

FLOTATION OF HEAT TREATED LOW RANK COALS AFTER DRY GRINDING WITH TAR ADDITION

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ABSTRACT

It is well known that low rank lignite coals have very poor floatability characteristics. However, heat pre-treatment of coal following dry grinding adding with some type of organic reagents causes much better flotation ability of coals. In this study, improvement of flotation behavior with treated lignite coal samples were investigated. During experimental work, special design heater was used in the temperature range between 300 °C and 600 °C, as well as heating time between 15 and 90 minutes. As a result of experimental works, it was observed that floatability of coals can be increased increasing with both process temperature and time, and also, tar additives create positive effect for the flotation results of treated coals because of the collector adsorption on the particle surfaces can be successfully achieved. Due to the overall investigation results subjected to this study; 89.9 percent clean coal by weight containing 10.1 % ash was obtained from flotation process. Ash content of flotation tailings also increase up to 45-50 %.

INTRODUCTION

There are abundant low-rank coal reserves in the world. Similarly, Turkey has also huge amount reserves of low quality lignite coals which are characterized by high ash, moisture, volatile matter and sulfur content. According to the decreasing high quality coal reserves as well as environmental restrictions studies for evaluate low-rank lignite coals as an energy resources are increasing. Furthermore the control pollutants such as sulfur dioxide, nitrogen oxides and particulate matter before burning of coal will require new technologies and processes that will upgrade low-rank energy resources to efficient, clean-burning fuels. These technologies will reduce and eliminate the economic and environmental impediments that limit the full

consideration of low-rank coals as a future energy resource.

Classification of coals is made on the basis of their rank which characterized in the natural series from lignite to anthracite. While low-rank coals are extremely hydrophilic, high-rank coals show inherently hydrophobic character, thus wettability of coal surface changes related to its unique chemical structure. On the other hand, polar groups such as carboxyl and phenols existed in low-rank coal lead to the better water adsorption effect adversely to high quality coals.

Existence of trapped water molecules in the pores and particularly at the particle surfaces makes coal more hydrophobic.

Chemical and physical features of coal can be changed with the removing of initial moisture and volatile matter from its original structure by applying of thermal heating process, therefore surface properties also change. It is obvious that such kind of excess species for coals cause enrichment difficulties, particularly for the flotation. Particle surfaces which are acquired hydrophobicity with pre-treatment as mentioned above, will be able to contact readily with collector reagent in flotation pulp. Flotation or oil agglomeration of lignite coal particles, in general, requires high quantities of oily collector to become floatable or achieve adhesion. This not only deteriorates the selectivity of separation but also uneconomic due to the adverse effect of the over-dosage.

EXPERIMENTAL

Material

Sub-bituminous washed coal sample taken from Soma-Manisa district, was subjected to this investigation. Clean coal samples representing of final product taken from coal washing plant contain 13.3 % ash, 18.1 % total moisture and 40.7 % volatile matter

on the dry basis. The size of the coals was reduced from 150 mm to 9 mm by a combination of laboratory type jaw and cone crusher. Standart coal analyses of coal sample are shown in Table 1.

Table 1. Standart analyses results of Soma washed coal.

Item	Original coal	Air-Dried coal	Dried coal
Moisture, %	18.12	12.40	---
Ash, %	10.85	11.62	13.26
Volatile matter, %	33.36	35.68	40.74
Fixed carbon, %	37.67	40.29	46.00
Total sulfur, %	0.84	0.89	1.02
Lower cal. Value, kcal/kg	4443	4807	5594
upper cal. Value, kcal/kg	4710	5039	5752

Method

Grinding and thermal heating experiments were conducted on with the coal samples which are crushed to minus 9 mm. Laboratory type ball mill with a dimension of 20 cm x 20 cm was used during dry grinding under the fixed ball charge. Tar quantity was added according to the dry coal weight which is used 500 grams for each experiment. Heat treatment experiments were performed in a 10 cm diameter cylindrical retort with an interior volume of 1500 cm³ as shown in the Figure 1. Around 650 gram of samples for each single tests were used.

During the every test, process output gases were collected throughout the cooling system which is connected to heater by pipe. All experiments were carried out between 300 °C and 600 °C of temperature with a duration time between 15 and 90 minutes. The samples charged in to the preheated furnace at determined (or certain) degree were taken out at the end of desired temperature and time. Weight losses were determined after cooling of samples to ambient temperature. Previously heated samples were ground in the dry ball mill with adding some amount of tar between 0.5 % and 4 % by weight. Finally, coal flotation with ground samples were carried out.

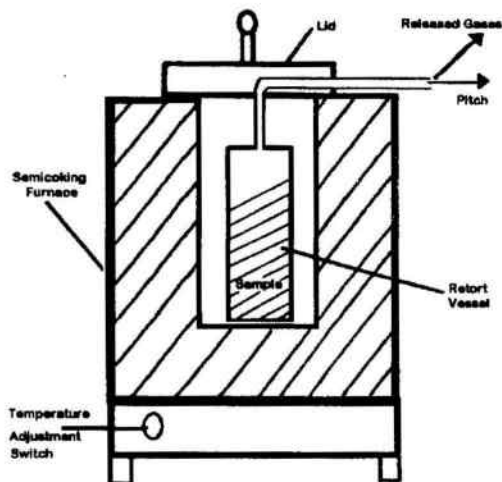


Figure 1. Laboratory type Heat Treatment Furnace

RESULTS AND DISCUSSION

During the flotation experiments with both untreated and treated coal samples, grinding size, heat treatment as well as tar additives were investigated as a main parameters in order to explain flotation behaviors.

Effect of Grinding Size for The Flotation

Representative samples which are crushed to minus 9 mm, were ground into three different size fraction. Due to the results, 97 % of coal by weight can be reduced to -0.5 mm at 20 minutes and 98% to -0.3 mm at 30 minutes while 80% of coal by weight can be reduced to -0.105 mm at 45 minutes. 0.5 mm size for the further experiments was chosen because of no significant differences for the flotation results were occurred although advanced of size reduction to finer size.

Effect of Tar Additives in Grinding Circuit for The Flotation

Tar quantity from blank to 4 % by weight for the untreated coal containing 18 % moisture was added during dry grinding followed flotation tests, that were carried out in Denver type flotation cell. Flotation parameters are given in Table 2. Effect of tar additives for the flotation recovery of untreated coal is also shown in figure 2.

Table 2. Flotation Parameters

Parameter	
Grinding size, mm	-0,5
Sample amount, grams	500
Solids ratio, %	20
Reagent type	90%kerosene+10% soOc.t.
Reagent amount, g/t	5000+2500
pH	Natural
Conditioning time, min.	5+5
Flotation time, minute	3+3

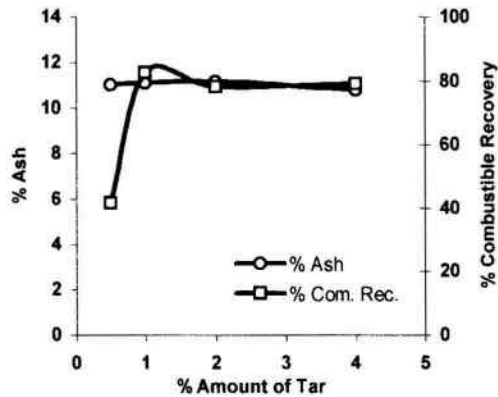


Figure 2. Effect of tar additives for the flotation behavior of coal

Acceptable flotation results in the terms of combustible recovery and ash content of coal were obtained in case of using 1 % tar additives. Thus, This amount was fixed for the further experiments during this investigation.

Effect of Heat Treatment for the Flotation

Original coal samples crushed to -9 mm were involved to heat pre-treatment at 300; 400; 500 and 600 °C with a duration time between 15 minutes and 90 minutes for each temperature condition. At the end of each heat pre-treatment study, samples were cooled in to the 70-80 °C temperature under the atmospheric condition, then, grinding in the ball mill by adding 1 % tar was performed with a 20 minutes grinding time in order to obtain desirable flotation size. Ground samples were also introduced to the coal flotation as explained its parameters before.

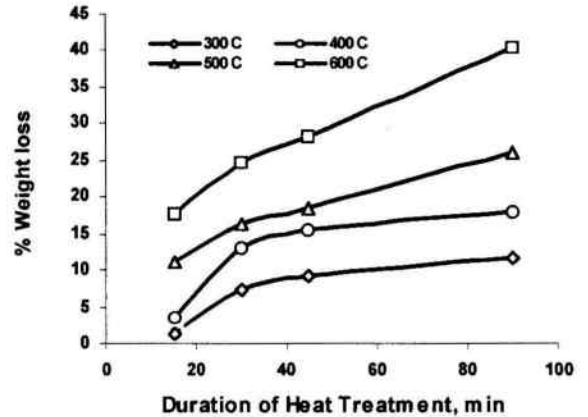


Figure 3. The effect of heating temperature and time for weight losses

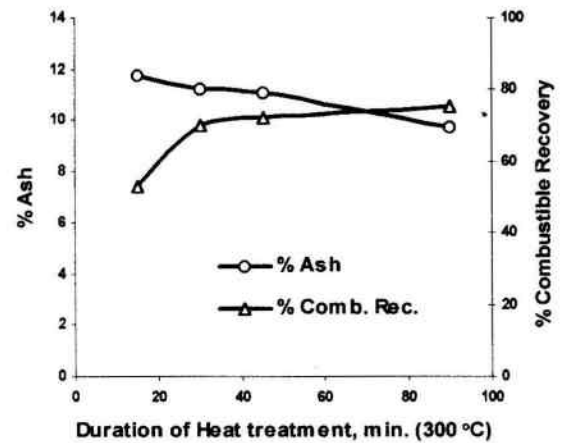


Figure 4. The changing of combustible recovery and ash content of clean coal as a function of heat pre-treatment at 300°C

Figure 3 shows the effect of heating temperature and time for weight losses in terms of volatile matter removal from original coal structure. Combustible recovery and ash content of clean coals obtained from flotation are illustrated as a function of heating temperature between 300°C and 600°C as well as heating time between 15 minutes and 90 minutes for each temperature intervals, in figures 4; 5; 6 and 7 respectively.

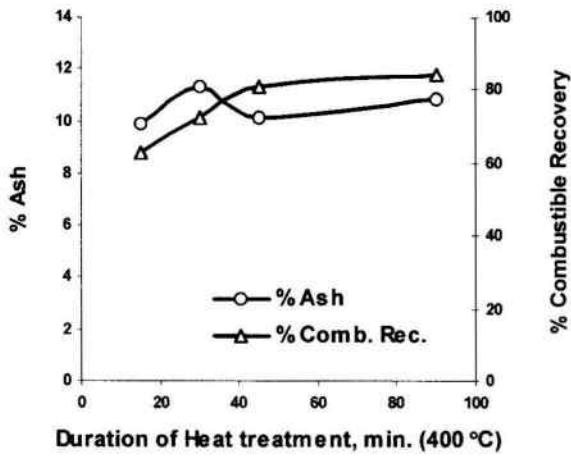


Figure 5. The combustible recovery and ash content of clean coal as a function of heat pre-treatment at 400°C

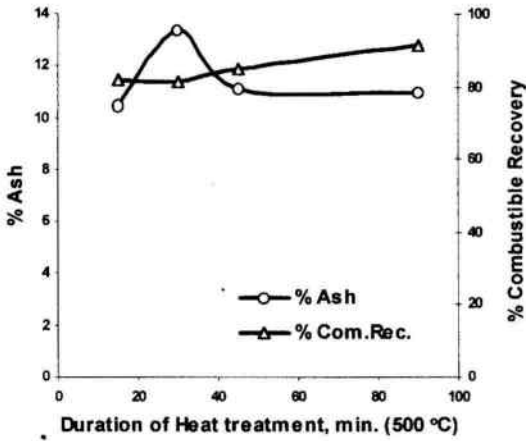


Figure 6. The combustible recovery and ash content of clean coal as a function of heat pre-treatment at 500°C

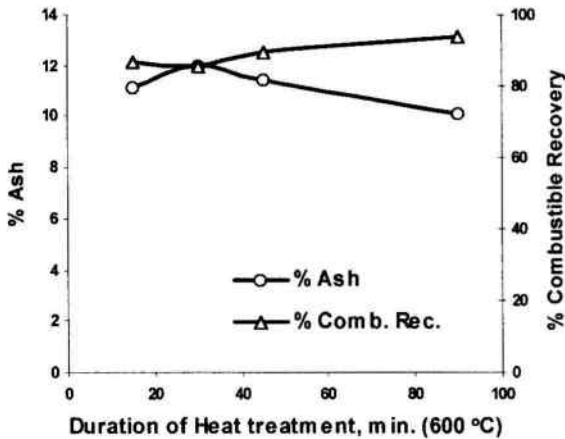


Figure 7. The combustible recovery and ash content of clean coal as a function of heat pre-treatment at 600°C

According to the thermal pre-treatment at 300 °C and 400 °C, no significant reduction both in moisture and volatile matter was observed, therefore, no promising success for flotation was achieved. However, it is noticed that volatile matter removal with an acceptable ratio started at beyond 500 °C thermal heating level. By the way, combustible recovery of clean coal increased sufficiently by flotation although no increases was observed for ash content. These results indicated that explicit relation can be pronounced between floatability of coal and its moisture and volatile matter content. Some oxygen containing groups can be distracted on sub-bituminous coal structure with a thermal heating pre-treatment at above 500 °C temperature. During this case, volatile gases such as CO, CO₂, O₂, H₂ and CH₄ can readily be removed from original coal structure (4). This phenomena can be expressed as an "artificial metamorphism" which is caused to the increases fixed carbon level while decreases oxygen and hydrogen constituents of coal. On the other word, increases of fixed carbon contents on coal structure related to the thermal heating process, causes increasing of coal rank artificially. It is well defined by several scientific investigation that hydrophobicity of coal surface increases increasing with its rank (5). As well known, flotation ability of coals can be ordered from sub-bituminous coal which contains higher moisture and volatile matter with a lower level fixed carbon to bituminous coal having opposite properties with sub-bituminous coal.

Due to the commitment of time during 300 °C and 400 °C thermal heating pre-treatment, no significant increases were observed on combustible recovery and ash content of clean coal till 45 minutes duration time. Whereas noticeable increases on combustible recovery of clean coal were determined depending on 90 minutes thermal heating both in 300 °C and 400 °C temperature levels.

Although there is no positive progress on flotation behavior of coal till 15 minutes duration time on both 500 °C and 600 °C heating temperature, however, it is noticed that successfully separation with flotation was achieved after 45 minutes duration time on above related temperature levels.

Clean coal having 10.1 % ash content with a 93.9 % combustible recovery can be obtained while middling and tailings contain 47 % and 52 % ash content respectively in case of flotation of 600 °C preheated sample in 90 minutes duration time.

Due to the overall test results, combustible recovery of clean coals acquired by flotation depend on different thermal heating temperature are shown in

Figure 8. Figure 8 summarize that increasing the temperature causes a significant increases in combustible recovery of clean coal.

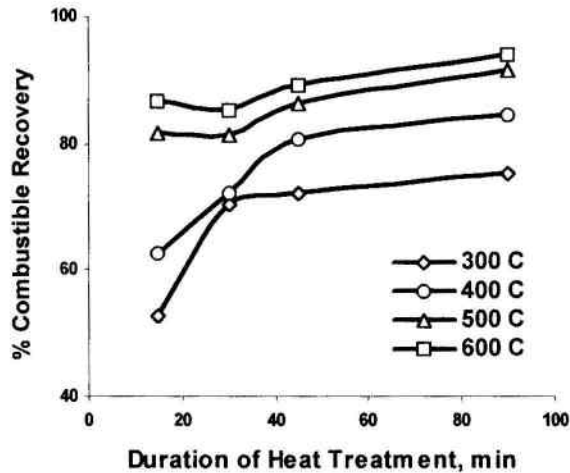


Figure 8. Combustible recovery of clean coal as a function of heat treatment

CONCLUSIONS

Based on the results presented in this investigation, we can end:

- 1- Moderate-temperature heating can be improve hydrophobicity of sub-bituminous coals, thus causes to create positive effect for the flotation. In our case better results were obtained increasing with heating temperature up to 600 °C. Similarly, duration time during heating process also creates positive effect for the flotation ability of coal particles.
- 2- The effect of tar additives on the floatability of coal is marginal. Hence 1 % tar adding also improved hydrophobicity of coal surfaces actively.
- 3- Although tar as an organic matter covers presumably coal surface, it is thought that adsorption kinetics of tar on coal surfaces should be proofed with further investigation.
- 4- It is possible that super quality clean coal can be successfully obtained with extension of clean circuit stage in the flotation process.

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