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PREPARATION BIO-LUBRICANT FROM CATFISHFAT

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ABSTRACT

Catfish fat treated to refined Catfish oil (RCfO) by degum, dewax and neutralize. Catfish fat was chemical conversion to ester Catfish oil (ECfO) for the purpose of enhancing the oxidation stability of Catfish fat. Blending bio-lubricant from ECfO and RCfO was constructed base on the 20W-50 engine lubricants formular, replacing mineral base oil as mixture of ECfO and RCfO. The characteristics of material and products were determined by TCVN standard and ASTM standard. Thermogravimetric analysis (TGA) measurements and Rancimat test were used in determination the oxidation resistance. The biodegradability of samples was analyzed by COD test and BOD test. From the analysis results show that, ECfO and RCfO can used as biobase oil. Blend of ECfO and RCfO in ratio (wt/wt) 75/25was not only able to meet the characteristics of 20W-50 engine lubricant but also high biodegradability. The preparation of bio-lubricant from Catfish fat can develop sustainably because of using alternative materials and environmentally friendly products.

Keywords: catfish fat, bio-lubricant, rancimat, thermogravimetric, biodegradability.

1. INTRODUCTION

The crude oil resources are scarced but the demands for petroleum products are increased. In addition, the environment is polluted seriously by the use of petroleum products and amount of the waste lubricants were bring many concerns about environmental pollution. This has prompted scientists to consider for research the bio-alternative products. The properties of vegetable oils and animal fat show that it is possible to produce lubricant from them [1]. From various vegetable oils, such as soybean oil, olive oil, palm oil, castor oil, peanut oil, and cottonseed oil. Properties of blend of soybean oil and olive oil was the best option for biolubricant [2]. In other research, a blend of palm oil and soybean oil nor soybean oil and castor oil was used as biolubricant [3]. Mixing vegetable oils together to form biolubricants is simple and easy to do it. However, the some properties of product may be poor so scientists have studied to improve the quality of biolubricants by the way of adding additives or chemical metabolism of vegetable oil molecules. Biolubricant was blend of castor oil and 5% (wt) improving viscosity additives (4% ethylene-vinyl acetate copolymer and 1% ethyl cellulose). The tribology properties (friction, wear, lubrication) of product were suitable for engine lubricant [4]. Canola oil and corn oil was added 2 %(wt) of Zinc Dialkyldithiophosphate as biolubricant. The coefficient of friction of biolubricant blends was low [5]. From soybean, bio-base oil was synthesized by epoxidation reaction and opening reaction of oxirane ring and the operation at low temperature of product was improved [6]. Karanja oil was converted into Karanja epoxidized oil and alkyl esters of it as biolubricant. Lubrication properties of product were meet the quality standards of lubricants [7]. Trimethylolpropane esters oil was formed from starting material Lunaria annua plant. The physicochemical properties of synthetic Lunaria oil showed that, it was high potential for lubricant base stock [8]. Biolubricant was created by reaction of Jatropha methyl ester andtrimethyl-propanol with sodium methoxy $NaOCH_3$ as catalyst. Pour point and Viscosity Index of Jatropha biolubricant was improved significantly [9]. It is clear that vegetable oils have the advantage of lubricating properties and the addition of additives or chemical transformation of the oil molecular structure has enhanced the quality of the biolubricant significantly [1].

Vietnam is a big exporter of fillet Catfish (pangasius fish) [10]. Fillet portion is only accounted of 1/3 of Catfish weight so the by products of Catfish processing was quite large and fat accounts for the largest share [11]. The time before, the large amount of Catfish fats were discarded or used for animal's food, only Saomai company produces cooking oil from Catfish fat in limited scale. So the Catfish fats is an abundant and available resources in Viet Nam. The preparation of lubricants from refined Catfish oil and synthetic ester of Catfish oil may be a process of sustainable development because of its renewable and environmentally friendly.

2. METHODOLOGY

2.1 Materials

Catfish fat was provided by the Agriculture and Food An Giang (Afiex).Mineral base oil SN500 and some additives were bought from Petrochemical Hoa Viet company, Dongnai, Viet Nam. Anhydride acetic and chemical analysis was obtained from Sigma - Aldrich. Others experimental chemicals originated from China, Vietnam. The equipments are potentionmetric titrator-Titrino PlusMetrohm (Switzerland), mechanical stirrer IKA RW16 basic (Germany) and magnetic stirrer IKA C - MAG HS 10 (Germany).

2.2 Physical treatment of Catfish fat

Catfish fat (CfF) was degumed, dewaxed and neutralized in order to refined Catfish oil (RCfO) [12][13].

2.2.1 Degum

250g of CfF and 5ml water were put into a glass beacher (500 ml), heated on 55°C. Mechanical stirrer was installed. The stirring time and speed of stirrer were carried out in range of (20, 40, 60 minutes) and (200, 400, 600 rpm). After the time finished, the whole mixtures were put into the separating funnel. When the mixtures were saturated completely, the upper liquid oils were received as products and it was determined the acid value for ASTM D664.

2.2.2 Dewax

Product of degum (250g) was put into glass beacher (500 ml) to dewax. Mechanical stirrer was installed and speed of stirrer was slightly. The cooling temperature and cooling time were studied in range of(8, 11, 14 °C) and (4, 7, 10h). After each cooling process finished, the Catfish fat was filtered by filtered cloth (d = 5µm) and product was determined the pour point according to the ASTM D2386.

2.2.3 Neutralize

Product of dewax (250g) and NaOH1N solution (molar ratio of alkaline and fatty acid at 1.5/1) were put into a glass beacher (500 ml), heated on 65°C. Mechanical stirrer was installed. The stirring time and speed of stirrer were carried out in range of (20, 40, 60 minutes) and (200, 400, 600 rpm). After the time finished, the whole mixtures were put into the separating funnel. When the mixtures were saturated completely, the upper liquid oils were received as products and the product was determined the acid value for ASTM D664.

2.3 Chemical conversion of Catfish fat

Ester Catfish oil (ECfO) was synthesized from Catfish fat by epoxidation reaction and opening reaction of oxirane ring. Two reactions were carried out at a fixed conditions, in flat bottom three necked glass reactor. The reaction mixture was stirred at speed of 1000rpm by magnetic stirrer.

Catfish fat Epoxidized reaction

The epoxidation reaction was carried out in molar ratio of hydrogen peroxide : acetic acid : double bond at 3 : 1 : 1, catalyzed sulfuric acid at 2% (wt) of hydrogen peroxide and acetic acid, temperature at 55 °C and time of 5hours.

Firt, hydrogen peroxide was added (very slowly) to the mixture of acetic acidand catalyzed sulfuric acid to created peracetic acid. Reaction mixture was stirred homogeneously, room temperature, 20h.Then peracetic acid was put into Catfish fat to perform the epoxidation reaction. After reactionfinished, the whole mixtures were put into the separating funnel in 4h. The upper liquid oils were received, washed by distilled water, placed in vacuum drying in 10h, dried in anhydrous mangenium sulfate. Final, product was determined the Iod value (TCVN 6122:2010) and epoxy content (ASTM D1652) to evaluate the conversion and performance of reaction.

Oxirane ring opening reaction

The oxirane ring opening reaction was carried out in molar ratio of anhydride acetic: oxirane at 1.5: 1, catalyzed sulfuric acid at 2% (wt) of epoxy, temperature at 80 °C and time of 5hours.

Catfish fat epoxidized oil was added (very slowly) to the mixture of anhydride acetic and catalyzed sulfuric acid to perform oxirane ring opening reaction. After reactionfinished, the whole mixtures were put into the separating funnel, 10h. The upper liquid oils were received, washed by distilled water, dried in a microwave oven. Final, product was determined epoxy content according to ASTM D1652 to evaluate the conversion of reaction.

2.4 Blending bio-lubricant

The bio-lubricant was blended from ECfO and RCfO base on the 20W-50 formular, fixing additives, replacing the SN500 as mixture of ECfO and RCfO, ratio (wt/wt) of ECfO/RCfO in 25/75, 50/50 and 75/25,was coded 25EO, 50EO and 75EO respectively. The properties of bio-lubricant blends and mineral lubricant 20W50 were determinated according to the standard of the engine lubricant. From here, it can evaluate the alternatives of bio lubricants.

2.5 Analysis

Acid number was determined by potentiometric titration method. Process carried out in 848 Titrino Plus-Metrohm, pH electrode in Industrial University of HCMC (IUH).Oxidation stability of samples was determinated by thermo gravimetric analysis (TGA) method and Rancimat test. TGA test was carried out in LINSEIS STA-PT 1600, under nitrogen, temperature in range of (25-800 °C), heating rate at 10 °C/minute in Biomass laboratory of Research Institute for Sustainable Energy, HCMC National University. Rancimat test was carried out in Rancimat 743 according to the EN 14112. Temperature was set up at 110 °C with an airflow of 10 l/h.The testes were carried out in laboratory analysis of petroleum products of Quality Testing Center Standard 3, Viet Nam. Biodegradability of samples was analyzed by biochemical oxygen demand test (BOD) and chemical oxygen demand test (COD). Sample of biolubricant was diluted 100 times and mineral lubricant was diluted 1000 times in distillated water.The properties of materials and products were determined according to the TCVN and ASTM standard in petroleum laboratory of HCMC University of Technology (HCMUT) and Industry University of HCMC (IUH), acid number (ASTM D664), iod value (TCVN 6122:2010), gravity density (ASTM D1298), flast point (ASTM D92), pour point (ASTM D2386), kinematic viscosity(ASTM D445), viscosity index (ASTM D2270).

3. RESULTS AND DISCUSSIONS

3.1. The physical treatment of Catfish fat

3.1.1 Result of degum

Experiments were conducted to study the effect of stirring speed and stirring time on the efficiency of

Catfish fat degum. The results on acid value of samples was show in Table-1.

Tables-1. Effect of stirring speed and stirring time on the yield of degum.

Speed (rpm)	**Time** (minute)	**Acid number** (mgKOH/g)		**Acid number reduction** (%)
		Product	**Material**	
200	20	3.820	3.823	0.07
200	40	3.812	3.823	0.29
200	60	3.808	3.823	0.41
400	20	3.798	3.823	0.66
400	40	3.792	3.823	0.81
400	60	3.787	3.823	0.95
600	20	3.781	3.823	1.10
600	40	3.764	3.823	1.54
600	60	3.753	3.823	1.83

The data in Table-1 showed that, the yields of degum increases as stirring speed and stirring time increases and the rate of decreasing of acid number as increasing of stirring speed is higher than that of stirring time increases. This can explained that, when the mixture is stirred strongly, the contaction of water with hyrophilic compounds occurs fully, so the productivity of degum increases. The optimum condition of degum is time of 60 minutes, speed of stirrer of 600 rpm and acid value of product is3.753 (mgKOH/g).Vegetables oil degumswith water, the acid number decreases about of 0.1 – 0.4 %, in 30 minute, 60 ^{o}C [13]. So the results of degum for Catfish fat of this study were acceptable.

3.1.2 Effect of the time and the cooling temperaturein dewax

Experiments were conducted to study the effect of cooling temperature and cooling time on the efficiency of dewax and results show in Table-2.

Table-2. Effect of the time and the temperature on the yield of dewaxing processing.

Cooling temperature (^{o}C)	**Time (h)**	**Pour point** ((^{o}C))	
		Product	**Material**
8	4	10	21
8	7	9	21
8	10	9	21
11	4	14	21
11	7	12	21
11	10	10	21
14	4	16	21
14	7	15	21
14	10	15	21

The data in Table-2 presented that, the pour point of product decreases in increasing of the time and decreasing of temperature. Dewax perfomed at optimum conditions (8°C, 7hours) and the pour point of product is 9°C while the pour point of material is 21 ^{o}C. So after dewax, the operation of Catfish fat at low temperature improved. When comparing to some bio-base oil such as Coconut oil, rubber seed oil, the pour point of them were in range of (-9 ^{o}C ÷22 ^{o}C)[14], so Catfish fat dewax of this study was effective.

3.1.3 Effect of time and temperature on the neutralize

Experiments were conducted to study the effect of stirring speed and stirring time on the efficiency of neutralize and results show in Table-3.

Table-3. Describes the effect of stirring speed and stirring time on the efficiency of neutralize.

Speed (rpm)	**Time** (minute)	**Acid number** (mgKOH/g)		**Acid number reduction** (%)
		Product	**Material**	
200	20	1.543	3.714	58.46
200	40	1.487	3.714	59.97
200	60	1.447	3.714	61.03
400	20	1.240	3.714	66.62
400	40	1.184	3.714	68.13
400	60	1.105	3.714	70.24
600	20	0.797	3.714	78.55
600	40	0.763	3.714	79.46
600	60	0.791	3.714	78.70

The data in Table-3 showed that, the yields of neutralize increases as speed of stirrer and the time reaction increases. However, the time increases

continuously over level of 40 minutes, the yields of neutralize decreases a few. This can explained that, the contraction of alkaline solution with fatty free acids occurs completely when the mixture is stirred strongly, so the productivity of neutralize reaches at the highest level. But the time increases further, the alkaline solution can react with some neutral components of Catfish fat or the emulsion of them can be created. The formation of emulsion is a problem in separation product. So the yield of the processes was be reduced. So the optimum conditions of neutralize is speed of stirrer of 600 rpm and time of 40 minute, acid value of product is 0.763 (mgKOH/g). When comparison with other researches for preparation bio-base oil, the acid value of soybean oil and almond oil was 1.8 (mgKOH/g) and 4.7 (mgKOH/g) [3] or in other research, the value acid of rubber seed oil and sunflower oil was 13 (mgKOH/g) and 4.0 (mgKOH/g) [14]. The acid value of refined Catfish oil of this study is lower than. After physical treatment, the oxidation resistance of Catfish fat can be improved. So using refined Catfish oil as the biobase oil can be had a great potential.

3.2 The chemical conversion of Catfish fat

3.2.1 Catfish fat epoxidation reaction

Iod value of Catfish fat and oxirane ring product was determinated and the result was71 and 8 (gIod/100g). The oxirane ring product is a colorless liquid. Sample was titrated according to ASTM D1652 to determine epoxy content (E) and E reached at 9.2% (wt), the yields reached at 83.49%.

3.2.2 Catfish fat oxirane ring opening reaction

The ester Catfish oil (ECfO) product was determined epoxy content (E) according to ASTM D1652 and E is1.2% (wt), so the conversion of oxirane ring opening reaction reached at 86.96%.

3.4 Blending bio-lubricant

3.4.1 Determination physicochemical properties of CfF, RCfO, ECfO and SN500

The physicochemical properties of CfF, RCfO, ECfO and SN500were determinated and the results showed in Table-4

Table-4. The properties of CfF, RCfO and ECfO and SN500.

Properties	CfF	RCfO	ECfO	SN500
Acid number (mgKOH/g)	3.82	0.763	0.15	-
Iod value (gIod/100g)	68.8	71	2	-
Oxidative stability				
Decomposition temperature (^oC) - Onset - 5 (%wt) - 10 (%wt) - 50 (%wt) - 90 (%wt) - 95 (%wt)	 161 286.5 298 318.5 338.5 345	 201 294.5 299 319 339.5 347	 255.5 315 343.5 430.5 517 532	 258.5 320 349.5 435 505 521
Induction time (h)	1.2	3.7	25	25
Gravity density	0.906	0.914	0.976	0.888
Flast point (^oC)	293	279	263	250
Pour point (^oC)	21	9	6	-6
Kinematic viscosity (cst) - 40^oC - 100^oC	 37.8 7.07	 29.6 8.27	 225 28.53	 78 11.5
Viscosity index	133.2	177.2	164.8	139.4

The data in Table-3.4 showed that, the physicochemical of RCfO and SECfO were improved much better than that of CfF and almost all of these properties meet the quality requirements of mineral base oil (SN500). Acid number and Iod value of ECfO is decrease much more than that of RCfO and this can predict that, oxidative of ECfO was improved. Compared with other studies on bio-base oil, acid number and iod value of Castor oil fatty acid methyl esters (COFAME) and epoxidized of COFAME was 1.65, 1.08 (mgKOH/g) and 84.6, 1.27 (gIod/100g) (Venu Babu Borugadda, Vaibhav V Goud, 2014) nor crude Jatropha and trimethylolpropane esters of Jatropha oil had a acid number of 4.65 and 0.52 (mgKOH/g)[16].

In both TGA method and Rancimat test, the oxidative stability of ECfO is higher than that of RCfO. It

can explain that, content of the double bonds of ECfO were reduced by experiencing two epoxydation and epoxy ring-opening reactions. Results of Rancimat test for 5 samples linseed oils was in range of (1.43÷1.52 h) [17]. The Rancimat induction times (h) of rapeseed oil, sunflower oil and soybean oils was 11.4, 10.2 and 7.1 (h) [18]. The oxidative stability index (OSI) in Rancimat test of soybean oil was 7.58 (h) [19]. In TGA test, the stable temperature of ECfO is higher than that of RCfO much more, 255.5°C and 201°C. Coconut oil and sunflower oil started to decompose in the range of (220 ÷ 230) and after 300 °C, decomposition of them happened much more [14]. Onset temperature in TGA of castor oil methyl esters (COME) was 203 (°C). Under N_2, decomposition temperature of COME at 10, 50, 90 (%wt) of sample was 200, 243, 310 °C[20]. Under O_2, onset temperature of Castor oil fatty acid methyl esters (COFAME) and epoxidized oil of COFAME was 218 and 305 °C (Venu Babu Borugadda, Vaibhav V Goud, 2014). The oxidation onset and signal maximum temperatures of epoxidized oleic acid (EOA) and products of opening EOA ring reaction was 75 °C, 164 °C and max 207 °C, max 256 °C[21]. ECfO started to decompose in 255.5 °C and after 315 °C, weight loss of it occurrs rapid. So the resistance oxidation of ECfO may be more stable than that of some biobase oil and that can be nearly equal to that of SN500.
The resuilt of TGA and Rancimat test of CfF, RCfO, ECfO and SN500 are described in table 4, Figure-1 and Figure-2.

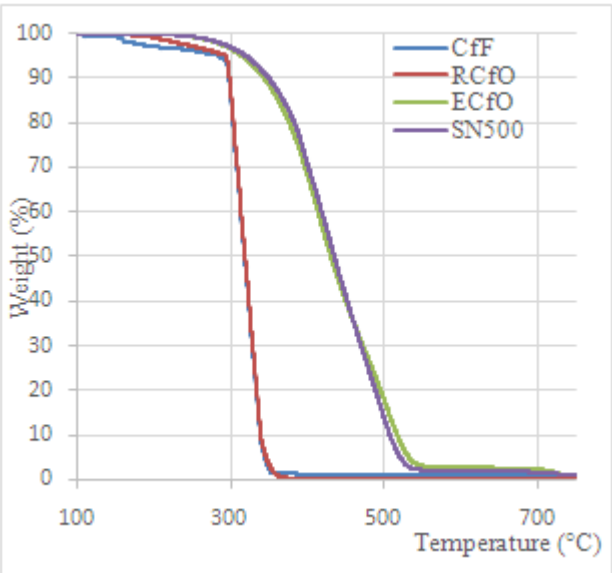

Figure-1. The curves TGA of CfF, RCfO, ECfO and SN500.

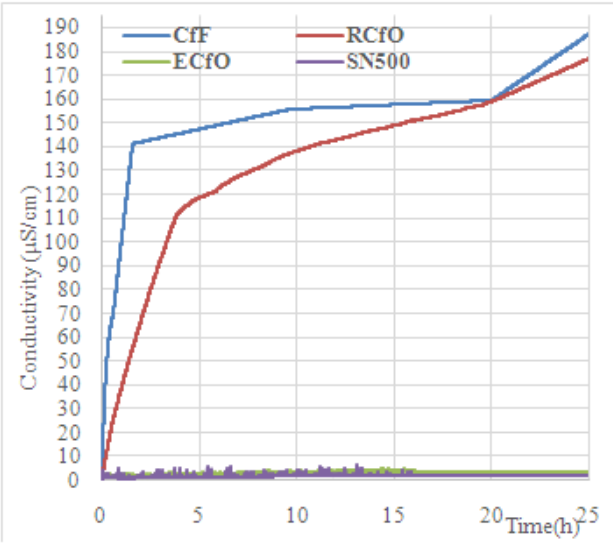

Figure-2. The oxidation stability of CfF, RCfO, ECfO and SN500 in Rancimat test.

The flash point, gravity and lubrication properties of RCfO and ECfOis higher than that of SN 500 and these properties of them can be approximately equal to that of some biobase oils in other researches. This may be explained by high molecular weight of Catfish fat. Sunflower, Coconut oil and rubber seed oil was the flash point of 332, 320, 295 °C [14]. In others, flash point of crude Jatropha and synthetic ester of Jatropha oil was 273 and 296 °C [16]. Synthetic ester of Jatropha oil was viscosity of 51.89 cSt (40 °C), 8.53 cSt (100 °C) and viscosity index of 140 [16]. The kinematic viscosity (at 40 °C, 100 °C) and the viscosity index of of rubber seed oil, coconut oil and sunflower oil was 32.8 cSt, 29 cSt, 27.8 cSt (at 40 °C), 7 cSt, 6 cSt, 6.1 cSt (at100 °C) and 182, 159, 176 respectively [14].With Karanja oil and epoxidized Karanja oil, viscosity (at 40 °C, 100 °C) and viscosity index of them was 40.2, 8.36 cSt, 190.64 and 256.2, 28.0 cSt and 143.99[7] nor the crude Jatropha oil and synthetic Jatropha oil, the viscosity (at 40 °C, 100 °C) and viscosity index was 51.73cSt, 10.75 cSt, 186 and 42.57cSt, 9.37 cSt, 183[9]. So it can say that, storage and use of lubricants, it blend of RCfO and ECfO can be more effective and safer.

The pour point temperature of RCfO and ECfO is improved much more than that of CfF. After dewax, amout of wax was removed and two groups of esters were attached to the double bonds, so the pour point was reduced. However, the methyl ester group did not prevent the crystallization of the product much more. In orthers biobase oil, pour point of crude Jatropha oil was 8 °C[9] nor the epoxidized canola oi (ECO)l and product of ring opening reaction of ECO with n-butanol had pour point of 10 °C and -5 °C or pour point of Karanja oil and Jatropha ethyl ester was 15.8 °C and -1 °C [20]. The pour point of RCfO and ECfO is higher than that of SN500. However, the climate of Vietnam and Asian countries, the average temperature in winter is over 10°C, RCfO and ECfO are

still suitable for bio-base oil, it should add the pour point depressant additives in blending.

3.4.2 Blending bio-lubricant

Blend of 20W50 lubricant was carried out and the result for formular 20W50 lubricant can be is described in Table-5 [22] [23]

Table 5. Composition of formular 20W50 lubricant.

Base oil stock (%wt)	Additives ((%wt)		
	Improving viscosity	Pour point depressant	Multifunction
85.938	6.727	0.916	6.419

Base on the 20W50 lubricant formular, bio-lubricants were blended. The physicochemical properties of 20W-50 engine lubricant and biolubricantblends were determinated and the results showed in Table-6.

Table 6. The physicochemical properties of 20W-50 engine lubricant and biolubricant.

Properties	25EO	50EO	75EO	20W50
Gravity density	0.95	0.955	0.96	0.89
Viscosity (cst) - 40°C - 100°C	 87.97 14.50	 127.57 18.38	 155.82 20.17	 176.29 19.96
Viscosity index	171.82	161.18	150.19	131.04
Flast point (°C)	265	259	255	250
Pour point (°C)	5	0	-6	-14
Oxidation stability				
Decomposition temperature (°C) - Onset - 5 (%wt) - 10 (%wt) - 50 (%wt) - 90 (%wt) - 95 (%wt)	 215.5 271.5 296 373.5 429.5 455.5	 230.5 287.5 314.5 395 439.5 450.5	 238 304 335 430 509.5 522.5	 270 324 352 435 505.5 520
Induction time (h)	10	14.5	16.1	25
BOD(mgO_2/mL)	128	132	115	1,580
COD (mgO_2/mL)	427	439	374	7,000

The data in Table-6 showed that, lubrication properties of three blend of biolubricant meet over the quality requirements of mineral engine lubricant 20W50.In addition, COD value and BOD value of biolubricant is lower than that of 20W50 much more, it can be predicted that, biodegradable ability of biolubricant is higher and it can be explained by the presence of oxygen in the molecular structure of RCfO and ECfO. In orther research for blending biolubricants, blend of 25% soybean oil and 75% synthetic diester has a kinematic viscosity (KV) of 13.88 (mm2/s) at 40 oC and 3.86 (mm2/s) at 100 °C, viscosity index (VI) of it reached at 187.3 [24]. Blend (6/4) of high-oleic sunflower and castor oil, adding 3% ethylene vinyl acetate copolymer (as modifing viscosity additive), KV (cSt) and VI of it was 200.7 (at 40 °C), 27.9 (at 100 °C) and 178[25]. The oxidation resistance of three blend of biolubricants increase in order 25EO, 50EO, 75EO, it is lower than that of 20W50 lubricant. In three blends of biolubricant, the difference for oxidative stability of 75EO blend and 20W50 engine lubricant is the smallest (Table.6, Figure-3 and Figure-4) and this result is consistent with the change in oxidation stability of ECfO, RCfO and SN500 described in Section 3.4.1.

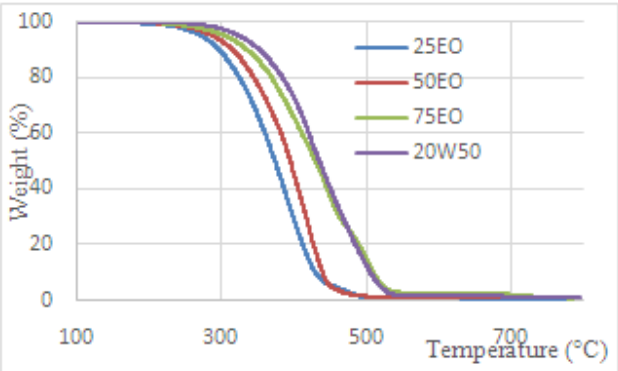

Figure-3. The curves TGA of biolubricants and mineral lubricant (20W50).

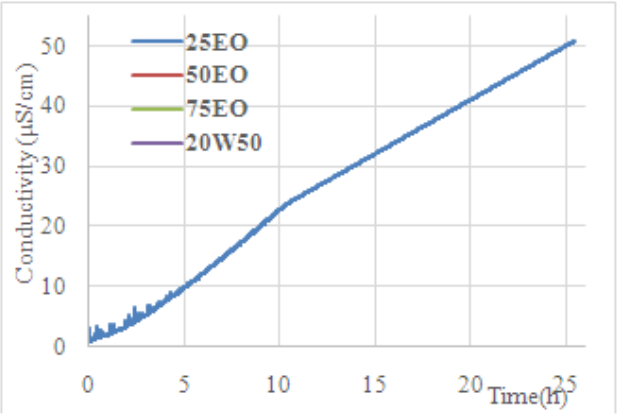

Figure-4. The oxidation stability of biolubricants and mineral lubricant (20W50) in Rancimat test.

Finally, blend of ECfO and RCfO in ratio (wt/wt) of 75/25 can meet nearly the quality standards of 20W50 engine lubricant. So it was chosen as bio-lubricant. Consequently, the storage and use of biolubricant, it blend of ECfO and RCfO will be safer, more efficient and contributing to reducing environmental pollution.

4. CONCLUSIONS

Catfish fat was treated and converted to refined Catfish oil (RCfO) and ester Catfish oil (ECfO). Optimum condition of degum, dewax and neutralize was indicated, time of 60 minutes and speed of stirrer of 600 rpm in degum, cooling time of 7hours and cooling temperature of 8 ^{o}C in dewax and 600 rpm, 40 minute in neutralize. Properties of RCfO was improved, pour point at 9 ^{o}C, acid value at 0.763 (mgKOH/g). The oxidative stability of ECfO was increased. Biolubricant was blended from ECfO and RCfO base on the 20W50 formular and the blend of ECfO and RCfO at ratio (wt/wt) of 75/25 selected as bio-lubricant. Biolubricant of this study not only meets the quality requirements of 20W50 but also is environmentally friendly.

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