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Increasing coal quality by oil agglomeration after ultrasonic treatment

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ABSTRACT

In the present study, oxidized and high sulphur fine coal was subjected to oil agglomeration process after ultrasonic treatment. Power and time of ultrasonic treatment were selected as variable. Combustible recovery, ash rejection, pyritic sulphur rejection, ash separation efficiency and pyritic sulphur separation efficiency of the agglomeration process with and without ultrasonic treatment were determined. In addition, calorific value of the clean coal produced by agglomeration process was measured. In the agglomeration process without ultrasonic treatment, calorific value, combustible recovery, ash rejection and pyritic sulphur rejection were obtained to be 6518 kcal/kg, 63.78%, 75.19% and 92.64%, respectively. Ultrasonic treatment enhanced the performance of oil agglomeration process. By the application of ultrasonic treatment before agglomeration process, calorific value, combustible recovery, ash rejection and pyritic sulphur rejection were increased to maximally 6939 kcal/kg, 66.13%, 87.24% and 97.44%, respectively. In the study, changes on the surface structure of the coal after ultrasonic treatment were also examined. Particle breakage, formation of cracks and cavities, and altering of surfaces into fresh-clean surfaces were observed. Increase in time and power of ultrasonic treatment had slight positive effect on ash and pyritic sulphur rejections.

1. Introduction

Ultrasonic treatment causes cavitation, that is, the formation, growth and collapsing of bubbles in a liquid. Collapsing of bubbles produces small areas of high pressure differences, resulting in micro turbulence [1–3]. When the ultrasound is applied to a mixture of particles—liquid and the bubbles collapse near the solid surface, a high speed jet of fluid is driven into the particles and this jet can deposit enormous energy densities at the site of impact [4]. One substantial benefit of ultrasonic treatment in mineral processing can be the removal of surface coatings and this is mainly achieved by cavitation. The simplest effect of energy released by cavitation is to help cleaning of surface of minerals suspended in the slurry [5]. A more complex effect may be to alter the surface chemistry of the mineral, thereby mediating its response to other substances in solution or to physical separation regimes [5,6].

As with flotation, oil agglomeration of coal relies on the differences in the surface properties of hydrophobic coal particles and hydrophilic inorganic gangue [7]. When a small amount of oil is introduced into an agitated suspension of coal particles, hydrophobic coal particles become oil coated and stick together to form agglomerates while the hydrophilic mineral water particles remain unaffected. The agglomerated particles can be separated from the other materials by a single screening operation or alternatively by floating and skimming. Performance of selective oil agglomeration depends on a great extend of the surface properties of solid particles [8,9]. Hydrophobicity of coal surfaces, hydrophilicity of mineral matter surface and liberation of mineral matter from coal are among the parameters required for efficient removal of mineral matter in coal by this technique [9].

Recently, several studies have been carried out to determine the usability of ultrasonic pre-treatment in coal processing. Majority of these studies are focused on increasing the flotation ability of coal by using ultrasonic pre-treatment [4,10–16]. The remaining studies included ultrasonic pre-treatment to enhance chemical desulphurization [17–20] and heavy medium separation [21]. However, no investigation on ultrasonic treatment of coal prior to oil agglomeration process in order to enhance the performance has been reported before. This study is the first attempt to treat coal with ultrasound before oil agglomeration. Coal sample used in this study differs from many coals used in agglomeration studies in that it shows typical characteristics of an oxidized coal with its brittle nature, poor floatability, high specific gravity, and high sulphate sulphur content. In addition, pyritic sulphur rejection that was omitted in many of the agglomeration studies was determined in the study. Moreover, bridging oil used in the study was low-cost waste vegetable oil contrary to other oil types used in most of the agglomeration studies. Furthermore, limited number of studies on surface examination of ultrasonically treated coal has been undertaken before. In the study, the effect of the ultrasonic treatment of coal on performance of oil agglomeration in terms of the combustible recovery and ash–sulphur

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removals was investigated and surface structure of the coal after ultrasonic treatment was examined.

2. Materials and methods

A coal sample from coal deposit located in Muzret (Artvin, Turkey) was used in this study. Proximate and sulphur analysis, and X-ray diffraction (XRD) analysis were performed. Proximate and sulphur analysis of the sample showed that Muzret coal had high ash and pyritic sulphur contents (Table 1). From XRD analysis, pyrite, kaolinite, montmorillonite, illite, calcite, gypsum, and quartz were determined to be the mineralogical components of the sample. Coal sample was dry ground to 0.5 mm for oil agglomeration tests. The waste sunflower oil which had been employed several times for frying chicken and meat was used in the experiments. It was used directly without any additional treatment after being filtered to remove pieces of food. The density of the waste sunflower oil was determined to be 0.918 g/cm3 with Alla France type hydrometer and kinematic viscosity was determined to be 35.81 mm^2/s by using Tanaka AKV-202 type viscometer.

At first, agglomeration test without ultrasonic treatment was undertaken. Cylindrical glass vessel (11.7 cm in diameter) with four removable baffles of 1.1 cm in width was used for the test. The agitation of vessel contents was performed using RZR 2021 type overhead stirrer equipped with a 45°-pitched blade turbine (four blade, 50 mm in diameter). Distilled water was used in the test. Initially, coal–water mixture (solid ratio: 10%) was conditioned at 1000 rpm for 5 min in order to achieve complete wetting of coal particles. Waste vegetable oil (10% of coal) was then added as bridging oil and coal–water–oil mixture was further agitated at 1400 rpm for 10 min. The test was carried out at the natural pH of the coal–water–oil mixture. After agglomeration, the suspension was transferred to a sieve with aperture of 0.5 mm to separate the agglomerates from water and tailings. In order to remove entrained mineral matter, agglomerates were carefully washed with water of 1.5 L. The agglomerates removed 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.

Secondly, coal–water mixtures (solid ratio: 10%) were subjected to ultrasonic treatment in order to determine the effect of ultrasonic treatment on surface properties of coal and performance of the agglomeration process. A laboratory type, high intensity ultrasonic generator (750 W, 20 kHz) equipped with a horn transducer system and a titanium alloy horn tip (13 mm in diameter) was used as a source of ultrasonic treatment. Power (9.5–72.8 W/cm^2) and time (0.5–7 min.) of the ultrasonic treatment were selected as variables. Some of the ultrasonically treated coal–water mixtures were used for scanning electron microscopy–energy dispersive spectroscopy (SEM–EDS) examination of coal particles. Other coal–water mixtures treated with ultrasonic generator system were subjected to agglomeration process at the same conditions and manner of agglomeration test without ultrasonic treatment. Polished section examinations of the feed, concentrate and tailings of the agglomeration process under microscope were performed to prove the success of the process on coal cleaning.

Table 1
Proximate and sulphur analyses of coal sample.

Components	Air dried	Dried
Moisture (%)	2.25	–
Ash (%)	34.85	35.65
Volatile matter (%)	10.73	10.98
Fixed carbon (%)	52.17	53.37
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 (kcal/kg)	4970	5084

Finally, agglomerates were analysed for ash and pyritic sulphur contents by using standard methods of analyses. The details of the methods of analyses can be found elsewhere [22]. The combustible recovery (CR), ash rejection (AR), pyritic sulphur rejection (PSR), ash separation efficiency (ASE) and pyritic sulphur separation efficiency (PSSE) were determined using the following equations: [23–26]

$$CR\,(\%) = (M_P \times CV_{P)}/(M_F \times CV_F) \quad (1)$$

$$AR\,(\%) = [1-((M_P) \times (A_P)/(M_F) \times (A_F))] \times 100 \quad (2)$$

$$PSR\,(\%) = [1-((M_P) \times (PS_P)/(M_F) \times (PS_F))] \times 100 \quad (3)$$

$$ASE\ \ (\%) = CR + AR - 100 \quad (4)$$

$$PSSE\,(\%) = CR + PSR - 100 \quad (5)$$

where, M_P: mass of dry, ash and oil free product (g) M_F: mass of dry, ash and oil free feed (g), CV_p: calorific value of dry and oil free product (kcal/kg), CV_F: calorific value of dry feed (kcal/kg), 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.%), and PS_P: pyritic sulphur in dry and oil free product (wt.%).

3. Results and discussion

Fig. 1 shows the comparison of the particle size distribution of coal before and after ultrasonic treatment. As seen in Fig. 1, particle size of the coal is reduced after ultrasonic treatment. Reduction of particle size shows that ultrasonic treatment produced particle breakage. Consistent with this finding, similar effects of ultrasounds on coal structure were reported by different authors [11,27]. SEM examination (Fig. 2) shows the surface structure of coal before and after ultrasonic treatment. As seen in Fig. 2, crack formations together with cavities and fresh surfaces on coal particles were created by ultrasounds. Breakage, crack and cavity formation can be attributed to the mechanical impact produced by powerful jets of collapsing cavitational bubbles. Ambedkar et al. [27] reported crack formation and particle size reduction, and proposed four stages for ultrasound-assisted coal breakage. These were pitting of the coal surface, formation of cracks on the coal surface, widening–deepening of coal surface cracks and breakage of coal particles.

As seen in SEM–EDS analysis (Fig. 3) of a selected dot on the coal surface, oxidized layer and mineral coatings on the coal surface were cleaned by ultrasonic treatment, resulting in increase in carbon content

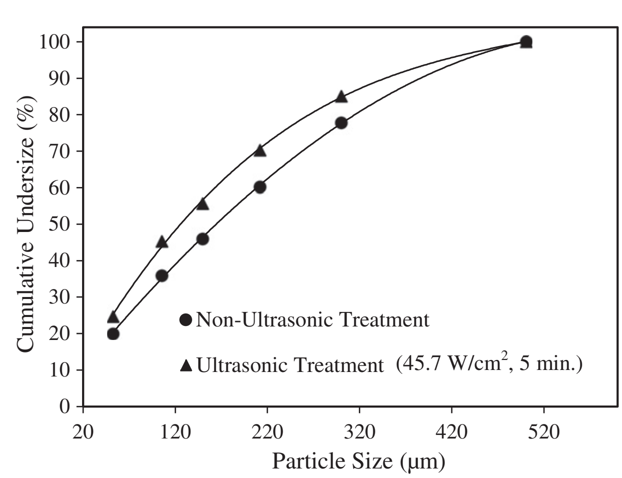

Fig. 1. Particle size distribution of coal before and after ultrasonic treatment.