.

The problems with the solvent refining process are the loss of the solvent, the low base oil yield, and the high sulfur content of the base oil product. At present, the solvent formulation has been improved, and the consumption of the solvent has been significantly reduced. Compared with the hydrofining process, the investment cost of the solvent refining plant is lower, and the technology is more mature.

The sulfuric acid-bleaching earth process is mature and requires relatively low investment cost. Many domestic refineries are using this method. However, the residual acid slag and alkali slag have a great impact on the environment. With the gradual strict environmental policy in China, the process will gradually be eliminated by the market.

2. Materials and Methods

2.1 Materials

The waste lubricating oil used in this study was provided by Tianan Chemical Co., Ltd (Macheng, Hubei Province, China). The color of the waste lubricating oil is carbon black, which is very thick at room temperature, and has an unpleasant pungent odor. Its properties are listed in Table 1.

Table 1. Performance evaluation results of the waste lubricating oil

Property	Value	Method
Viscosity (mPa·s)/40℃	78.50	LVDV-1 viscometer
Acid value (mg·KOH/g)	0.59	GB/T264-1983
Moisture (%)	0.55	GB/T260-2016
Mechanical impurity (%)	1.42	GB/T511-2010
Ash (%)	0.57	GB/T508-1985

The chemical reagents of isopropanol, n-butanol, isobutanol, and butanone were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China).

Table 2. Properties and prices of alcohol and ketone solvents*

Solvent	Isopropanol	n-butanol	Isobutanol	Butanone
Boiling point (℃)	82.5	117.7	108.1	75.9
Freezing point (℃)	-88.5	-90.2	-108.0	-85.9
Price (￥YMB/ton)	7800	7600	7400	7500

*All solvents listed in Table 2 have intermediate toxicity and stable at room temperature.

The activated bleaching earth (also called activated clay) was provided by Zhengzhou Fengquan Water Treatment Plant (Henan, China). The physical and chemical properties of the products are shown in Table 3.

Table 3. Physical properties and composition of the activated bleaching earth

Property	Index	Composition	Weight %
Appearance	White fine powder	SiO_2	62.34
Surface area	＞130m^2/g	Al_2O_3	17.24
Moisture	＜5%	Fe_2O_3	2.73
Decolorization	＞154	FeO	0.12
Heavy metal content	＜10mg/kg	TiO	0.15
Arsenic content	＜3mg/kg	CaO	2.09
Free acid	＜0.1%	MgO	5.44
Granularity (<0.076mm)	＞95%	MnO	0.15

The activated bleaching earth was dried in an oven at 110°C for 2 hours prior to the experiments.

2.2 Characterization of Lubricating Oil

Viscosity was measured by using the LVDV-1 rotary electronic viscometer (Shanghai Jingtian Electronic Instrument Co., Ltd, China). The acid value was measured by the indicator titration method according to the national standard GB/T264-1983. The moisture content of the oil was measured by the distillation method according to the national standard GB/T260-2016, while the ash content was determined according to the national standard GB/T508-85.

Mechanical impurities in the oil refer to solid small particles that cannot be dissolved in a solvent such as toluene or gasoline. To measure mechanical impurities, the oil was first dissolved in the toluene, which was followed by filtration (GB/T511-2010).

Color of the oil (*i.e,* chromaticity) could reflect the degree of refinement and its stability. A lighter oil color generally indicates a higher degree of purification of the waste lubricating oil [21]. The color of the recycled oil was determined according to ASTM D1500-12. The sample oil and standard swatches are placed under a certain light source, and the color is compared to determine the ASTM color of the oil.

2.3 Thermal Settlement

Thermal settling is used as a pretreatment method, which is an effective physical method of removing the mechanical impurity from a large volume of waste lubricating oil, which has the advantages of low energy consumption and easy handling. For each test, about 60 ml of waste lubricating oil was added to a 100 ml beaker, which was placed into an oven at 40-80 °C for 3-24 h. The mechanical impurity content was measured after each test.

2.4 Thermal Extraction and Sedimentation

Solvents such as short-chain alcohols and ketones have poor dissolving ability for substances with relatively high molecular weight such as asphaltenes, colloids, gray matter and additives, but strong dissolving ability for oil components. Heating can reduce the viscosity of the waste lubricating oil, making the extraction process more complete. Therefore, high molecular weight substances settle down during thermal extraction and sedimentation. Commonly used extractants are polar solvents such as C3 or C4 alcohols and butanone, which showed good solubility to the waste lubricating base oil [22, 23].

Considering the cost, solvents of isopropanol, n-butanol, isobutanol, and butanone were used for comparison.

2.5 Response Surface Methodology for Thermal Extraction and Sedimentation using Double Solvents

To further analyze the effect of using two extraction solvents on thermal extraction flocculation, the response surface methodology was used to design the experiments (Table 4).

Table 4. Box-Behnken response surface test design factors and levels

Level	A: solvent to oil	B: solvent ratio	C: Temperature (°C)
-1	2:1	1:4	30
0	4:1	2.13:1	50
1	6:1	4:1	70

The yield of waste lubricating oil is the response value, and three factors of A, B, and C are the ratio of solvent to oil (A: the volume ratio of the extractant: mass of crude lubricating oil), the solvent ratio (B: the volume ratio of n-butanol: isobutanol), and temperature (C), respectively. This three-factor three-level response surface analysis included 17 extraction experiments. Each extraction experiment was performed for 45 min, and then the solvent was distilled under reduced pressure at 70°C.

2.6 Bleaching with Activated Bleaching Earth

A certain amount of waste lubricating oil after extraction and sedimentation was accurately weighed into a three-necked flask. A relatively excess amount of activated bleaching earth was added to determine the effect of temperature on the degree of refining. After finding the optimum temperature, the amount of activated bleaching earth was adjusted to select the best proportion of activated clay. The stirring speed was fixed at 300 rpm, and the stirring time was fixed at 30 min. After the stirring, the upper layer solution in the three-necked flask was placed in a beaker that was placed in a constant temperature drying oven at 70°C for 6-8 hours. The final upper yellow liquid was the purified waste lubricating base oil.

The most intuitive color of waste lubricating oil is used as the evaluation standard. According to ASTM D1500-12, the final result is represented by the color code (*i.e.,* chromaticity) of 0.5-8. When the color of the oil exceeds 8 or more, it is necessary to add kerosene and then measure. This experiment simplifies the measurement method and treats it with 8 when the chromaticity exceeds 8. The smaller the number, the lighter the color of the oil and the higher the degree of refining, and vice versa.

3. Results and Discussion

3.1 Pretreatment with Thermal Settlement

The contents of the mechanical impurities of the waste lubricating oil after thermal settling are shown in Figure 6. As the settling time increased, the five curves gradually become flat. After 24-h, it can be regarded as a horizontal line. Therefore, 24-h was considered as the optimal settling time.

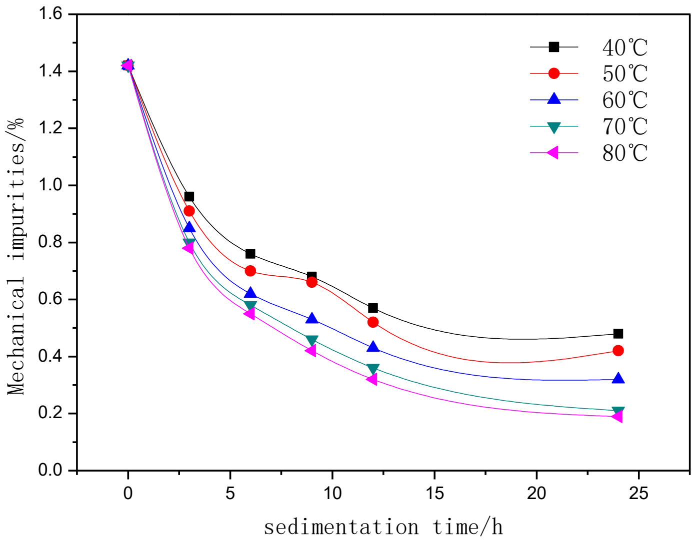

Figure 6. The effects of thermal settling temperature and time

The increase in the pretreatment temperature can significantly reduce the amount of mechanical impurities in the waste lubricating oil. At 70°C and 80°C, the change of mechanical impurity content in waste lubricating oil was small. Because a higher temperature can increase the cost and the oxidative deterioration of oil, 70°C was considered as the optimal settling temperature. Subsequent tests used the waste lubricating oil after thermal settling at 70°C as experimental materials.

3.2 Thermal Extraction and Sedimentation using Single Solvent

Four solvents of isopropanol, n-butanol, isobutanol, and butanone were tested. The solvent was mixed with the waste lubricating oil in the ratio of 1:1, 2:1, 3:1, 4:1, and 6:1, and then heated at 50°C for 30 min. The yield of waste lubricating oil was measured after purification. It was found that butanone had no obvious extraction effect and was basically miscible with lubricating oil. Figure 7 shows the relationship between the solvent to oil ratio and the yield of the recycled waste lubricating oil. When the ratio was higher than 3:1, there is no obvious yield change.

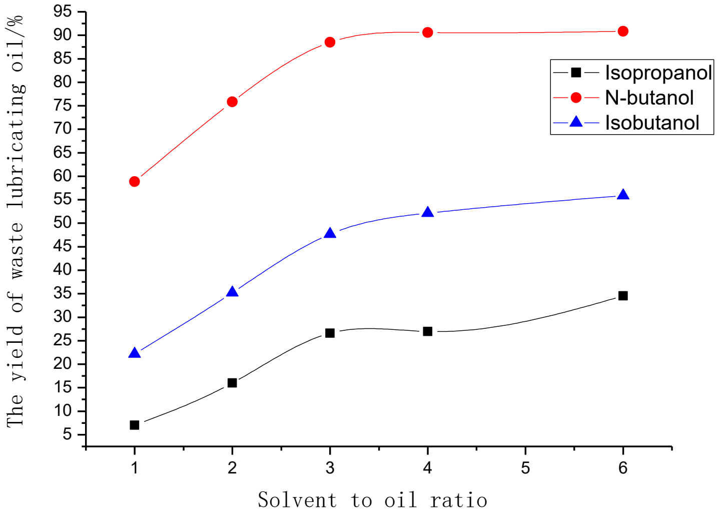

Figure 7. Effect of single solvent extraction on the yield of recovered lubricating oil

3.3 Thermal Extraction and Sedimentation using Double Solvents

The selection of two solvents in the thermal extraction and sedimentation tests was based on the conclusions of single-solvent extraction. N-butanol has a strong ability to dissolve effective components of the waste lubricating oil, and isobutanol can reduce the ash content. Therefore, a combination of n-butanol and isobutanol was selected as the extractant.

Table 5. Analysis of variance of regression equation

Source of variance	sum of squares	Degree of freedom	Mean squares	F value	P value*	Significance
Model	4928.83	9	555.84	32.77	<0.0001	significant
A	3262.30	1	3262.30	195.22	<0.0001	significant
B	425.01	1	425.01	25.43	0.0015	significant
C	31.13	1	31.13	1.86	0.2146	
AB	48.30	1	48.30	2.89	0.1329	
AC	59.99	1	59.99	3.59	0.1000	
BC	45.77	1	45.77	2.74	0.1419	
A^2	323.95	1	323.95	19.39	0.0031	significant
B^2	188.81	1	188.81	11.30	0.0121	significant
C^2	436.26	1	436.26	26.11	0.0014	significant
Residual	116.97	7	16.71			
Lack of Fit	86.04	3	28.68	3.71	0.1190	Not significant
Pure error	30.94	4	7.73			
Cor. total	5045.81	16				
		$R^2=0.9768$		$R_{ADJ}^2=0.9470$		

* The cut-off value for P value was 0.05.

The results are shown in Figure 8. The multivariate quadratic regression fitting was performed, and the regression model of the process parameters was established. The coded regression equation for the specific yield Y is:

$$Y(\%)=82.95+20.19\times A+7.29\times B+1.97\times C-3.48\times A\times B-3.87\times A\times C-3.38\times B\times C-8.77\times A^2-6.70\times B^2-10.18\times C^2 \quad \text{(Equation 1)}$$

A, B, and C, respectively, correspond to the coding values of the three factors, and the values are in the range of -1 to 1. Therefore, the regression equation can be used instead of the test real point to calculate the experimental results.

The larger the F value of the dependent variable, the greater the effect on the yield of the waste lubricating oil. It can be seen from Table 5 that the solvent to oil ratio (A) has the greatest influence on the yield of the waste lubricating oil, and the solvent ratio (B) has the second influence on the waste lubricating oil yield, and the temperature C has the least influence on the waste lubricating oil yield.

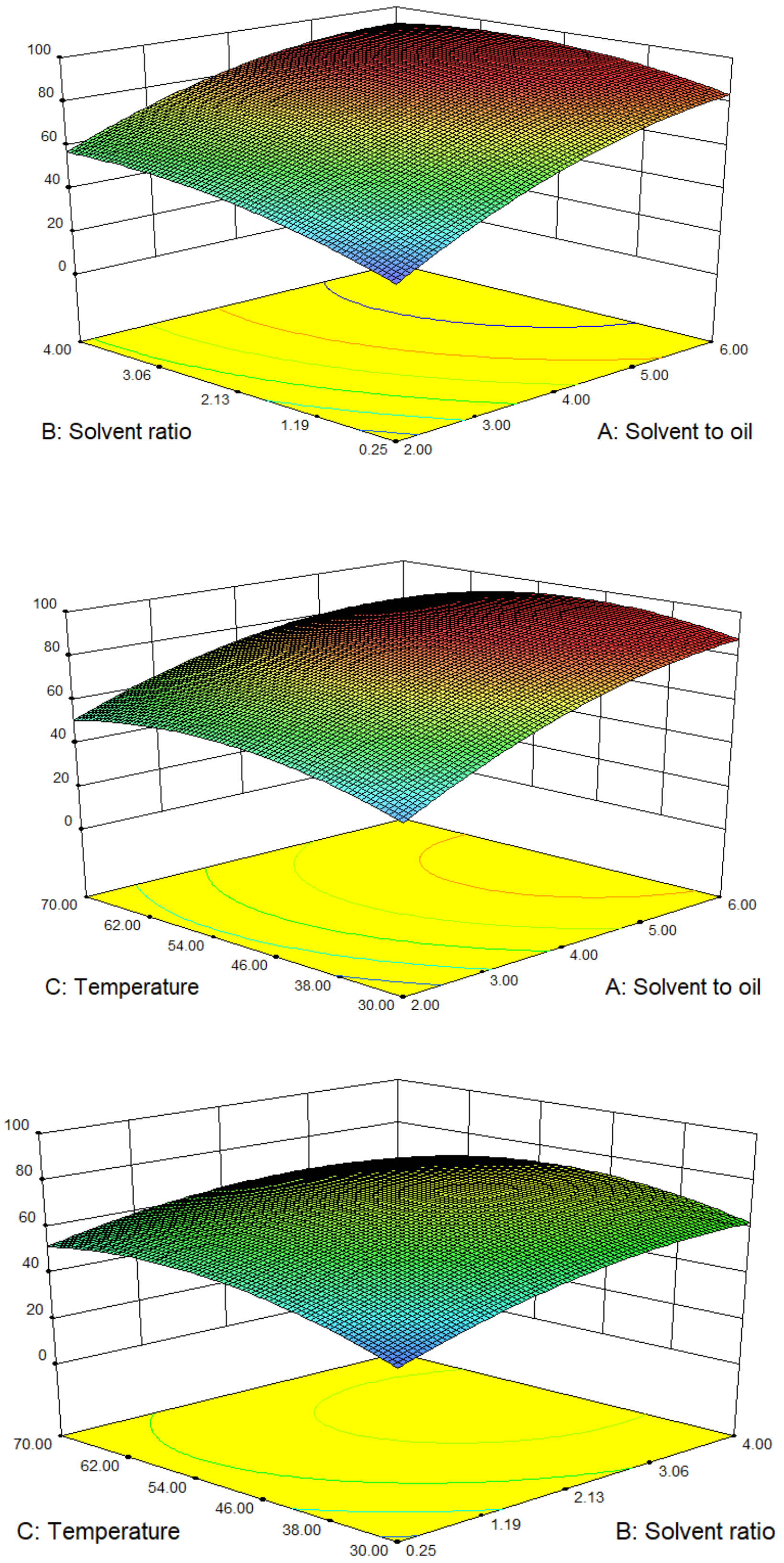

Figure 8. 3D response surface map of the thermal extraction and sedimentation using double solvents

Table 6. Simulated optimal values for the two solvents

Number	Solvent:Oil	Solvent ratio	Temperature /°C	Yield/%	Desirability	
1	6	2.7	47.09	95.1234	0.364	Selected
2	6	2.7	46.99	95.1227	0.364	
3	6	2.7	47.94	95.1227	0.364	
4	6	2.7	47.29	95.1222	0.364	
5	6	2.7	46.85	94.1213	0.364	

Based on Equation 1, the optimal conditions were calculated (Table 6). The optimal conditions were the ratio of solvent: waste lubricating oil of 6:1, n-butanol: isobutanol (vol ratio) of 2.7:1, temperature of 47.1°C, and stirring time of 45 min.

Table 7. Verification of the optimal conditions for the two solvents

Number	Solvent:Oil	Solvent ratio	Temperature /°C	Yield /%
1	6	2.7	47.1	91.56
2	6	2.7	47.1	89.65

The optimal conditions were further experimentally verified. The yield of the waste lubricating oil product obtained under the above conditions was 90.60 ± 1.35%, and the viscosity at 40°C was 50.2 mPa·s, and the ash content was 0.23%. Intuitively, the appearance color is lighter than the single extractant n-butanol as a solvent but still reddish brown. The acid value was 0.1 mg·KOH/g.

Compared with the single extractant n-butanol, the yield of the recovered waste lubricating oil of the double-solvent extraction was slightly increased and the ash content decreased. In this test, the ratio of the solvent to the lubricating oil has the greatest influence, the solvent ratio has the secondary influence on the yield of the waste lubricating oil, and the temperature has the least effect. The optimal conditions experimentally verified were close to the simulated values, indicating that the method has certain persuasive power, and there are certain rules for each factor and response value.

3.4 Bleaching with Activated Bleaching Earth

The waste lubricating oil treated with thermal extraction and sedimentation was further purified by using activated bleaching earth. As shown in Figure 9, when the temperature was below 110°C, the chromaticity of the waste lubricating oil did not change too much, and the activated bleaching earth obtained was dark yellow and did not play an adsorption role. When the bleaching temperature rose to 120°C, the situation changed significantly, and the chromaticity of the waste lubricating oil dropped sharply. The bleaching earth obtained at this time was dark brown, and the colored materials in the oil were basically taken out. When the temperature was raised to 130 ° C, the oil is already clear. Therefore, the choice of 125°C for the bleaching temperature, not only can get better oil, but also reduce heat and save costs.

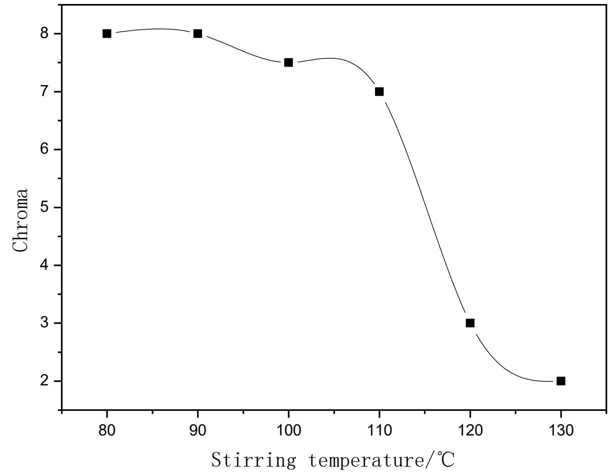

Figure 9. Relationship between temperature and oil color under excessive bleaching earth

After determining the bleaching temperature to be 125°C, the relationship between the amount of bleaching earth added (% of white soil quality: oil quality) and the color and the recovery rate of the final base oil were determined (Figure 10).

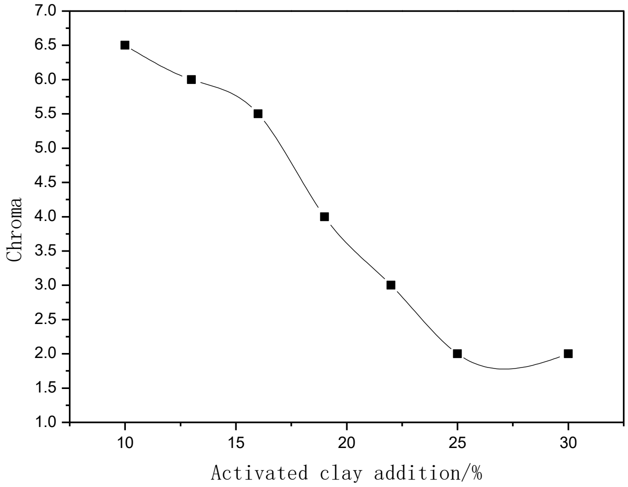

Figure 10. Effect of the amount of activated bleaching earth on the chromaticity

There is a strong correlation between the amount of activated bleaching earth and the degree of refining of the oil. As the amount of activated bleaching earth increased, the color of the waste lubricating oil gradually decreased. When the amount of activated bleaching earth reached 25%, the color of lubricating oil was basically unchanged and became a relatively pure yellow transparent liquid. Figure 11 shows the change of appearance chromaticity of the waste lubricating oil with the addition of activated bleaching earth.

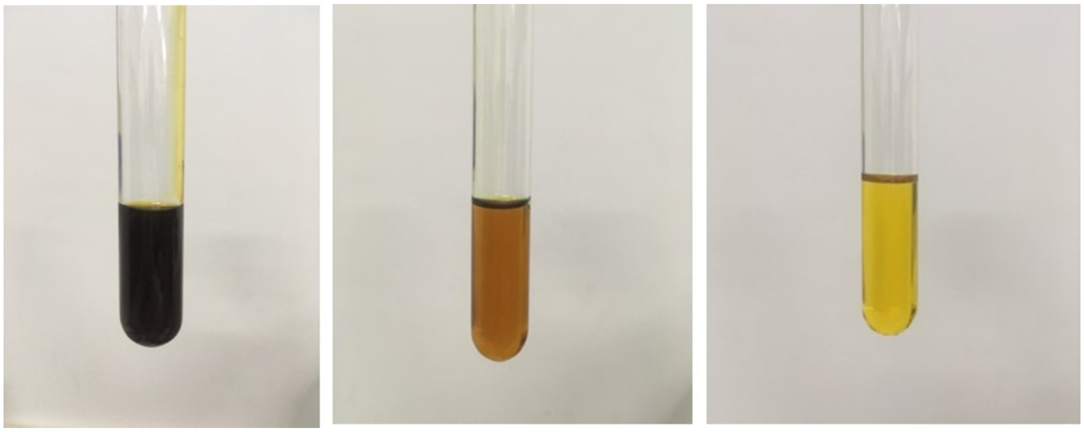

Figure 11. Appearance chromaticity change of waste lubricating oil
Left: the waste lubricating oil after thermal extraction and sedimentation; Middle: Treated with 15 wt% of activated bleaching earth; Right: Treated with 25 wt% of activated bleaching earth

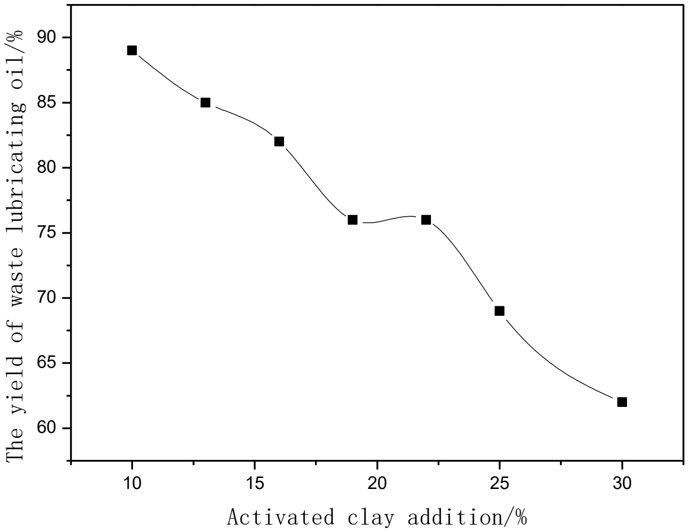

Figure 12. Effect of the amount of activated bleaching earth on the yield of waste lubricating oil

As can be seen from Figure 12, as the amount of activated bleaching earth increased, the yield of waste lubricating oil has been decreasing. On the one hand, too much bleaching earth will absorb a part of the oil, resulting in a decrease in the amount of recycled oil. On the other hand, due to the rise of the bleaching earth, the bleaching earth in the oil is difficult to settle in the process of the final thermal settling, and the separation speed is slow, resulting in oil turbidity and lowering the yield of the base oil.

In summary, in order to achieve a better purification effect, the amount of bleaching earth added was 25 wt%, and the yield of refined lubricating oil was 69%.

Table 8. Properties of recycled lubricating oil

Properties	Value
Dynamic viscosity@40℃ (mPa•s)	24.0
Kinematic viscosity@40℃ (mm^2/s)	21.8
Viscosity index	98.2
Acid value (mg•KOH/g)	0.04
Moisture (%)	0
Mechanical impurities (%)	0
Ash content (%)	0.02
Pour point (℃)	-14

Table 9. HVI-100 Lubricant Standard

Kinematic viscosity @40℃ (mm^2/s)	Appearance	Chromaticity	Viscosity index	Pour point (℃)	Acid value (mg•KOH/g)	Ash content (%)
20~22	Transparent	1	100	<-9	<0.02	<1.5

Comparing Table 8 with Tables 9, it is found that the finally obtained lubricating oil sample basically conforms to the standard of the grade HVI-100 lubricating oil.

4. CONCLUSIONS

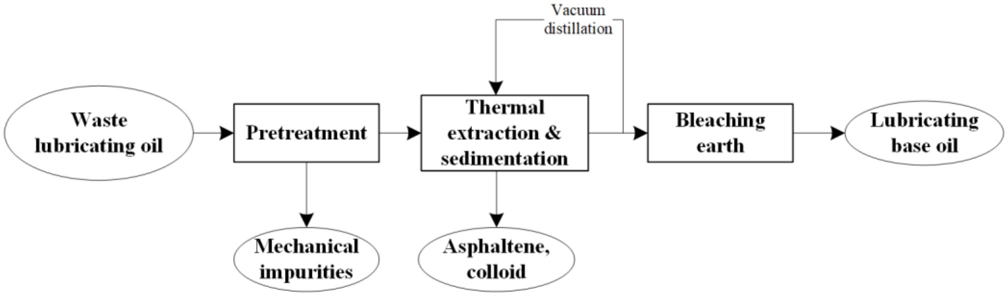

Figure 13. Green refining process of waste lubricating oil developed in this study

This paper proposes a new process for the regeneration of waste lubricating oil into lubricating base oil (Figure 13). The process has three stages. The first pretreatment stage removes mechanical impurities and large particulate matter from the waste lubricating oil via thermal settling. By comparing the effects of different temperatures and settling times on mechanical impurities, a reasonable settling time of 24 h and an optimum settling temperature of 70°C were found. The second stage is a thermal extraction and sedimentation process to remove impurities such as asphaltenes and gums. The thermal extraction and sedimentation process was carried out using a single solvent of isopropanol, n-butanol or isobutanol. It was found that the most economical ratio of the ratio of the solvent to oil was 3:1, the stirring time was 30 min, and the operation temperature was around 50°C. When using double solvents of n-butanol and isobutanol, the optimal conditions were the solvent to oil ratio of 6:1, the solvent ratio of 2.71:1, and the temperature of 47.1 °C. Under these conditions, the yield of the oil experimentally obtained was 90.60±1.35%. The third stage is bleaching with the activated bleaching earth, which adsorbs the pigments and the impurities such as colloid and asphaltene. The optimal temperature was determined as 125°C when the mass of bleaching earth was 25 wt% of the oil mass. The refined lubricating oil is basically in accordance with the standard of the HVI-100 lubricating base oil. The total recovery rate of the process was about 63.5%.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this paper.

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