재자원화기술

Development of Treatment Process for Residual Coal from Biosolubilization

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Abstract

This study introduced a treatment process that was developed to treat Indonesian low-rank coal with high-ash content, which has the same characteristics as residual coal from the biosolubilization process. The treatment process includes separation of ash, solid-liquid separation, pelletizing, and drying. To reduce the ash content, flotation was performed using 4-methyl-2-pentanol (MIBC) as frother, and kerosene, waste oil, and cashew nut shell liquid (CNSL) as collectors. The increasing amount of collector had an effect on combustible coal recovery and ash reduction. After flotation, a filter press, extruder, and an oven drier were used to make a dried coal pellet. Then another coal pellet was made using asphalt as a binder. The compressive strength and friability of the coal pellets were tested and compared.

Keywords : Drying, Extrusion, Flotation, Low-rank coal, Solid-liquid separation, Biosolubilization

1. Introduction

Coal is a promising energy source because of its abundant availability. For the most part, there are two types of coal: high-rank coal and low-rank coal. Low-rank coal has lower heating value, but it is cheaper and more available in large quantities than high-rank coal [1]. Based on 2013 data, the total world coal production was around 7.8 billion tons and accounted for 30.1% of the global primary energy [2]. However, since it is unstable and easily oxidized, the solid form of coal is difficult to handle, transport, and store. Several attempts have been made to convert coal to another form of coal; these previous attempts have included thermochemical gasification and coal-slurry combustion, which are harmful for the environment and consume a large amount of energy [3,4].

Coal solubilization is an energy process that can convert a solid to a liquid fuel that performs in extreme conditions; this is accomplished primarily through chemical processes that consume a large amount of energy. In comparison, the biological treatment of coal, or biosolubilization, can also promote the formation of clean-burning gasses, such as methane, and liquid fuels, such as alcohols. Since it uses a near ambient temperature, near ambient pressure, near neutral pH, and produces a non-hazardous by-product and waste, a biological process consumes less energy and is environmentally friendly. In addition, biological treatment for coal has also been used for desulfurization and demineralization. Biotechnological coal processing was developed under two different processes: aerobic and anaerobic under mesophilic (under 40 ℃) and thermophilic (40 ~ 70 ℃ or higher) conditions [5].

Low-rank coal has become a significant source for bioconversion, which is usually in the form of ultrafine particles. It has a poly-aromatic structure and contains carbon-oxygen bonds, including phenolic, carboxyl, ether, and ester groups, similar to the structure of lignin, which can be degraded by ligninolytic fungi and bacteria by oxidizing phenolic and or non-phenolic aromatic groups within the lignin molecule. These similarities provide an easy method of converting coal into specific products through biological agents that involve the release of radical electrons during the aromatic group's oxidizing process; therefore, the process can spontaneously fragment and depolymerize the macromolecules [6].

Low-rank coal also shows higher susceptibility to biosolubilization than high-rank coal where its susceptibility decreases with the increasing rank of the coal [7]. Low-rank coal contains more of the aliphatic group, more oxygen, fewer condensed ring structures, and more inter-macromolecular linkages than high-rank coal; this influences the ability of enzymes to convert the coal. Two step processes have been performed in biosolubilization: coal solubilization, used primarily in alkaline conditions to produce a water-soluble coal polymer, and then depolymerization to cleave the intermediate products using specific microorganisms or enzymes to produce gaseous or liquid fuel [6].

After biosolubilization, residual coal remains as a by-product; it contains less volatile matter and a fixed carbon content. The residual coal, in the form of slurry, contains water, microorganism cells, and the remaining growth media. Since the biological agents are not able to convert one hundred percent of the original coal, the residual coal still has sufficiently high carbon content and it can be used as a solid fuel for another use. After biosolubilization, the residual coal has less carbon and hydrogen content and more nitrogen, oxygen, and sulfur content, as compared with the original coal [8]. The amount of the aromatic group and the oxygen-containing groups (phenolic or carboxylic groups and ether bonds) both decrease. The remaining growth media, which contain several minerals (K, Mg, Na, Zn, Mn, Co, Ni, Fe, etc.), now exists as an ash. The high nitrogen content also results in high ash content and applies especially in more soluble products, which indicates its metal-chelating ability [8]. Ash and the specific mineral in the ash influence the combustion processes when coal is used as a fuel. It can increase the burning time and decrease the combustion efficiency due to the presence of an ash layer [9]. Therefore, some steps should be performed to reduce the ash content.

Flotation was introduced to reduce the ash content and was followed by solid-liquid separation, extrusion, and drying. These steps were performed to make a coal pellet from the residual coal. The flotation process is used for separating small particles by adding some specific chemical in the mineral/water slurry to make specific mineral surfaces adhere to air bubbles and rise to the surface for removal while others remain in the liquid phase [10].

Solid-liquid separation, such as through filter press, belt press, centrifugation, and precipitation, is performed to get and gather the residual coal into a solid form. A filter press, which uses a filter and retaining cakes as a solid component, applies pressure to filter out the liquid component from the slurry. It can be used to separate suspensions that contain finer particles and/or higher solid content into a batch, semi-continuous, or continuous configuration. A belt press, which is classified as a continuous process, also applies pressure between two tensioned filter cloths to drain the liquid component by mechanical expression to produce a very dry cake. This simple method can discharge the cake naturally and the cloth can be cleaned using a spray [11]. Centrifugation applies centrifugal force to flocculate a high relative weight of solid components in order to separate them from a liquid component. A disc stack type with a nozzle discharge can be used to clarify and thicken the feed to produce solid sludge. It is applicable for slurry with a particle size range of 0.1 - 100 μm and a concentration range of 0.2 - 10 wt%. It can also handle toxic, flammable, and volatile feeds in some batch, semi-continuous, and continuous configurations [11]. Precipitation applies gravity separation to settle the solid components; it occurs naturally for very fine particles. Therefore, it does not require so much energy. In this study, although a filter press was selected and performed to separate the solid and liquid components, it does not rule out the possibility of using another method.

The next step is to shape the coal into a specific form. Roller press and extrusion molding can both be applied using the desired cross-section. A roller press machine squeezes the particles between two counter rotating rollers. When the feed is a fine dried particle, the roller press molding can be used to shape the feed continuously by two rollers. In comparison, extrusion molding uses a screw to shape the coal continuously through a mold in wet or dry conditions [12]. However, if the feed (fine particles of coal) is dried before being shaped, a risk of spontaneous combustion may occur. Therefore, to get the final products, extrusion molding will be applied first and the drying will follow. The aim of this study is to determine the best
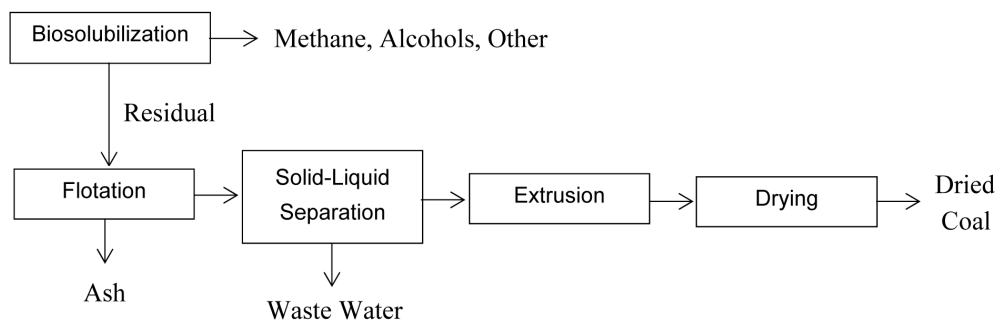

Figure 1. Block diagram of developed treatment process of residual coal.

operating conditions of each unit process that is being used to treat the residual coal from the biosolubilization process, so it can be applied on a commercial scale.

The entire integrated treatment process can be seen in Figure 1. After a laboratory scale of the biosolubilization process was carried out, only a small amount of residual coal remained and it was not enough to use as a feed to perform the process in this investigation. Therefore, low-rank coal was used as a feed in the form of coal-water slurry instead of residual coal. Low-rank coal, which has similar characteristics to those of residual coal, was chosen.

2. Materials and methods

2.1. Samples

KIDECO and Glencore are both coal miners and suppliers located in Indonesia. The coals of both are sub-bituminous, which is defined as a low-rank coal. The proximate and elemental analysis of each sample is shown in Tables 1 and 2, respectively.

Table 1. Proximate analysis result of raw coal

Coal		Moisture (wt%)	Volatile matter (wt%)	Ash (wt%)	Fixed carbon (wt%)
KIDECO	As received	26.12	36.74	5.22	31.92
	Dry basis	-	49.73	7.07	43.21
Glencore	As received	7.62	23.82	31.99	36.57
	Dry basis	-	25.78	34.63	39.59

Table 2. Elemental analysis result of raw coal in dry ash free basis

Coal	Carbon (wt%)	Hydrogen (wt%)	Nitrogen (wt%)	Oxygen (wt%)	Sulfur (wt%)
KIDECO	75.16	4.87	0.85	19.11	0.003
Glencore	75.65	2.87	1.25	18.60	1.63

A thermogravimeter (Leco Co., USA) was used to perform the proximate analysis based on ASTM D7582. A TruSpec Elemental Analyzer (Leco Co., USA) and an SC-432DR Sulfur Analyzer (Leco Co., USA) were used to perform the elemental analysis. The calorific value as-received coal of KIDECO was 19.06 MJ kg^{-1} and Glencore was 12.79 MJ kg^{-1}. It was measured using a Parr 6400 Calorimeter (PARR Co., USA). The size of the coal used in this study was less than 75 μm, which was the same as the coal from the previous biological reaction.

In addition to the coal, some chemical reagents were used in this investigation because the flotation process needs chemicals as frother and collectors. To that end, 4-methyl-2-pentanol (MIBC) was used as a frother to generate bubbles during the flotation process. Kerosene, waste oil, and cashew nut shell liquid (CNSL) were used as collectors. Kerosene was mostly used, but waste oil was used to take an advantage from a waste, and CNSL, which is an extracted vegetable oil, was purchased from Vietnam through the KFI Corp. (around $600/tonne). The heating value of each sample was measured using a Parr 600 Calorimeter (PARR Co., USA); the result for kerosene was 46.89 MJ kg^{-1}, waste oil was 44.30 MJ kg^{-1}, and CNSL was 39.46 MJ kg^{-1}.

2.2. Flotation test

The PJFM003 (Standard Industrial Co., Republic of Korea), as shown in Figure 2, was the experimental device used for flotation. In a main cell or reaction vessel, an impeller is combined with a sparger: 5 L of liquid or solution can be stirred at a speed of up to 1,700 rpm and air can be supplied and dispersed using a sparger, which is located below the agitator. It has a skimmer to collect suspended solids and it moves by rotating. A sample mixture contained 100 g of coal; 1.5 L of water was put into the reaction vessel and stirred at a speed of 1,200 rpm for 10 minutes. Then 0.1 - 15 g kg^{-1} coal of collector was put into the vessel and stirred again for 5 minutes. After that, 0.4 g kg^{-1} coal of frother was added and stirred for 1
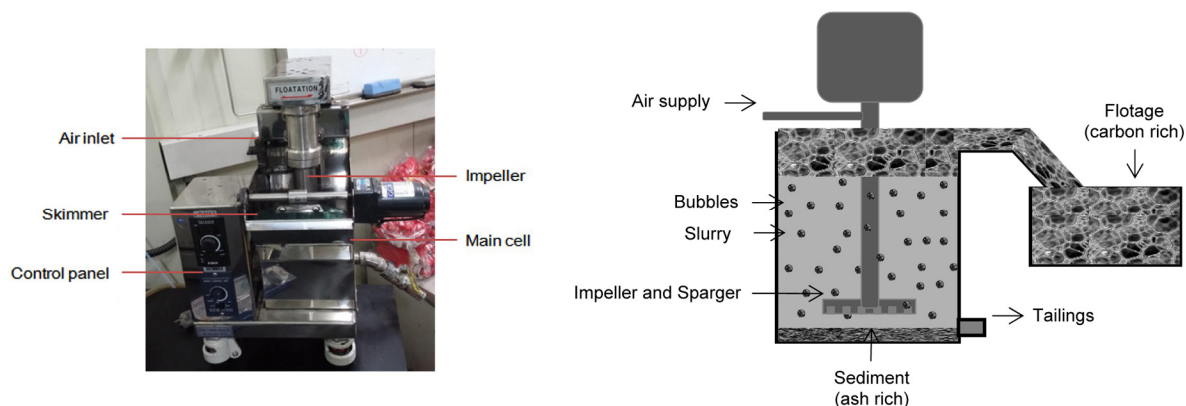

Figure 2. Flotation machine (PJFM003).

minute; the air was supplied by opening the air inlet. The bubbles were generated and attached to the coal at the same time as the air injection and the skimmer collected the froth of the sample as a suspended solid.

After collecting suspension for 5 minutes and reducing the air bubbles, separated precipitate and suspended solid were generated. Then, to determine the flotation yield, flotage as floated fraction and sediment as non-floated fraction were weighed and calculated on a dry basis. Figure 2 shows the principle of the flotation process. The impeller and sparger produced bubbles because of the existence of frother after air injection and the bubbles came into contact with the particles of the slurry. The collector, which has a lower density than water, increased the amount of the coal that attached to the bubbles and hovered over the surface of the water with the froth or foam; the high-density components simultaneously sank to the bottom. This method can generally separate the hydrocarbon and the ash content of the coal.

2.3. Solid-liquid separation

A filter press was used in this stage; the equipment can be seen in Figure 3. The feed was in the form of slurry and it filled into the slurry tank with a bottom valve that had previously been closed. After that, the cover was closed and sealed. Filter cloth was put between the filter plate and the filter chamber to retain the solid content of the slurry. Since this equipment uses pressure to separate solid and liquid content, a manual pump was used to increase the pressure gauge to 200 kPa and then the filter plate and the filter chamber were connected. The slurry tank, or the pressure reservoir tank, had already been connected with the filter equipment and the bottom valve of the slurry was opened by compressed air pressure. The separation was then started and the filter plate formed and discharged the filtrate. The filtrate drop was maintained at 1-2 drops per minute by sustaining the air injection. After finishing, a regulator in the top of the slurry tank was opened to release the pressure and the bottom valve of the slurry tank was closed. The excess pressure was released by opening the cover of the slurry tank. The valve of the manual pump was turned in a counter clockwise direction to release the pressure in the filter plate. Then, the solid cake in the filter chamber can be collected. The principle of this method is illustrated in Figure 3.

2.4. Extrusion and drying

A pellet extruder was used to make the coal pellet (see Figure 4). It has a mold with a diameter of 25 mm. The mold can shape the pellet to a specific length. It also has a feeder and a screw rod as a mixer, which is used to mix the coal inside the extrusion chamber, and it has a length of 600 mm and diameter of 3 mm. A motor geared with 3.75 kW of power and a speed of 20 rpm moves the screw rod.
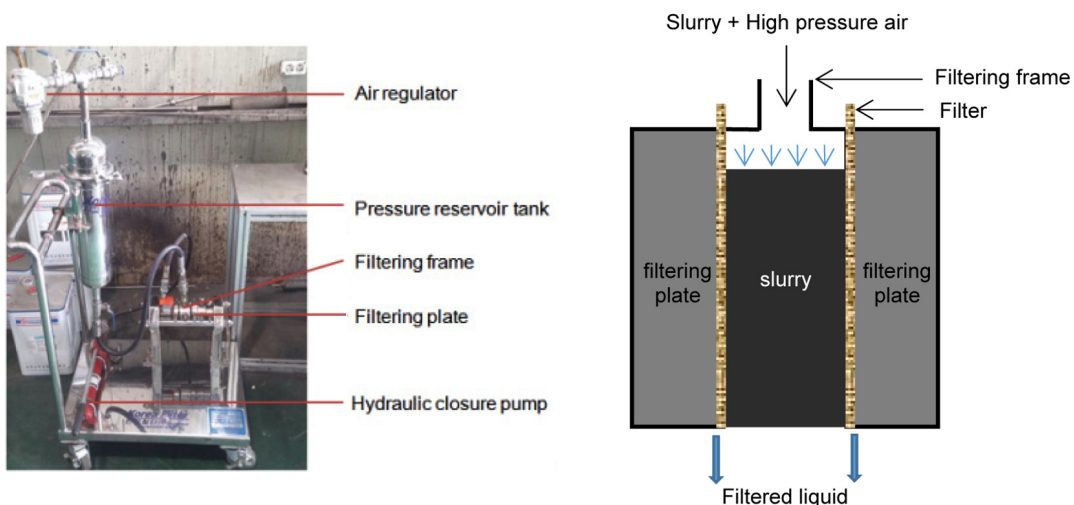

Figure 4. Pellet extruder.

Figure 3. Filter press equipment.

Figure 5. Pellets made by extrusion.

Figure 6. Compressive strength measuring device.

The Glencore coal was pulverized less than 75 μm to produce an extruded sample. To produce the coal pellets, 1 kg of coal was mixed with 500 g of water using a screw mixer at a speed of 10 rpm. Figure 5 shows the pellets being made by the pellet extruder. To evaporate part of the moisture content after extrusion, drying was applied at 107 ℃ for 30 minutes, 60 minutes, and 70 minutes. To compare the drying efficiency of the coal pellet, another experiment was performed using a coal cake from the solid-liquid separation process; this coal cake was dried at 107 ℃ for 60 minutes. An electric oven was used to dry the samples.

After drying, the compressive strength was measured for each of the pellets using a compressive strength measuring device (Figure 6). The force was measured when the compression pump and the hydraulic cylinder broke the pellet. The mean value was determined by measuring 10 of the coal pellets.

The asphalt emulsion was used as a binder to enhance the strength of the pellets (40 wt% asphalt and 60 wt% water mixture are used primarily for road paving). Asphalt emulsion can either be purchased or produced directly from oil. Asphalt emulsion has the advantage because it is an easy to handle liquid and the viscosity is not so high. Pellets that were prepared in the same way as the previous experiment were added to the asphalt emulsion in a mixture of 1 kg of coal, 400 g of water, and 100 g of asphalt emulsion (4 wt% asphalt). Pellets were made and then dried for 60 minutes at 107 ℃ in the electric oven and the moisture content was measured.

In addition to compressive strength, the strength of the pellet was measured by its friability after the binder was added. The breakage rate of the drop test of the pellets was measured to compare their friability. In this method, 10 pellets were dropped onto a hard surface from a height of 2 meters and then sieved using a 4-millimeter sieve filter. This was repeated three times; the fracture ratio is defined as:

$$Friability \ (\%) \ = \ 100 \ \times \ \frac{final \ mass}{initial \ mass} \tag{1}$$

where final mass is the mass of the briquettes retained on the 4-millimeter sieve and the initial mass is the mass of the briquettes at the beginning of the friability testing [13].

3. Results and Discussion

3.1. Flotation test

In the flotation test which the process relies on differences in physicochemical surface properties of particles and hydrophobic character of mineral surfaces, collector and frother have each role [14,15]. Collector is used to selectively adsorb into surfaces of mineral particles, make them hydrophobic, and increase the contact angle so that bubbles will adhere to the surface at the solid-liquid interface. Besides collector, MIBC as a frother is used to stabilize air bubbles and form a stable froth layer that can be removed before the bubbles burst by decreasing the surface tension at the air-liquid interface [10,14,16].

KIDECO coal was used to test the effect of using different collector. Two types of collector had been used, which were kerosene and CNSL. The collector was added as much as 5 g kg^{-1} coal which is 0.5 wt%. Combustible coal recovery and ash reduction were determined after the test as shown in Figure 7. Combustible coal recovery is the percentage of quantity of retained suspended solid in the flotage, while ash reduction is the loss of ash in suspended solid and remained as sediment which
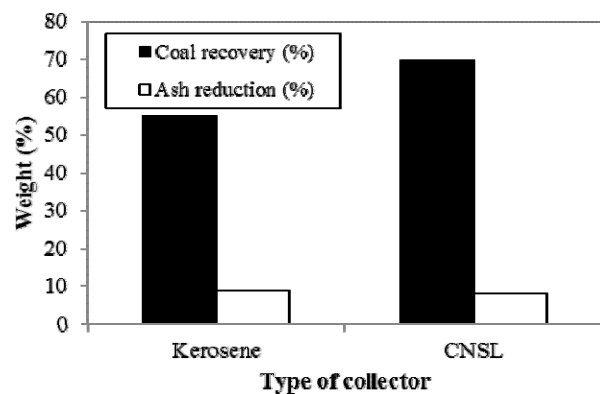

Figure 7. The effect of collector addition to combustible coal recovery and ash reduction of KIDECO coal (collector: 5 g/kg coal).
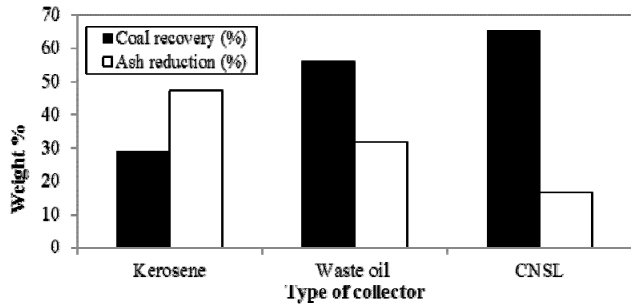
Figure 8. The effect of collector addition to combustible coal recovery and ash reduction of Glencore coal (collector: 15 g/kg coal).

is separated by flotation. Combustible coal recovery shows a higher result when using CNSL than kerosene, while ash reduction shows a higher result when using kerosene than CNSL. Both results show only slightly differences because the ash content of KIDECO coal is not higher enough as shown in Table 1 and resulted in difficulty to compare the effect of collector. Therefore, Glencore coal was used to make comparison easier when determine the effect of using different types of collector in the next experiments.

In the following results, Glencore coal was used to determine the flotation characteristic using three different types of collector, which are kerosene, waste oil, and CNSL by adding 1 wt% of collector which equal to 15 g kg coal in concentration. As shown in Figure 8, kerosene can reduce much ash than the other type of collectors, but with smaller amount of recovered coal. Kerosene is a liquid hydrocarbon that derived from refined petroleum and has low viscosity to disperse and spread over the coal which resulting on more ash reduction. Other than that, waste oil gave a result of the percentage of combustible coal recovery and ash reduction which both between the other types of collector. Figure 8 also shows that using CNSL gave higher combustible coal recovery than other collectors.

All of the collectors that had been used are classified as non-ionic collectors which are non-polar hydrocarbon. They have already partially hydrophobic parts and can enhance the hydrophobicity of coal as well without affecting the ash-forming minerals, while the interaction strength between coal's surface and interfacial water molecules becomes weak and make the flotation to effectively occur. There is different ability of each collector to be filled into the pores of coal and spread onto the surface of coal. The CNSL can easily be filled and spread by coal because CNSL is a hydrocarbon component of plant with high affinity, same as coal. Component with high affinity can easily associated with other molecules, which in this case is coal [10,17-19]

It means that there will be more coal being suspended solid in the flotage. Opposite to combustible coal recovery, the ash reduction using CNSL shows lower reduction and the ash con-

tent of flotage is also higher than the other types of collector as shown in Figure 8 and Figure 9, respectively.

Glencore coal, 0.4 g frother/kg coal, and 0.1 - 15 g collector/kg coal were mixed in this following flotation test. Figure 10 shows the combustible coal recovery and ash reduction results. The increasing amount of collector can increase the collection amount of the flotage, increase the ash content in the flotage, and increase flotage and sediment's ash contents, linearly, because the collector is in the form of co-attached with floated coal as it is apart from non-floated coal as shown in Figure 11.

The collector can be trapped inside the coal resulting higher combustible coal recovery and more coal was lifted out as flotage when using large amount of collector. This result is same as the previous study which both combustible coal and ash content of flotage increase with increasing of collector concentration
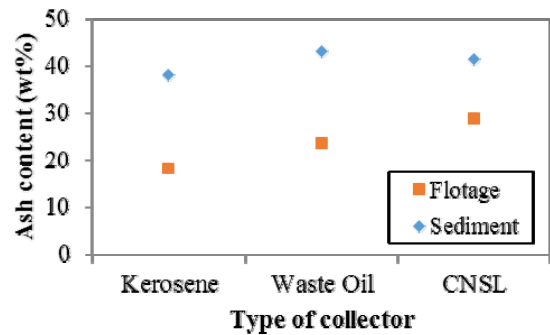

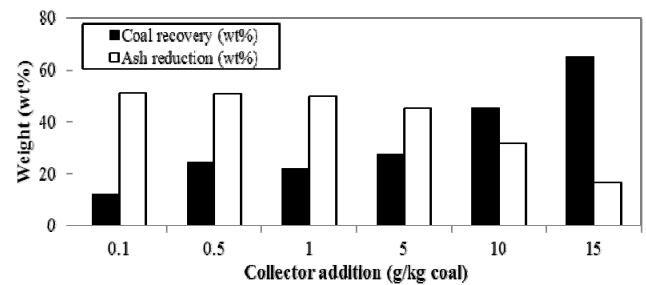**Figure 9.** The effect of collector addition to ash content (15 g/kg coal).

Figure 10. The effect of collector addition to combustible coal recovery and ash recovery of Glencore coal (CNSL).
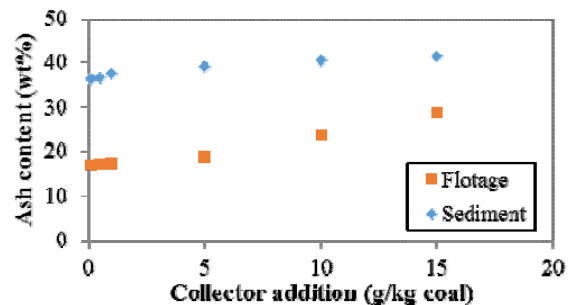

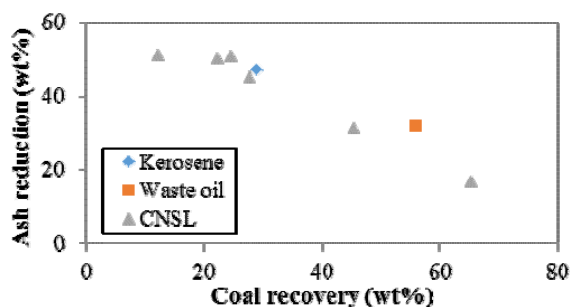**Figure 11.** The effect of collector addition to ash content (CNSL).

Figure 12. Correlation between combustible coal recovery and ash reduction using different collector.

[20]. Meanwhile, the ash reduction became lower because coal was separated without followed by the separation of ash into the sediment. The relationship between combustible coal recovery and ash reduction is shown in Figure 12.

3.2. Solid-liquid separation

After being separated from most of the ash content, moisture content of Glencore combustible coal had to be reduced by filter press. The feed was 2 L of coal slurry resulting 65 - 150 g of solid cake. The moisture content was around 50 wt% and it will be used as extrusion feed. Coal slurry concentration affects the time needed to complete the separation process. The less concentration and volume of coal slurry, the faster separation will be. The particle size can also affect the time required for solid-liquid separation. The very fine particle is expected to reduce time needed to discharge the liquid. The filtered water was clean and can be recycled for flotation process or another process. Besides filter press, centrifugation and precipitation can also be used to drain the liquid phase after flotation.

3.3. Extrusion and drying

The solid cake was used to make coal pellet by extrusion. Coal pellet was dried by 107 ℃ for 30 minutes, 60 minutes, and 70 minutes using electric oven to evaporate partial moisture content. The moisture content after drying were 25 wt%, 12 wt%, and 7.6 wt%, respectively. Besides coal pellet, coal cake from filter press process which was not used as a extrusion feed, was used as a sample for drying process. After coal cake was being dried for 60 minutes, the moisture content was measured to be 26 wt%. The drying rate of coal pellet was faster than coal cake proved by the amount of remaining moisture content after being dried for a certain duration. Besides, part of the moisture of coal pellet was reduced during extrusion due to pressure. In the other hand, coal cake has a high attachment with water and exists as a lump. Therefore, coal cake does not have enough surface area for drying compared with coal pellet. However, in order to make a large surface area, the lump should

Table 3. Compressive strength of pellets

| Sample | Compressive strength (kg$_f$) | | | |
| | Without Binder | | | With Binder |
	Pellet with 25 wt% moisture content	Pellet with 12 wt% moisture content	Pellet with 7.6 wt% moisture content	Pellet with 11.7 wt% moisture content
1	5.5	37.0	34.7	70.2
2	11.5	25.4	15.2	59.7
3	5.8	26.8	21.1	53.5
4	8.7	34.9	38.1	58.8
5	4.2	33.7	20.0	58.8
6	6.5	26.0	52.7	60.6
7	8.9	37.8	30.1	59.9
8	4.8	24.3	30.8	52.8
9	7.9	26.1	26.5	70.1
10	8.2	34.1	24.6	51.3
Average	7.2	30.6	29.4	59.6

be divided into a number of pieces by pelletizing. The drying rate is important parameter to reduce the capacity of dryer.

Dried pellet becomes a great product after a series of treatment. Dried pellets produced by extrusion can prevent the risk of fire and loss of fine powder, and also it does not generate dust directly as dry powder coal. The compressive strength of dried pellet with different moisture content can be seen in Table 3. The compressive strength of dried pellet which has 25 wt% of moisture content is 7.2 kg$_f$ which is lower than the other pellet due to high moisture content. Dried pellet with 12 wt% and 7.6 wt% of moisture content tend to be similar, so the maximum compressive stength can be obtained from coal with moisture content around 10 wt%. The compressive strength of coal is an important thing to determine as it relates to transfer and long-term storage. High value of dried pellet's compressive strength is needed to keep the coal well in the same shape.

Pellets with asphalt as the binder were dried for 60 minutes at 107 ℃ in the electric oven and the moisture content after drying was 11.7 wt%. The compressive strength of the pellets with the binder is given in Table 3. Compared to the coal pellet without binder which has similar moisture content, the com-

Table 4. Friability comparison based on drop test

| Sample | Pellet with binder | | Pellet without binder | |
	Mass (g)	Percentage of friability (%)	Mass (g)	Percentage of friability (%)
Initial	123.0		128.8	
After 1st drop	121.8	99.0	122.3	95.0
After 2nd drop	118.5	96.3	115.5	89.7
After 3rd drop	113	91.9	107.7	83.6

pressive strength is increased about two fold as shown Table 3. Table 4 shows the friability measurement result. The percentage friability of coal using binder is higher than without binder. It means that dried pellet with binder has a high durability. The binder can fill the pores of coal which resulting in high durability of the coal and makes coal hard to be broken because of the great binding of the coal particles. Besides, the binder coats and deactivates the active functional groups on the coal's surface [21,22]

4. Conclusion

In this study, the optimal treatment process for residual coal from biosolubilization was developed through an evaluation on suitability of each process; flotation, filtration, extrusion and drying. Binder test was also carried out for quality improvement of treated coal. Although a test for actual residue coal has not yet been performed, it will not be much different from the current results as it was used for low-rank coal with a high ash content which is similar to the actual residue coal. The treatment process developed in this investigation will contribute to eco-friendly utilization of unused low-rank coal

Acknowledgment

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