TẠP CHÍ KHOA HỌC
TRƯỜNG ĐẠI HỌC QUY NHƠN

Nghiên cứu điều chế mỡ bôi trơn từ dầu nhờn thải tái sinh

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TÓM TẮT

Mỡ bôi trơn được điều chế bằng cách tổng hợp từ dầu khoáng và chất làm đặc. Trong nghiên cứu này, mỡ bôi trơn được làm từ chất làm đặc như NaOH, dầu lạc (PO) được kết hợp với dầu nhờn thải tái sinh (RLO) trong phòng thí nghiệm. Kết quả cho thấy tỷ lệ phù hợp của NaOH/PO/RLO là 3,7/13/18, thời gian và nhiệt độ tối ưu cho tổng hợp mỡ bôi trơn là khoảng 55 phút và 105-110 ^{o}C. Mỡ thành phẩm thu được có nhiệt độ nhỏ giọt và độ xuyên kim là 116 ^{o}C và 176 mm.

Từ khóa: *Mỡ bôi trơn, dầu nhờn thải tái sinh, dầu lạc.*

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A study on the preparation of greases from recycling of used lubricating oil

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ABSTRACT

Greases were made by combining a fluid lubricant and a thickener. In this research, the grease was synthesized from thickener materials such as NaOH, and peanut oil (PO), which dispersed in recycled lubricating oil (RLO) in the laboratory. The result shows that the suitable ratio of NaOH/PO/RLO was 3,7/13/18; the achieved time and temperature for synthesizing grease were about 55 minutes and 105 - 110^{o}C, respectively. It was demonstrated that the optimal grease dropping point and the needle penetration were 116^{o}C and 176 mm, respectively.

Keywords: *Greases, recycled lubricating oil, peanut oil.*

1. INTRODUCTION

Nowadays, lubricating oil plays an important role in industrial manufactory and daily life. Application of lubricants is to lubricate moving parts, to reduce friction and to reduce equipment's wear. However, lubricants have some drawbacks such that they cannot lubricate some positions such as bearings at extremely high temperature, or equipment at large load of working condition. As a result, lubricants are replaced by greases. Today, greases are used with relatively large quantities, and they have properties and effects that lubricating oil does not have.[1-6] There are many sources of materials and methods to manufacture greases for various usages. In addition, using recycled lubricants to replace mineral oil for making grease is necessary. Currently, the process of manufacturing grease from recycled lubricants has not been produced on an industrial scale. In fact, this process has only been studied in laboratory and done manually. Utilizing lubricant or waste is denatured and deteriorated by the oxidation. There are valuable hydrocarbons remaining in the composition of discarded waste lubricant, which will have an impact on the environment after releasing without any treatment. Many methods have been applied to recycle them regarding to reusing this source and environmental issues. After regenerating, lubricant is provided with additives to be reused as lubricating oil.[7-12] However, recycled lubricants have some drawbacks such as color, viscosity and some other parameters are still lower than TCVN 8939-9: 2011, so they can only be used for agricultural machinery. To solve these shortcomings, recycled lubricating oil was studied from the aspect of its combination with secondary peanut oil to make grease.

2. EXPERIMENT

2.1. Raw materials: Recycled lubricating oil, peanut oil (Quy Nhon)

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2.2. Chemical: NaOH 96% (China), $BaCl_2$ (China), HCl 36% (Vietnam), KOH 85% (China), phenolphtalein solution 1%, ethyl alcohol 99.5% (Vietnam), glycerin.

3. RESULTS AND DISCUSSION

3.1. Recycle waste lubricants

The waste lubricants are recycled by acid treatment method and the basic four-step procedure as follows:

Step 1: Dehydrate and remove mechanical impurities

The vehicle waste oil is collected and filtered by a vacuum filter to remove large-sized impurities. It is then heated at 110^{o}C for 15 minutes to dehydrate and evaporate the light fuel in the oil.

Step 2: Treat the waste lubricants with acetic acid

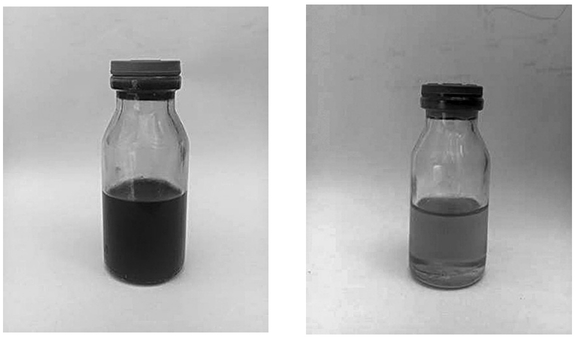
100 ml of the dehydrated waste oil (in Step 1) is poured into a 250 ml beaker under at 50 ^{o}C. Then 17.5 ml of acetic acid is added at the concentration ≥ 99.5%. The mixture sample is stirred for an hour at 50 ^{o}C. After that, the sample is cooled down to room temperature and centrifuged for 20 minutes at rotating speed of 6,000 rpm to separate the residue from the waste oil. The results show that after recycling this oil has a light brown color and its viscosity was 54.5 cSt at 40 ^{o}C.

Step 3: Neutralize acid with NaOH solution

80 ml of acid-treated oil is put into a 250 ml beaker, and stirred under to the heat of 80 ^{o}C, then 7 ml of 45% NaOH solution is added into the prepared mixture. After 30 minutes, the results reveal that after recycling the oil sample is light reddish brown (Figure 1), and its viscosity is 78.4 cSt at 40 ^{o}C.

Step 4: Clean oil with adsorbent

50 grams of Binh Dinh clay mineral material is added into 100 ml of base treated oil. The mixture is homogeneously stirred for 30 minutes at 110 ^{o}C. The obtained product -collected using vacuum filtrationis named recycled lubricating oil.



(a) (b)

Figure 1. Photo images of (a) Motorcycle waste lubricant, and (b) Lubricants after regeneration

3.2. Produce lubricants from recycled lubricating oil

The production of lubricants from regenerated waste lubricants with a thickener using the saponification reaction between NaOH and peanut oil is carried out as follows:

Step 1: The raw materials are prepared including recycled lubricating oil, NaOH 30% and peanut oil.

Step 2: The optimal NaOH/PO ratio is investigated in reaction time and reaction temperature to create a thickener.

Step 3: The ingredients are added into a 250 ml glass beaker as summarized in Table 1.

Table 1. Ingredients raw materials for the manufacture of lubricants

Recycled Lubricating oil (grams)	NaOH 30% (ml)	Peanut oil (grams)
18	3.7	13

Step 4: The prepared beaker is heated in glycerin bath using electric stove for the saponification reaction to occur at 85 ÷ 90 ^{o}C for 45 minutes, and keeping stirring during greasing with stirring speed 60 ÷ 70 rpm.

Step 5: To get the stable fat mass homogenization phase, the mixture solution should maintain from 85 ÷ 90 ^{o}C in Step 4 to 105 ÷ 110 ^{o}C for varying duration (10 min), the consistency and color of the grease are observed and the electric stove is turned off.

3.3. Investigate the saponification process experimentally to create a thickener

3.3.1. Optimal ratio between NaOH and peanut oil (NaOH/PO)

The experiment is performed for 40 minutes at 80 ÷ 85 ^{o}C, and kept stirring at 60 ÷ 70 rpm with specific NaOH/PO ratios. The result is shown in Table 2.

Table 2. Optimal ratio between NaOH and peanut oil (NaOH/PO)

Ratio NaOH/PO (ml/g)	Value balance NaOH (%)	Comments Result
3.0/13	0	Liquid, light white, oil film
3.7/13	0.078	Solid, light yellow
4.0/13	0.115	Solid, light yellow
4.5/13	0.202	Solid, light yellow
6.0/13	0.37	Slightly hard, light yellow
7.5/13	0.557	Hard, lumpy, light yellow

Greases highly depend on the quality of the thickener (saponification). Therefore, the optimal NaOH/PO ratio plays an important role to improve the fat quality. As can be seen in Table 3.2, the optimal NaOH/PO ratio is 3.7 ml NaOH and 13 grams PO for the saponification reaction.

3.3.2. Effect of reaction time on soap consistency

The reaction time is investigated to optimal the soap consistency. Experimental results are shown in Table 3.

Table 3. Reaction time affecting the consistency of the soap

Sample	Time Counter Response (minutes)	Remarks on consistency of soap
1	35	Liquid, white
2	4	Slightly thick, white
3	45	Solid, smooth, smooth, light yellow color
4	50	Solid, porous, light yellow
5	55	Slightly hard, porous, light yellow
6	60	Hard, lumpy, straw yellow

From the above results, it is obvious that the reaction time to create the optimal thickening soap sample is 45 minutes. This value is very important to make a preliminary assessment and predicts the thickener quality. Based on this, we can make adjustments to create the optimal grease product.

3.3.3. Effect of reaction temperature on soap consistency

Since the consistency of the soap sample depends on the temperature of the saponification reaction, the optimum temperature of the soap reaction is further investigated. The survey results are presented in Table 4:

Table 4. Effect of temperature on the quality of a soap sample

Sample	Temperature (^{o}C)	Density of soap
1	75÷80	Slightly liquid and smooth
2	80÷85	Solid, smooth
3	85÷9	Slightly hard, gritty
4	90÷95	Hard, lump

According to the sensory evaluation, the optimal reaction temperature for saponification is 85 ^{o}C ÷ 90 ^{o}C. At this temperature, the soap

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sample has a moderate consistency, smoothness, and suitability for thickening in the process of grease formation. At higher temperatures, the consistency increases, but the soap sample appears to be thick, hard, and it solidifies into a block and has a burning odor.

3.4. Experimental investigation of grease synthesis process

3.4.1. Optimal ratio between the amount of recycled lubricating oil (RLO) and the thickener (NaOH/PO)

Experimental results are presented in Table 5 as follows.

Table 5. Optimal ratio between the amount of RLO and the thickener (NaOH/PO)

Sample	RLO (grams)	Results and comments about fat
1	16	Thick, hard, slightly gritty, compact, bad stickiness, dark yellow
2	18	Solid, viscous, lustrous on the surface, dark yellow begins to turn red
3	2	Slightly loose, straw-yellow, smooth, glossy grease surface
4	22	Liquid, yellow
5	24	Liquid, yellow

As can be seen from Table 5, Sample 2 has the optimal consistency, color, and the glossiness in comparison to the other samples. Therefore, Sample 2 is chosen as the optimum ratio of raw material to produce grease.

3.4.2. Effects of reaction time on the quality of lubricants

The quality of the grease such as consistency, needle penetration, drip temperature, etc. is quite dependent on the reaction time. Therefore, the reaction time is varied from 40 minutes, 45 minutes, 50 minutes, 55 minutes, 60 minutes respectively with the reaction temperature of 85 ÷ 90 ^{o}C in the saponification phase, and 105 ÷ 110 ^{o}C at the homogenizing stage to create fat. The optimal rate of raw materials (NaOH/PO/Ethnic minorities) is 3.7/13/18 while stirring speed is adjusted at 60 ÷ 70 rpm. Then, the experiments are proceeded to determine the dropping point and needle penetration of the above samples.

The experimental results in Figure 2 show that the reaction time to create a grease with a suitable consistency is 55 minutes (saponification time is 45 minutes and grease homogenization time is 10 minutes), and the reaction time is 10 minutes. This application of the resulting grease has a good consistency and color. Especially, it appears to be lustrous on the grease surface.

Thus, a grease sample with a reaction time of 55 minutes has the optimal drip temperature of 118 °C.

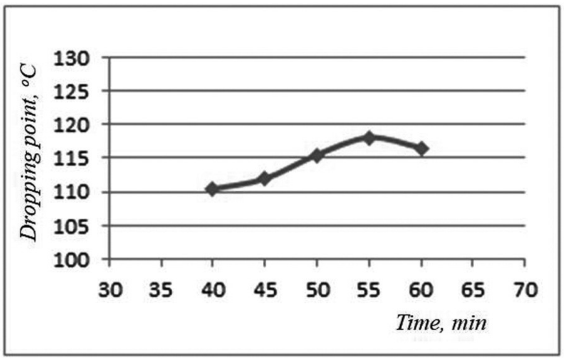


Figure 2. Effect of reaction time on dropping point of grease

3.4.3. Effect of the reaction time on the needle penetration of grease

The experimental results of investigating the effect of reaction time on the needle penetration of grease are shown in Figure 3.

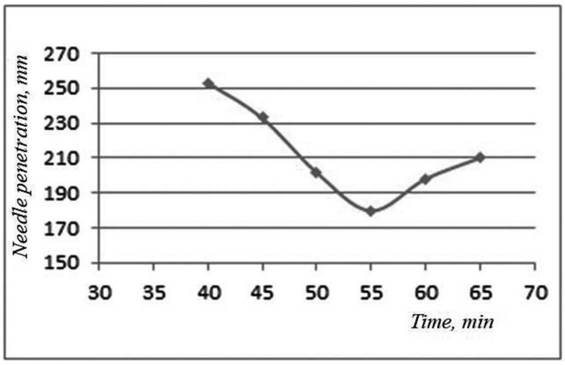


Figure 3. Effect of reaction time on needle penetration of grease

So the grease with a needle penetration of 176 mm is chosen as the grease sample to ensure the smallest needle penetration, which means that the fat consistency is the optimal parameter.

3.4.4. Effect of reaction temperature on the quality of the grease

To investigate the effect of reaction temperature on the quality of the grease, we fix all the investigated factors at the saponification stage, and stir in the grease homogenization stage with a duration of 10 minutes in different temperature ranges. Experimental results are shown in Table 6.

Table 6. Effect of reaction temperature on the quality of the grease

Sample	Temperature stage copper most fat (°C)	Comment on the results	
		Grease consistency	Dropping point (°C)
1	100÷105	Liquid, straw yellow, smooth	112.5
2	105÷110	Solid, yellow bold, smooth, glossy	117.5
3	110÷115	Solid, gritty, from red to shiny yellow	113.5
4	115÷120	turning to liquid	-
5	120÷125	Colloidal solution, lost ability to lubricate the surface, dark red	-

The optimal fat homogenization stage is formed in the range temperature of 105 ÷110 °C. At this uniform temperature, the resulting grease has a smooth, color and luster that ensures the basic properties of the grease. Herein, the fat has a dropping point of 117.5 °C.

3.5. Comparison of experimental results at Chemical Engineering Laboratory, Quy Nhon University and measured results at Quality Standard Technical Center 2 (Da Nang)

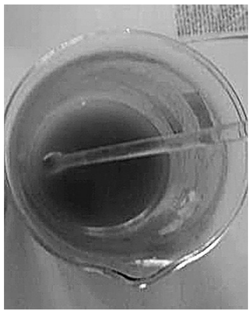
Table 7. Comparison of sample measurement results

Serial	Expense	Results measured at the lab of technology, catalytic materials and petro chemicals	Measurement results at the Center for Standards Technical, Metrology and Quality 2
Dropping point (°C)	ASTM D 566	117.5 ÷118	116
Needle penetration (mm)	ASTM D 217	178	176

Based on the experimental data shown in Table 7, we can see the reliability of measured data, analyzing fat samples at the Chemical Engineering laboratory. With the obtained data: the dropping point is 116 °C and the needle penetration is 176 mm, some machine parts can be used to lubricate some machine parts with a working temperature of less than 116 °C.

Some pictures of synthetic grease samples are showed in Figure 4

(a)



(b)

(c)

(d)

(e)

Figure 4. Photo images of fat samples during synthesis process

a) Solid, dark yellow, smooth, glossy

b) Liquid, yellow

c) Thick, hard, slightly gritty, poor adhesion, dark yellow

d) Thick, hard, slightly gritty, poor adhesion, dark yellow

e) Solid, gritty, yellow falls red

4. CONCLUSIONS

On the basis of the obtained experimental results, we can reach the following conclusions:

- The most optimal ratio between NaOH/PO/RLO to synthesize good quality grease is 3.7/13/18 (ml/g/g).

- The optimal reaction temperature for grease synthesis is 85 ÷ 90 ^oC in the saponification stage and 105 ÷ 110 ^{o} C in the grease homogenization stage.

- The optimal reaction time for grease synthesis is 55 minutes (45 minutes for the saponification phase and 10 minutes for the grease homogenization phase).

- The optimal dropping point of the grease in the experimental process with the raw material ratio, reaction time, and optimal reaction temperature is 116 ^oC.

- The needle penetration of the grease determined in the optimal experimental process is 176 mm.

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