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Effect of particle size on cleaning of high-sulphur fine coal by oil agglomeration

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ABSTRACT

An oxidized fine coal sample with high sulphur content was subjected to oil agglomeration process in order to determine the effect of coal particle size (−0.5, −0.25, −0.125 mm) on the performance of the process at different oil dosages (5, 10, 15, 20%). Maximum combustible recovery, ash rejection, pyritic sulphur rejection and sulphate sulphur rejection were achieved to be 75.23%, 97.56%, 99.23% and 98.15%, respectively. Increase in coal particle size affected the ash and pyritic sulphur rejections adversely. However, higher combustible recovery was obtained at coarser coal particle sizes. Scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS) analysis and polished section examination of feed coal, clean coal and tailings support the success in rejection of considerable amount of ash and sulphur sources from the coal. Microscopic examination also showed an increase in agglomerate size with increases in coal particle size and oil dosage.

1. Introduction

Large amounts of fine coals, which are produced during excavation and cleaning activities of coals, are generally discarded. This leads to loss of valuable energy matter and potential environmental problems. Therefore, recovery of fine coals has economic and environmental benefits [1–4]. Froth flotation, flocculation, oil agglomeration [5–7] and enhanced gravity separators [6] are used to clean fine coals. Flotation is the most commonly used cleaning technique in today's coal preparation plants [8,9]. However, it does not produce high quality concentrates [10]. Although froth flotation columns have been introduced to deal with this problem, their ability to treat middling and weakly hydrophobic pyrite particles is not satisfactory [8,11,12]. Flocculation suffers from selectivity problems [4]. Enhanced gravity separators have complex mechanical structure and high operation costs [11], and dewatering is a great problem due to dilute slurry of the product [13]. Oil agglomeration is superior to above mentioned fine coal cleaning methods due to its benefits, such as, high recovery [5,14,15], suitability to process oxidized coals [16,17] and coals with clay slimes [18], simplicity in operation and cheaper dewatering stage [4].

The techniques of oil agglomeration involve preferential wetting of organic matter of coal by oil from coal–water slurry in an agitated condition. The hydrophobic coal particles are agglomerated by oil droplets and separated by screening. Hydrophilic mineral matter is retained in the water phase [3,19].

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Several studies have reported that performance of the agglomeration process is affected by various parameters including, solid content, oil dosage, oil type, agglomeration time, agitation speed, coal particle size and pH of the medium [2,3,5,7,19–23]. Amongst these parameters, solid content, oil dosage, agglomeration time and agitation speed have been widely investigated. However, coal particle size has been comparatively investigated fewer number of times. Unlike the previous studies in which other variables were generally kept constant when investigating coal particle size, this study touches on the effect of coal particle size on the performance of the agglomeration process at different oil dosages. In addition, this study is special in terms of some other aspects. Coal sample used in this study differs from many coals used in agglomeration studies as it shows typical characteristics of an oxidized coal with its brittle nature, poor floatability, high specific gravity, and high sulphate sulphur content. Waste sunflower oil was used as agglomerant. Moreover, determination of pyritic sulphur rejection, sulphate sulphur rejection and microscopic illustration of changing of agglomerate size which were omitted in many of the agglomeration studies were touched in the present study. Photographic examination of agglomerates depending on coal particle size and oil dosage was also carried out.

2. Materials and methods

A coal sample from Muzret (Artvin-Turkey) coal deposit was used in this study. Proximate and sulphur analyses of the sample show that Muzret coal has high ash and pyritic sulphur content (Table 1). Fine coal sample was dry ground to size fractions of −0.5 mm, −0.25 mm and −0.125 mm for oil agglomeration tests. Particle size distributions of the samples were illustrated in Tables 2–4. The waste sunflower oil

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Table 1
Proximate, sulphur and calorific value analyses of the coal sample.

Proximate analysis	Air dried	Dried
Moisture (%)	2.25	–
Ash (%)	34.85	35.65
Volatile matter (%)	10.73	10.98
Fixed carbon (%)	52.17	53.37
Sulphur analysis	**Air dried**	**Dried**
Sulphate sulphur (%)	0.99	1.01
Pyritic sulphur (%)	5.44	5.57
Organic sulphur (%)	1.3	1.33
Total sulphur (%)	7.73	7.91
Calorific value analysis	**Air dried**	**Dried**
Calorific value (kcal/kg)	4970	5084

was used in the experiments. It was used directly without any additional treatment after being filtered to remove pieces of food. The density and viscosity of waste sunflower oil were measured to be 0.918 g/cm^3 and 35.81 mm^2/s, respectively.

Separation sieve size is one of the most important parameters which affect the combustible recovery. Therefore, preliminary study was conducted to determine the separation sieve size. Particle size distribution of agglomerates showed that over 50% of the agglomerates are in the fraction of −0.6 + 0.5 mm [18]. Therefore, it was considered that selection of separation sieve with size over 0.5 mm would cause lost of substantial amount of coal. In addition, particle size distribution of feed coal (Table 2) showed that the amount of coal with particle size just below 0.5 mm was not seemed to be high. It was convinced that it was not possible for potential non-agglomerated particles whose size is close to 0.5 mm to retain on the separation sieve during an efficient agglomerate washing stage. Therefore, separation sieve with an aperture size of −0.5 mm was selected for agglomerate recovery.

Cylindrical glass vessel (11.7 cm in diameter) with four removable baffles of 1.1 cm in width was used for the tests. The agitation of vessel contents was performed using an RZR 2021 type overhead stirrer equipped with a 45°-pitched blade turbine (four blade, 50 mm in diameter). Distilled water was used in the tests. Coal samples with different particle sizes (−0.125, −0.25 and −0.5 mm) were mixed with water (solid ratio: 10%). Coal–water mixtures were conditioned at 1000 rpm for 5 min in order to achieve complete wetting of coal particles. Waste sunflower oil (5–20% of coal) was then added as bridging oil and coal–water–oil mixture was further agitated at 1400 rpm for 10 min. The tests were carried out at the natural pH of the coal–water–oil mixture. After agglomeration, the suspensions were transferred to a recovery sieve with an aperture of 0.5 mm to separate the agglomerates from water and tailings. In order to remove the entrained mineral matter, agglomerates were carefully washed with 1.5 L water. Rejected agglomerates from the sieve were vacuum filtered and de-oiled by washing with acetone. Oil-free agglomerates were dried at 105 ± 5 °C. After drying, agglomerates were weighed and stored for analyses.

Finally, agglomerates were analysed for ash, pyritic sulphur and sulphate sulphur contents by using standard analyses methods. The details of analyses methods can be found elsewhere [18]. The combustible recovery (CR), ash rejection (AR), pyritic sulphur rejection (PSR), sulphate sulphur rejection (SSR), ash separation efficiency (ASE), pyritic sulphur separation efficiency (PSSE) and sulphate sulphur separation efficiency (SSSE) were determined using the following equations: [7,13,21,24]

$$CR(\%) = [(M_P/M_F)x((100-A_P)/(100-A_F))]x100 \quad (1)$$

$$AR(\%) = [1\text{-}((M_P)x(A_P)/(M_F)x(A_F))]x100 \quad (2)$$

$$AR(\%) = [1\text{-}((M_P)x(A_P)/(M_F)x(A_F))]x100 \quad (3)$$

$$SSR(\%) = [1\text{-}((M_P)x(SS_P)/(M_F)x(SS_F))]x100 \quad (4)$$

$$ASE(\%) = CR + AR\text{-}100 \quad (5)$$

$$PSSE(\%) = CR + PSR\text{-}100 \quad (6)$$

$$SSSE(\%) = CR + SSR\text{-}100 \quad (7)$$

where, M_F: mass of dry feed (g), M_P: mass of dry and oil-free product (g), A_F: ash in dry feed (wt.%), A_P: ash in dry and oil-free product (wt.%), PS_F: pyritic sulphur in dry feed (wt.%), PS_P: pyritic sulphur in dry and oil-free product (wt.%); SS_F: sulphate sulphur in dry feed (wt.%), and SS_P: sulphate sulphur in dry and oil-free product (wt.%);

In addition to above mentioned tests, scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS) analysis by SEM-EDS equipment and polished section examination by an ore microscope were carried out to compare the feed coal, clean coal (concentrate) and tailings in terms of ash and sulphur sources. Photos of agglomerates were also taken by using a stereo microscope in order to examine the size of agglomerates.

3. Results and discussion

As seen from Fig. 1, combustible recovery increased with increasing coal particle size at all oil dosages. This can be attributed to the formation of larger agglomerates with increasing size of coal (Fig. 2). Fine size coal

Table 2
Particle size distribution of the coal sample (−0.5 mm).

Particle size (mm)	Weight (%)
−0.5 + 0.3	22.16
−0.3 + 0.212	17.54
−0.212 + 0.15	14.11
−0.15 + 0.106	10.10
−0.106 + 0.053	15.93
−0.053	20.16
Total	100

Table 3
Particle size distribution of the coal sample (−0.25 mm).

Particle size (mm)	Weight (%)
−0.25 + 0.212	12.79
−0.212 + 0.15	18.31
−0.15 + 0.106	14.38
−0.106 + 0.053	21.88
−0.053	32.64
Total	100

Table 4
Particle size distribution of the coal sample (−0.125 mm).

Particle size (mm)	Weight (%)
−0.125 + 0.106	11.24
−0.106 + 0.75	15.84
−0.75 + 0.053	19.82
−0.053	53.10
Total	100