THE OPTIMALIZATION OF LUBRICANT WASTE RECYCLING WITH LOW RANK COAL AS CONTAMINANT ABSORBANCE

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ABSTRACT

The research of lubricant waste recycling by means of low rank coal as absorbent is one of the ways to render efficient the oil consumption also to maximize the value of low rank coal. This research is a continuation research with a fixed variable, in which the amount of coal is increased to 20% from the amount of the lubricating oil and the heating time which took 2 hours was variated with the heating temperature variable from 150°C to 350°C, the size of coal granule –8+10, -12+14 and –20+24 mesh. The research output shows that the optimum temperature of the lubricating oil recycling by means of coal as absorbent is reached in the temperature of 300°C for the coal with the size –12+14 mesh. In this condition Ca content reduce from 1447 to 150 ppm, Zn reduce from 887 to 17,4 ppm, Fe reduce from 47,1 to 43,5 ppm, Ni reduce from 15,4 to 6,2 ppm and Cr and Cu are all absorbed. The coal resulted from the recycling process can be used as a direct fuel with the calorific value between 5000 to 6500 cal/g and the colour of the lubricating oil resulted from separation (base oil) is yellowish dark.

1. BACKGROUND

Lubricating oil is one of the most important materials for mechanical device, starting from big engines in industries to vehicle machines. Without lubricant oil machines cannot be operated well. The use of lubricating oil increases due to the rapid technology development, so the quality of the lubricating oil must also be in accordance with the technology development.

The increasing use of industries machines, vehicle engines, etc., implies to the increasing needs of lubricating oil. Thus, it also causes a burden to the environment due to the lubricant waste. The recycling of lubricant waste is one of the alternatives in the efficiencies of oil consumption which decreases year by year, also in can reduce the pollution due to the lubricant oil waste.

Indonesia is the country with an enormous natural resource one of them is coal. Sumatera, Kalimantan and Java are the islands with abundant coal deposits, however, most of the coal are low rank coal.

The recycling of lubricant waste by using low rank coal as absorbent is not merely a way for the efficiency of oil consumption but also as a way to maximize the value of low rank coal. Besides that, it is hoped that by means of this, the recycling of lubricant waste will be more economical and better quality.

By means of recycling lubricant waste, not only it can be a way for the efficiency of natural resources, in particular oil as a raw material for producing lubricating oil, but also as a way to decrease the pollution to the environment causes by lubricant waste which is difficult to overcome.

Aims and goals of this research is to find out the optimum condition and the characteristics of lubricating oil produced from the recycling of lubricant waste by means of low rank coal as absorbent.

It is hoped that from this research we will find out the optimum condition of the recycling of lubricant waste by means of low rank coal as absorbent and also we can get references, particularly in the process of lubricant waste recycling, to be developed to a higher level until it reaches the possibilities to be commercialized.

2. THE RECYCLING OF LUBRICANT WASTE

The increasing usage of factory machines, vehicles engines, etc., implies to the increase usage of lubricating oil, which also causes the increasing of pollution to the environment due to lubricant waste.

The research on the recycling of lubricant waste was carried out to rehabilitate lubricant waste so it can still be used. This is an important matter for large instances who uses lubricant waste in order to save energy.

The degradation of lubricant waste in a high temperature will produce organic compounds such as organic acids, various (ketone) and alcohols. As these organics is polar in the recycling of lubricant waste we have to separate polar organics from non-polar, which are not degredate (Bagasari, 1989)

The recycling of lubricant waste is aimed to remove all of the contaminator from the lubricating oil. Besides various metal which comes from the worn down of machines there are other contaminator such as water, gasoline, diesel fuel, particles, carbon, asphalten and mud (Wartawan, 1985 and Kontawa, 1984.)

Lubricant waste have contaminators such as chloride compounds, phosphorus, metals like Ca and Zn which comes from additive, also metals like Fe, Ni, Cr, Mg, etc., which produced from the burning process. Those contaminators can be reduced or even removed by the process of lubricant waste recycling.

3. RESEARCH METHODOLOGY

The research of lubricant waste recycling was carried out by using one type of low rank coal as the absorbent. The coal used in the research is Samarangau coal from East Kalimantan sent by PT. KIDECO, and the lubricant waste was sent by Lemigas, which was taken from the lubricant storage and reservoir. The lubricant was produced from all kinds of machines. This fixed variable is variated with heating temperature variable and the size of coal particle.

The heating temperature started from 250, 275, 300, 325 to 350^{O}C, the heating takes 2 hours (120 minutes), and the size of coal particle are –8 +10, -12 +14 and –20 +24 mesh. For the coal with the size of –8+10 mesh, the heating variable started from the temperature of 150 to 350^{O}C. In this research we utilize the coal as much as 20% from the amount of the lubricating oil, which will be recycled in each of the research.

Coal ability to absorb the contaminators in the coal was examined through lubricating oil analysis before and after the recycling of lubricating oil.

4. EXPERIMENT PROCEDURE

4.1 Coal Preparation

Dry the coal sample in open air at (room) temperature for ± 2 days. In the first milling use a jaw crusher and in the second milling use a hammer mill. Then, divide the sample into two, one for physic and chemical analysis of the coal and the other for recycling experiment of lubricant waste. The sizes of coal for the experiment are –8 +10, -12 +14 and –20 +24 mesh, and for the analysis is 60 mesh.

4.2 Coal Characteristic

The coal, which is going to be used in the experiment, has to be analyzed to find out its characteristics and rank, includes:

- Calorific value
- Proximate analysis
- Ultimate analysis
- Ash composition analysis

4.3 Lubricating Oil Characteristic

The analysis of lubricant waste includes Ca, Zn, Fe, Ni, Cu and Cr. The sample was oxidized by adding H_2SO_4, then it was heated on a hot plate and a burner. After that is was burned in a furnace on the temperature of 700^{O}C. The sample was then dissolved with HNO_3 and was analyzed by using Atomic Absorption Spectrofotometry (ASTM 5863, 2002).

4.4 The Heating

The heating was done in a 500 ml autoclave, which is equipped with a temperature controller and a pressure indicator. The steps of heating are as follow:

- Weigh the Samarangau coal as much as 40 grams (20% from the lubricating oil)
- Weigh the lubricant waste (200 grams)
- Put the coal into the autoclave tube
- Put in the lubricant waste which are going to be recycled
- Close the tube tightly and then put it into the heater jacket
- Turn on the activator motor and adjust the temperature in accordance to the variable determined
- The heating is done during the operation that is 2 hours (120 minutes)
- During the operation, turn on the motor, and the temperature and pressure increase
- After the operation, turn off the motor, and the temperature controller of the autoclave, then coal it down until it reach the room temperature
- Take out the tube and throw away the gas from the autoclave tube
- Take out the sample from the autoclave tube and put it in the container

4.5 Filtering

- Prepare the lubricant sample from the heating process
- Prepare the filtering equipments by using filter paper No. 42 and then connect the filtering to the vacuum machine
- Filter the sample by putting it into the filtering bit by bit
- The filtering was done twice until the coal is separated from the lubricants waste
- The lubricating oil from the filtering was analyzed as in the first steps

The leftovers coal was then cleaned by using hexane and dried. After it was dried and there were no lubricating oil left, the coal was analyzed as in the first steps.

5. THE OUTPUT AND DISCUSSION

5.1 Raw Material Characteristic

Before using it for the research, Samarangau coal was analyzed first to find out the characteristics and its rank. The analysis of the coal is mentioned in Table 1.

Table 1. Analysis result of Samarangau coal

Analysis (*Air Dried Basis/ADB*)	%
Proximate	
Total Moisture, *as received*	32,11%
Inherent Moisture	22,33%
Ash	2,15%
Volatile matter	38,05%
Fixed carbon	37,47%
Calorific Value	5048 cal/g
Ultimate	
Sulfur	0,10%
Carbon	53,59%
Hydrogen	6,19%
Nitrogen	0,69%
Oxygen	37,28%
Coal rank	Sub-bituminous
Rvaverage	0,45

As seen in Table 1, the Samarangau coal has a relatively high moist water content, that is 22,33%. The high water content causes the coal to be reactive if it is heated and it will form pores, which are able to absorb the substances around it. Ash content seems to be low, as expected, because ash will disturb the coal ability to absorb metals or polluter in the lubricant waste.

Besides proximate and ultimate analysis, the coal ash was also analyzed before it was used as the absorbent in this recycle process. The analysis output is mentioned in Table 2.

Table 2 shows that Fe_2O_3 content dominates the ash content of the Samarangau coal, that is 38,39%, the CaO content are also seems to be high, that is 20,84%.

Table 2. Analysis result of ash coal Samarangau composition

Analysis	(%)
SiO_2	16,9
Al_2O_3	-
Fe_2O_3	38,89
CaO	20,84
MgO	13,89
K_2O	0,14
Na_2O	0,81
TiO_2	1
SO_3	3,28
P_2O_5	3,26
MnO_2	-

Lubricant waste used in this research was first analyzed; the analyzed output is included in Table 3.

To compare the lubricant waste and the new lubricant, we include the analyzed output of the new lubricant in Table 3. Metal content in the lubricant waste seems to increase is high if we compare to the metal content in the new lubricant oil. Thus, to reuse the lubricant waste, the metal content has to be removed by using low rank coal as the absorbent.

Table 3. Analysis result lubricant waste and lubricating oil

Analysis (ppm)	Lubricant waste	New lubricant oil
Ca	1447	138
Zn	877	117
Fe	47,1	-
Ni	15,4	-
Cr	1	-
Cu	6,4	-

5.2 The Recycling of Lubricant Waste

The recycling of lubricant waste is focused on the de-metalization process, that is the process to reduce the metal content in the lubricant waste, by adding coal into the lubricant waste in the operation condition determined. The operation condition applied is the output of former research (Monika, et, al., 2003). The maximum time gained from the former research is 2 hours (120 minutes) and the total sum of coal added is 20% from the amount of lubricating oil used in this research. The increasing variables in this research are the heating temperature and the size of coal (granule).

5.2.1 The Effect of Heating Toward Metal Reduction in the Lubricant Waste (Coal size –8 +10 mesh)

To know the effect of heating temperature towards metal reduction in the lubricant waste, it has been experimented 8 heating temperature variables with the coal size of –8 +10. The research output can be seen in the following table.

Table 4 shows that metal content Ca, Zn, Fe, Ni, Cr and Cu in the lubricant waste are reduce as the heating temperature increases after adding the coal with the size of –8 +10 mesh. In the low temperature (150 – 275^{O}C) the metal content are still fluctuating, especially Ca and Fe is which seems to increase. The increase of Ca and Fe predicted

Table 4. The Effect of Heating Toward Metal Reduction in the Lubricant Waste (Coal size –8+10 mesh)

Coal size	Temperature °C	Ca ppm	Zn ppm	Fe ppm	Ni ppm	Cr ppm	Cu ppm
-8+10 mesh	150	2043	184	74.7	-	0.8	3.3
	175	2358	119	64.8	12.5	0.9	4.8
	200	225	120	12.0	8.3	0.8	1.2
	250	448	288	22.1	-	0.9	-
	275	1417	15.6	34.1	-	0.4	-
	300	84.9	11.8	14.6	0.8	-	-
	325	-	6.4	11.4	0.6	-	-
	350	-	236	-	-	-	-

that in the temperature of 150^{O}C and 175^{O}C the process condition are not stable yet, thus the coal has not absorbed Ca and Fe. The increase Ca and Fe content still has to be investigated and observed thoroughly. However, other metal such as Zn, Ni, Cr and Cu start to reduce in the temperature of 150^{O}C. This proves that coal can absorb metal/contaminant in the lubricant waste as it has pores, which can be opened in the mixing by heating. The increase of heating temperature causes the coal to be reactive and coal pores will be opened, so that it can absorb metals in the lubricant waste. However, the higher the temperature, there is a tendency to damage the structure of the lubricant waste and the coal starts to crack until oil will come out from the coal and mixed with the lubricant waste. The analysis output also can be seen in Figure 1. Absorbent percentage of metal contaminant is included in Table 5.

Tables 4 and 5 shows that in the temperature of 150^{O}C, Zn, Ni, Cr and Cu reduces, for Zn reduce from 877 ppm to 184 ppm (absorb 79,04%), Ni reduces from 15,4 until it can not be detected as the content is low, Cr reduces from 1,0 ppm to 0,8 ppm (absorb 20%) and Cu reduces from 6,4 to 3,3 ppm (absorb 48,43%). The optimum temperature of heating for the coal with the size of –8 +10 as the contaminant absorbent is differ, depends on the metals in the lubricant waste.

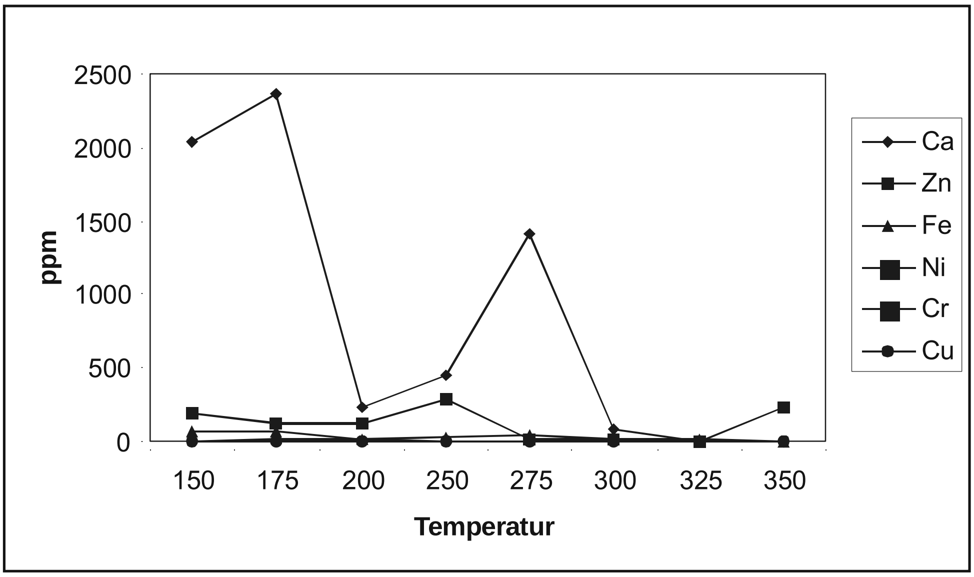

Figure 1. The Effect of Heating Toward Metal Reduction in the Lubricant Waste (Coal size –8+10 mesh)

Table 5. The Effect of Heating Toward Metal Reduction in the Lubricant Waste (% absorb) Coal size –8+10 mesh)

Coal size	Temperature ºC	% Absorb Ca	% Absorb Zn	% Absorb Fe	% Absorb Ni	% Absorb Cr	% Absorb Cu
-8+10 mesh	150	-	79,04	-	-	20	48,43
	175	-	86,44	-	18,83	10	25
	200	84,38	86,32	74,52	46,1	20,1	81,25
	250	69,03	67,2	53,08	-	10	-
	275	2	98,18	27,6	-	60	-
	300	94,16	98,63	69	94,8	-	-
	325	-	99,28	75,8	96,1	-	-
	350	-	73,09	-	-	-	-

5.2.2 The Effect of Heating Toward Reduction of Metals in the Lubricant Waste (Coal size –12 +14 mesh)

In this step we examine the effect of heating temperature of the recycling of lubricant waste towards the absorbent of metals by using coal size –12+14 mesh. The examined temperature started from 250^{O}C until 350^{O}C. The research output can be seen in Tables 6 and Tables 7.

Tables 6 and 7 show that metals that contaminate generally reduces by the increase of heating temperature, but Fe still seems fluctuating. In the temperature of 250^{O}C Ca content reduces to 60,19%, the ability to absorb reduces from 1447 ppm to 575 ppm. The metal that was all absorbed by coal in the temperature of 250^{O}C is Cu (100% absorb). The contaminant, which was absorbed by the coal with the size –12+14 mesh in the temperature 258^{O}C, is Zn 75,26%, Fe 25,26%, Ni 29,87% and Cr 30,00%. Cr and Cu is the ones which were absorbed most by coal. The analyzed output can be seen in Figure 2.

5.2.3 The Effect of Heating Temperature Toward the Reduction of Metal in the Lubricant Waste (Coal size –20 +24 mesh)

In this last step we examine the effect of heating temperature towards metals (absorbility) by using coal with the size –20+24 mesh. The examined temperature starts from 250 to 350^{O}C. The research output could be seen Table 8 and Table 9.

Tables 8 and 9 show that the size of coal –20+24 mesh the absorbing result tend to be not stable depends on metals, which are absorbed. The percentage of (absorbility) of Ca increases in line with the increase of heating temperature. It can be conclude that the smaller the size of coal the better it can absorb Ca. Zn, Ni, Cr and Cu seems to be fluctuating by the increase of heating temperature. For those metals, Zn, Ni, Cr and Cu the smaller the size of coal the more unable to absorb comparing to the coal with size –12+14 mesh. Fe which are absorbed most by coal happens in the temperature of 300^{O}C.

Table 6. The Effect of Heating Toward Reduction of Metals in the Lubricant Waste (ppm) (Coal size –12+14 mesh)

Coal size	Temperature ºC	Ca ppm	Zn ppm	Fe ppm	Ni ppm	Cr ppm	Cu ppm
-12+14 mesh	250	575	217	35.2	10.8	0.7	-
	275	253	44.7	35.4	7.7	0.9	2.0
	300	152	17.4	43.6	6.2	-	-
	325	-	5.2	16.5	4.2	-	-
	350	-	6.7	-	-	-	-

Table 7. The Effect of Heating Toward Reduction of Metals in the Lubricant Waste (Coal size –12+14 mesh) (% Absorb)

Coal size	Temperature ºC	% Absorb Ca	% Absorb Zn	% Absorb Fe	% Absorb Ni	% Absorb Cr	% Absorb Cu
-12+14 mesh	250	60,19	75,26	25,26	29,87	30	-
	275	82,51	94,9	24,84	50	10	68,75
	300	89,49	98,01	7,43	59,74	-	-
	325	-	99,44	64,97	72,73	-	-
	350	-	99,24	-	-	-	-

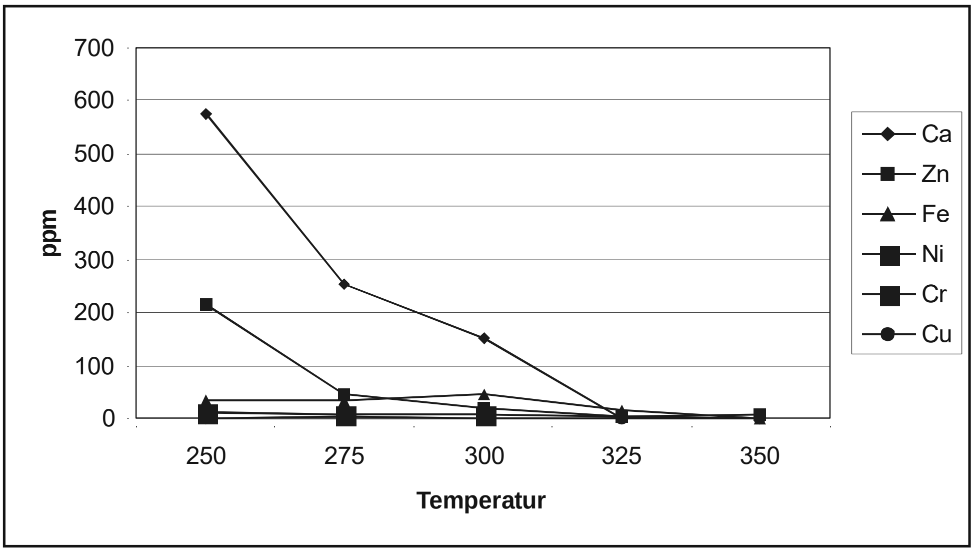

Figure 2. The Effect of Heating Toward Reduction of Metals in the Lubricant Waste (Coal size –12+14 mesh)

Table 8. The Effect of Heating Temperature Toward the Reduction of Metal in the Lubricant Waste (Coal size –20+24 mesh) (ppm)

Coal size	Temperature ºC	Ca ppm	Zn ppm	Fe ppm	Ni ppm	Cr ppm	Cu ppm
-20+24 mesh	250	490	1021	41.8	7.5	-	8.6
	275	254	14.4	19.5	10.5	0.5	4.6
	300	148	236	-	6.9	-	1.2
	325	71.8	996	20.0	-	-	-
	350	41.5	280	7.7	-	3.4	-

Table 9. The Effect of Heating Temperature Toward the Reduction of Metal in the Lubricant Waste (Coal size –20+24 mesh) (% Absorb)

Coal size	Temperature ºC	% Absorb Ca	% Absorb Zn	% Absorb Fe	% Absorb Ni	% Absorb Cr	% Absorb Cu
-20+24 mesh	250	66,14	-	11,25	51,3	-	-
	275	82,45	98,36	58,6	31,82	50	28,12
	300	89,77	73,09	-	55,19	-	81,25
	325	95,02	-	57,54	-	-	-
	350	97,09	60,07	83,65	-	-	-

Ca reduces relatively positive, in which before the recycling was 1447 ppm, reduces to 41,5 ppm (absorb 97,09%) in the temperature of 250 to 350^{O}C. For Zn that can be absorb by the coal with the size –20+24 mesh must be examined more thoroughly because the result was tend to be negative. As can be seen in Table 8, there is an increase of Zn content in the recycled lubricant waste, in which before the recycling was 877 ppm increased after the lubricant was recycled into 1021 ppm in the temperature of 250^{O}C. The same thing happens in the same temperature for Cu, there is an increase from 6,4 ppm to 8,6 ppm. The research output in that condition also can be seen in Figure 3.

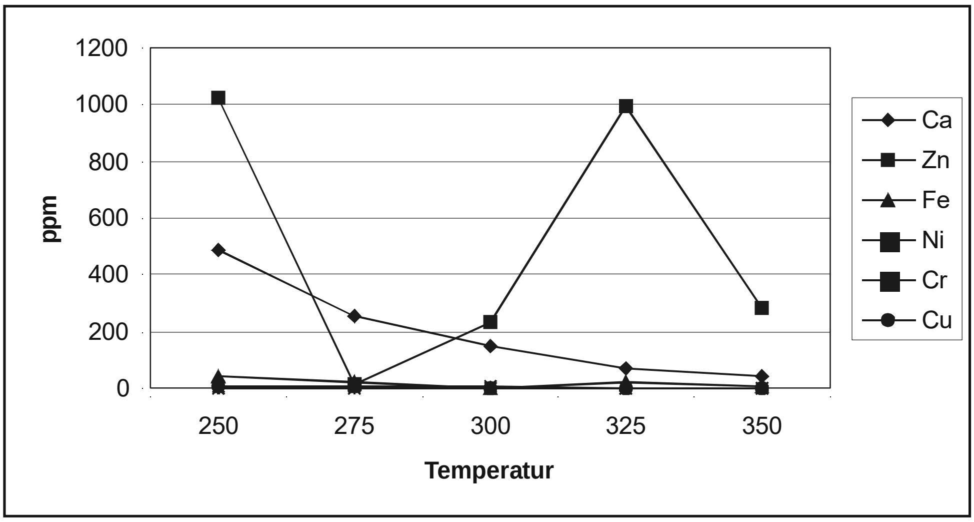

Figure 3. The Effect of Heating Temperature Toward the Reduction of Metal in the Lubricant Waste (Coal size –20+24 mesh) (ppm)

5.2.4 The Comparison of the New Lubricating Oil, Lubricant Waste and the Recycled Lubricating Oil

From the result of the recycling of lubricating oil by means of coal as the absorbent, it can be conclude that the reduction of Ca, Zn, Fe, Ni, Cr and Cu content to be various. Based on the research output included in Picture 4.2, it can be seen that the coal with the size –12 +14 mesh can optimally absorb the metal that contaminate in the temperature of 300^{O}C. However, the expected heating temperature is not higher than 150^{O}C, because in the temperature higher than 150^{O}C will change the structure of lubricant waste.

One of the ways to know whether the recycling process has succeeded is from the comparison of the analysis result of the new lubricating oil, lubricant waste and the recycled ones, as included in Table 10.

Based on the data from Table 10, we can see that Zn in the recycled lubricating oil can be absorbed by the coal with the size –8 +10 in the temperature of 150^{O}C. If we compare with the new lubricating oil, the value of Zn content is nearly the same, it happens likewise for Ni content in the recycled lubricating oil if we compare to the Ni content in the new lubricating oil, in which it is unable to be detected.

Table 10. The Comparison of the New Lubricating Oil, Lubricant Waste and the Recycled Lubricating Oil

Lubricating oil	Ca Ppm	Zn ppm	Fe Ppm	Ni ppm	Cr ppm	Cu ppm
New lubricating oil	138	117	-	-	-	-
Lubricant Waste	1447	877	47,1	15,4	1	6,4
Recycled lubricating oil (-8+10, 150^{O}C)	2043	184	74,7	-	0,8	3,3
Recycled lubricating oil (-12+14, 250^{O}C)	575	217	35,2	10,8	0,7	-
Recycled lubricating oil (-12+14, 300^{O}C)	150	17,4	43,5	6,2	-	-

If we compare the new lubricating oil with the lubricating oil resulted from the recycling process in the temperature of 300°C by means of coal with the size –12+14 mesh as the contaminant, the result is nearly the same except for the Fe and Ni content. However, on the whole, there is an increase in the result of this recycling process comparing to the result of the former experiment, as the mixing and the temperature controller is more controlled.

5.2.5 The Effect of the Recycling Process Towards the Characteristic of Coal

This research also examines the effect of the recycling process of lubricant waste by means of coal as contaminant absorbent towards the characteristic of the coal itself after the process. The result of the research can be seen in Table 11. The analysis output included in Table 11 can be compared with the analysis output of the coal before the recycling process, which included in Table 1.

Tables 1 and 11 show that the content of ash, flying substance, solid carbon, calorific value, and the total of sulfur content increase in the coal after the recycling process, however, water content seems to be reducing. The increase of ash content is because the coal absorbs metals that contaminate in which if it is burnt it may change into ash. The same thing happens to the sulfur content, which increases after the recycling process. The sulfur content comes from the lubricant waste, which can be absorbed by the coal. These two components, ash content and sulfur also moist water content, will affect other content such as calorific value and flying substance. From this analysis output it can be concluded that the coal used to recycle the lubricant waste can be reused as fuel.

In this experiment, the separation of coal from the lubricating oil resulted from the recycling process was done into two filtering steps. However, the recycled lubricating oil is still muddy and it has a yellowish black colour. The filtering result from this research is relatively better as the residue can be separated.

Table 11. The Effect of the Recycling Process Towards the Characteristic of Coal

Coal size Sulfur%	Temperature ºC	Inherent moisture%	Ash %	Volatile matter%	Fixed carbon%	Calorific value cal/g	Total Sulfur%
-8+10	150º	14.65	3.20	43.31	38.84	5489	0.39
	175	12.95	2.94	44.35	39.76	5628	0.46
	200	10.85	3.93	45.46	39.76	5838	0.54
	225	8.57	3.11	46.38	41.94	6158	0.57
	250	5.55	4.20	39.55	50.71	6378	0.75
	275	6.22	3.99	43.63	46.16	6348	0.68
	300	6.68	3.30	47.52	42.50	6431	0.64
	325	7.11	3.81	39.10	49.98	6544	0.84
	350	6.49	6.10	39.68	47.77	6649	1.21
-12+14	250	7.12	3.93	45.46	49.49	6176	0.71
	275	5.92	2.99	42.94	48.15	6311	0.76
	300	7.86	3.90	40.67	47.57	6289	0.79
	325	9.51	3.81	38.64	48.04	6200	0.85
	350	4.58	4.12	35.76	55.54	6776	0.97
-20+24	250	7.49	3.89	43.79	44.83	6111	0.76
	275	5.97	4.52	42.19	47.32	6281	0.83
	300	*	4.61	*	*	*	0.89
	325	15.15	2.60	33.98	48.27	5866	0.88
	350	11.47	5.96	33.64	48.66	6096	0.96

Note:* = Sample is finish

6. CONCLUSION AND SUGGESTION

6.1 Conclusion

From the result of the experiment, we can conclude as follow:

- Observing from the effect of operation temperature towards the absorption of metals that contaminate in the lubricant waste, we can conclude that in the low temperature between 150^{O}C to 175^{O}C metals that contaminate are not optimally absorbed (Ca and Fe) by the coal. However, other metals such as Zn, Ni, Cr and Cu can be absorbed by the coal in the temperature between 150^{O}C to 175^{O}C. The higher the operation temperature, the more metals that contaminate which can be absorbed by the coal.

- Observing from the effect of the size of coal granule towards the absorption of metals that contaminate, we can conclude that the smaller the size of coal granule the more metals that contaminate which can be absorbed. In this research the size of coal granule with the best absorbing ability is –12+14 mesh.

- The process optimum temperature gained in the temperature of 300^{O}C for the coal with the size –12+14 mesh. In this condition Ca content reduces from 1447 to 150 ppm, Zn reduces from 877 to 17,4 ppm, Fe reduces from 47,1 to 43,5 ppm, Ni reduces from 15,4 to 6,2 ppm and Cr and Cu were all absorbed.

- The coal resulted from the recycling process can be used as a direct fuel with the calorific value relatively high, between 5400 to 6700 cal/g, and the moist water content is between 4 to 15%.

- The lubricating oil resulted from the recycling process still have a dark colour, even though it doesn’t have any more coal residue in it.

6.2 Suggestion

- The heating temperature should not be higher than 150^{O}C, so it still needs a further research the variable of temperature lower than 150^{O}C.

- The separation of coal from the lubricant waste must use a good filter, which can produce base oil in accordance with the standard.

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