



Role of recovery sieve size in upgrading of fine coal via oil agglomeration technique

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ABSTRACT

Although recovery sieve size has vital importance for the success of the oil agglomeration technique for upgrading of fine coals, its effect on the process has been generally omitted in previous studies. Therefore, the present study was undertaken to fill the gap in this area and draw attention to the importance of recovery sieve size. An oxidized fine coal sample was subjected to oil agglomeration process and effect of separation sieve size on the success of the process with regard to combustible recovery and ash–sulphur rejection was investigated. 72.90% of ash, 90.90% of pyritic sulphur, and 87.75% of sulphate sulphur were removed from the coal with combustible recovery of 76.53%. Increase in recovery sieve size had positive effect on removals of ash and sulphur. Recovery sieve size was found to be extremely important for the performance of the process in that combustible recovery reduced sharply with increasing sieve sizes. It was concluded that recovery sieve size should be the same size with the top feed size.

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1. Introduction

Modern coal excavation methods have caused an increase in the amount of fine coals [1–3]. Fine coals disposed to environment should be benefited in terms of both prevention of environmental pollution and loss of energy sources. However, coal beneficiation methods used for moderate and large coals are not efficient and economic for fine coals [1]. Techniques of flotation, oil agglomeration and flocculation are applied generally for upgrading the fine coal. Among them, oil agglomeration has superiorities over others. It is more suitable for oxidized coal and coals including clay slimes. Higher recovery, cleaner product, easier and cheaper dewatering are its other advantages [1–3].

In oil agglomeration, the difference in hydrophobic character of coal and associated gangue mineral is a separating factor [4]. Hydrophobic coal grains are attached to each other and form agglomerates by bridging force of oil drops. Mineral particles cannot contact with oil drops due to their hydrophilic character. Screens are generally used to separate agglomerated coal grains and mineral matter. Surface properties of coal have vital importance for efficient oil agglomeration [5–7]. In addition to hydrophobic character of coal surface, liberation degree of coal is also an important parameter for the success of the operation [6].

Previous studies undertaken on oil agglomeration of coal can be divided into three groups. First group includes kinetic studies undertaken for determining the kinetics of agglomerate growth [8–11]. Kinetic studies generally revealed that kinetics of the agglomerate can be

defined by second order rate expression and evaluation of d_{50} is important in determination of size distribution of agglomerates. Second group includes optimization and/or experimental design studies undertaken for determination of most important parameter and optimum values of process parameters to obtain maximum performance and comparison of influence of each parameter [12–17]. Third group includes studies undertaken for individual effects of one or more parameters or various pretreatments on oil agglomeration of coal [2,5,6,18–34]. Although various parameters such as solid ratio, oil dosage, oil type, agglomeration time, stirring speed, coal type, coal particle size, pH of the media, washing water amount, and salt content have been investigated in above mentioned three groups of studies, oil dosage, solid ratio, agglomeration time and stirring speed had been the most widely investigated parameters. It can be generally stated that an increase in these parameters had positive effect on agglomeration up to a special value of the parameter after which a negative effect was observed.

Although recovery sieve size has a vital importance for the performance of the oil agglomeration process, very limited knowledge in studies [25,35] has been reported before relating to its effect on combustible recovery and ash–sulphur rejections. Therefore, a detailed work is required to investigate the relationship between recovery sieve size and agglomeration performance in terms of combustible recovery and ash–sulphur rejections. Coal sample used in the present study is special due to its oxidized structure. It has high brittleness, sulphate sulphur content and specific gravity. Waste sunflower oil was used as an agglomerant. Moreover, pyritic and sulphate sulphur rejection of the process was investigated unlike the most of the previous coal agglomeration studies which have been contented with ash rejections.

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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

2. Materials and methods

Coal sample was taken from Muzret coal deposit in Turkey. A fine coal sample of ≈ 150 kg was taken from coal deposit. Large portion of the sample was below 0.5 mm. Remaining little amount of sample whose size was over 0.5 mm was reduced to -0.5 mm by controlled dry grinding. The sample was divided by repeated coning and quartering until one fourth of initial sample (≈ 37.5 kg) was obtained. Then, chute riffles having different chute opening sizes were used as repeated cycles for further dividing and mixings until the feed amounts (≈ 46 g) was obtained. Final sample was completed to ≈ 50 g to be used in tests by adding small samples taken from squares of chessboard placed samples.

From the proximate analysis of the sample, it can be seen that it contains a significant amount of ash and pyritic sulphur (Table 1). XRD analysis of the sample revealed that clay minerals (kaolinite, illite, montmorillonite), calcite, gypsum, and quartz are present in the coal as mineral matters. Particle size of coal sample was reduced to size fractions of -0.5 mm, -0.25 mm and -0.125 mm for experiments. Particle size distributions of the samples were illustrated in Tables 2–4. As bridging material, waste sunflower oil was used after a filtration process applied for eliminating the remaining food particles. The density of the waste sunflower oil was determined to be 0.918 g/cm³ with an Alla France type hydrometer and kinematic viscosity was determined to be 35.81 mm²/s by using a Tanaka AKV-202 type viscometer. Zeta potential measurement was carried out for coal sample and waste sunflower oil by using Zetasizer Nano-ZS90. Cu, Zn, Ti, P, Sr, V, Cr, Mn, As, and Rb were determined to be in a coal sample by using an EDXRF spectrometer.

Agglomeration experiments were undertaken in a cylindrical glass vessel whose diameter was 11.7 cm. Four portable baffles were inserted to the vessel. The stirring process was achieved by means of a RZR 2021 type overhead stirrer. Water was distilled before the experiments to remove ions of calcium, magnesium, chlorine, sulphate, etc. from water. These ions in undistilled water affect the hydrophobicity and agglomeration ability of coal since they change pulp potential and also pH of the medium. Because each laboratory has top water with different structures, water used in test was distilled for providing test results to be comparable with other previous or further agglomeration studies undertaken in different laboratories. However, agglomeration test

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

using top water or spring water to be used in agglomeration plant should be undertaken before installation of agglomeration plant.

Coal samples with three different sizes (-0.125 , -0.25 and -0.5 mm) were mixed with water (solid ratio: 10%). Coal–water mixtures were stirred at 1000 rpm for 5 min to provide perfect wetting coal grains. The oil (10 wt.% of coal) was then put as an agglomerant and the mixture of coal–oil–water was stirred at 1400 rpm for 10 min. The experiments were performed at an ambient pH of the mixture (5.36). Recovery sieve was used to separate agglomerates from water and mineral matters. Size of the recovery sieve changed in the range of 0.125–0.5 mm, 0.25–0.6 mm, and 0.5–1 mm for feed size of -0.125 , -0.25 and -0.5 mm, respectively. Agglomerates were washed with 1.5 L water to remove the entrained mineral matter. Then, vacuum filtering and acetone washing for de-oiling were applied for agglomerates. After drying of oil-free agglomerates at 105 ± 5 °C, weighing was carried out and cleaned coal products were stored for analyses. Photos of different stages of agglomeration tests and agglomerates produced from feed coals of different particle sizes were illustrated in Figs. 1 and 2, respectively.

Finally, analyses for ash, pyritic sulphur and sulphate sulphur were undertaken. Parr and Powell method [36,37] was used for pyritic and sulphate sulphur analysis. Sulphate sulphur is determined by extraction of sulphur by dilute hydrochloric acid. Water and dilute hydrochloric acid are added to pulverized (-250 μ m) coal sample in a beaker. The mixture was boiled gently following by filtering and washing. Filtrate is diluted with water after addition of hydrochloric acid. Solution is boiled slowly and barium chloride is added. Sulphur is precipitated as BaSO₄ and the amount of sulphur was calculated after igniting the crucible including BaSO₄ plus filter paper. For the pyritic sulphur determination, extracted coal residue from sulphate sulphur analysis is digested with dilute nitric acid and the solution is allowed to stand at room temperature for 4 days. At the end of the period coal residue is filtered off and filtrate is evaporated. The filtrate residue is moistened with a few drops of hydrochloric acid and again evaporated to dryness. The residue is dissolved in hydrochloric acid and diluted with water. Solution is boiled slowly and barium chloride is added. Sulphur is precipitated as BaSO₄ and amount of sulphur was calculated after igniting the crucible including BaSO₄ plus filter paper. ASTM-D 3174-89 [37, 38] was used for ash analysis. For the ash determination pulverized coal sample (-250 μ m) was put in crucible and heated in a furnace at 750 °C. After heating, crucible is removed from the furnace and cooled. Ash percent is calculated. Appearances from pyritic sulphur and ash analyses can be seen in Fig. 3.

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

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

Particle size (mm)	Weight (%)
$-0.125 + 0.106$	11.24
$-0.106 + 0.075$	15.84
$-0.075 + 0.053$	19.82
-0.053	53.10
Total	100

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