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Flotation and leaching of hard coals for production of low-ash clean coal

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Abstract: Coal has been one of the most important fossil fuels meeting the world`s commercial energy demand for decades. The use of coal in electricity production is an inevitable alternative for developing countries such as Turkey. This study focused on the cleaning process of Zonguldak coal for the production of low ash, low sulphur, and high-calorie coal concentrate. Flotation and atmospheric/autoclave leaching experiments were applied for this purpose. A product with 1% ash, 0.37% sulphur and 8390 kCal/kg of calorific value was obtained under the best experimental conditions.

Keywords: flotation, hard coal, autoclave leaching, atmospheric acid/base leaching

1. Introduction

The common feature of all possible situations related to world energy supply is the expectation that the energy need will increase in the 21st century. Coal has the largest fossil fuel reserve in the world and will retain its place in meeting the world's energy needs. Approximately 29% of global energy consumption in 2015 provided by coal (BP, 2017). The direct combustion efficiency of coal is relatively low and CO_2 emissions are higher than other methods. There is a global concern about the use of coal for energy production. The reason is that conventional coal-fired power plants spread high levels of greenhouse gases (GHG), mostly carbon dioxide (CO_2) and nitrous oxide (NO_X), sulphur dioxide (SO_2), and some particles (IEA, 2017). To stop global warming, many countries around the world aim to limit greenhouse gas emissions from coal. However, this is not an easy task to implement. Theoretically, two basic solutions are proposed to overcome this problem. The first one is to reduce coal consumption for energy production. Nevertheless, this is not possible in the short-medium term due to the increasing need for global energy. The other solution is to turn coal into less polluting or cleaner fuel, which is more suitable for energy generation than the first solution (Melikoglu, 2018).

A significant portion of the ash-forming mineral substances (such as carbonates, sulphates, phosphates, oxides, and sulphides) which are commonly found in the structure of coal, must be removed before the coal combustion. This process prevents erosion and corrosion in coatings during the combustion of coal and eliminates the need to apply some emission reduction processes to combustion gases.

Flotation method is widely used to remove mineral matter from coal and to produce coal concentrate with low ash and sulphur content (Umit, 1997). There are important flotation applications in different countries around the world such as the United States, Australia, China, India, Germany, and Russia (Bentli, 2000). Various studies have been conducted on the coal production process with low ash and sulphur (Atak and Onal, 1991; Ozbayoğlu and Bilgen, 1997; Steel and Patrick, 2001; Karaca and Onal, 2003; Sonmez et al., 2005; Dash et al., 2007). In these studies, the efficiency of acid/base leaching methods was investigated besides flotation to reduce the ash and sulphur content of coal, and significant results were obtained from these studies.

Before the Second World War, the first studies on clean coal production were done by Crawford in Germany (Crawford, 1951). Reggel et al. (1976), Stambaugh (1978), Yang (1979), Waugh and Bowling

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(1987) developed processes involving the use of NaOH. In these processes, mineral substances were removed from coal in NaOH solution at 200°C. Aluminosilicate compounds were converted into insoluble sodalites under the specified conditions and adsorbed in coal matrixes. The acid leaching can be applied to dissolve sodalites which are insoluble in NaOH solution. Bolat et al. (1998) studied on demineralization of coal using different acids (HF, HCl, HNO_3, and H_2SO_4) alone and in combination with 0.5 N NaOH solution. After extraction with 0.5 N NaOH, the sample was washed with of 10% HCl solution. It is concluded that when the NaOH leaching process is carried out, the coal ash content can increase due to the formation of sodium salts. Waugh and Bouling (1984) removed 90% of the mineral matter of coal by using NaOH solution under pressure at a temperature of 220°C. Wang et al. (1986) worked on a 15.5% ash containing coal and reduced the ash content to 2.2% using NaOH solution under pressure at a temperature of 460°K. According to another study, a clean coal concentrate containing 12% ash with the 92% ash removal was produced from a coal containing 55% ash by reverse flotation (Stonestreet and Franzidis, 1988).

In 1994, one of the earliest studies on CCTs was realized by Ozbayoğlu and Mamurekli in Turkey. In this study, they found that 2.2% ash and 0.4% total sulfur-containing coal could be produced from Zonguldak bituminous coal by using heavy medium separation and flotation methods (Ozbayoglu and Mamurekli, 1994). Ateşok et al. (2001) produced a concentrate containing 8.3% ash with 81% combustible yield using a 16.3% ash containing coal. Muknerjee and Borthkur (2001) removed 43-50% of ash and sulfur using NaOH and HCl in their study. In a study managing by Gubta et al. (2007), yielded approximately 11% ash clean coal from 24.5% ash coal via flotation process. Yasar et. Al (2018) produced clean coal from tailings containing 3.0% sulphur and 54.6% ash from the Tuncbilek Coal Washery of Turkish Coal Enterprise with nearly 1.3% sulphur and 29.9% ash content. Finally, and most recently, Sahinoglu (2018) cleaned a high sulphur ash coal by using the flotation method. In this purpose, this study aimed to produce a super clean coal concentrate with low ash and sulphur contents to be used in internal combustion engines by the combination of flotation and leaching methods.

2. Materials and methods

2.1. Materials

The hard coal sample obtained from Zonguldak Kozlu was used in the study. The coal sample was in the size of 2 - 5 mm with a higher calorific value of 7715 kcal/kg, the ash content of 10.62%, a volatile matter of 28%, constant carbon content of 62%, and sulphur content of 0.39%. The coal sample was crushed below 1 mm, and the wet ground below 150 µm using a ball mill. The XRD analysis of the sample was performed by powder X-ray diffraction (XRD) using RIGAKU-Dmax-2200 PC equipment, operating at 20 kV and 40 mA with Cu–Kα radiation. The XRD patters of the sample is shown in Fig. 1. Based on the XRD diffractogram given in Fig. 1, the coal sample contained mostly clay, pyrrhotite, dolomite, calcite, and quartz minerals.

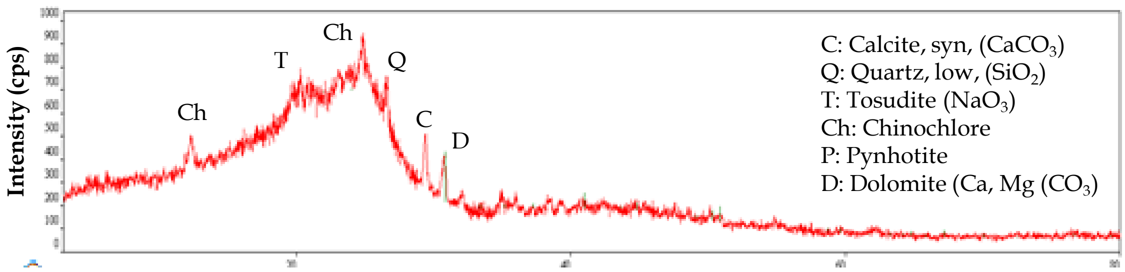

Fig. 1. XRD results of the coal sample

2.2. Methods

2.2.1. Flotation experiments

A sub-A flotation machine with speed control was used for flotation experiments which were conducted in a 2 dm^3 flotation cell. A kerosene/water emulsion was prepared using an IKA-Eurostar stirrer, this emulsion was used as a collector for the flotation experiments at 50, 300, and 550 g/Mg concentrations. The experiments were performed at solid/liquid ratios (W/W) of 10%, 15%, and 20%. Aerofroth 70 (Solvay) was used as a frother in flotation at concentrations of 20, 50, and 80 g/Mg. Na_2SiO_3 (MERCK, 122.06 g/mol) was used as a depressant for silicates with 1000, 3000, and 5000 g/Mg dosages. 5 min. of conditioning time, 1500 rpm agitation speed, 10 min. of flotation time, and 1060 rpm agitation speed were applied in the flotation experiments. The airflow rate was kept as 1.2 dm^3/min during the flotation. Tap water was used for the experiments at room temperature.

The results of the flotation experiments were evaluated based on weight recovery, ash content, and combustible recovery values. The combustible recovery was calculated using Eq. (1):

$$Combustible\ Recovery\ (\%) = \left[\frac{M_c(100-A_c)}{M_f(100-A_f)}\right] x100 \quad (1)$$

where A_c is ash content of clean coal, A_f is ash content of the feed, M_c is mass of clean coal, and M_f is mass of feed.

2.2.2. Leaching experiments

In the present study, atmospheric acid leaching experiments were performed using HCl (MERCK, 37 % solution, v/v). The experiments were conducted in an Erlenmeyer flask at 5% solid/liquid ratio (w/w) under the following experimental conditions; 2, 4, 6, mol/dm^3 HCl; 40, 60, 80°C; 1, 3, 5 h of leaching time.

In basic atmospheric leaching, the acid leaching product with 5% was subjected to NaOH leaching at 80°C for 3 h. NaOH (MERCK, 40 g/mol) was used in the basic atmospheric leaching experiments at 1, 2.5, and 4 mol/dm^3 concentration in an Erlenmeyer flask.

The pressure basic leaching experiments were performed in a 1 dm^3 titanium autoclave (Parr Inc., USA) equipped with a heating mantle, a PID temperature controller and a variable speed stirrer operated at 600 rpm. In the basic pressure leaching experiments, the acid leaching product mixed with 4-2.5-1 and 0.5 mol/dm^3 NaOH (MERCK, 40 g/mol) under pressure in the autoclave. The slurry was subjected to different temperatures at 220-180 and 130°C for 1, 2, and 3 h.

After each leaching experiment, the leachate was removed by filtering. The residue was washed with warm distilled water, dried at 80°C, and subjected to ash and sulphur analysis.

2.2.3. Flowsheet of the experimental program

The flowsheet given in Fig. 2 was used in the laboratory studies. The flotation method was applied to remove mineral matter from coal in the first stage. The flotation concentrate obtained by the application of the optimal flotation conditions was subjected to HCl leaching after grinding below 45 µm to determine the optimal HCl leaching conditions. The leaching product which was obtained under the optimal HCl leaching conditions was subjected to atmospheric and autoclave NaOH leaching to obtain a super clean coal concentrate.

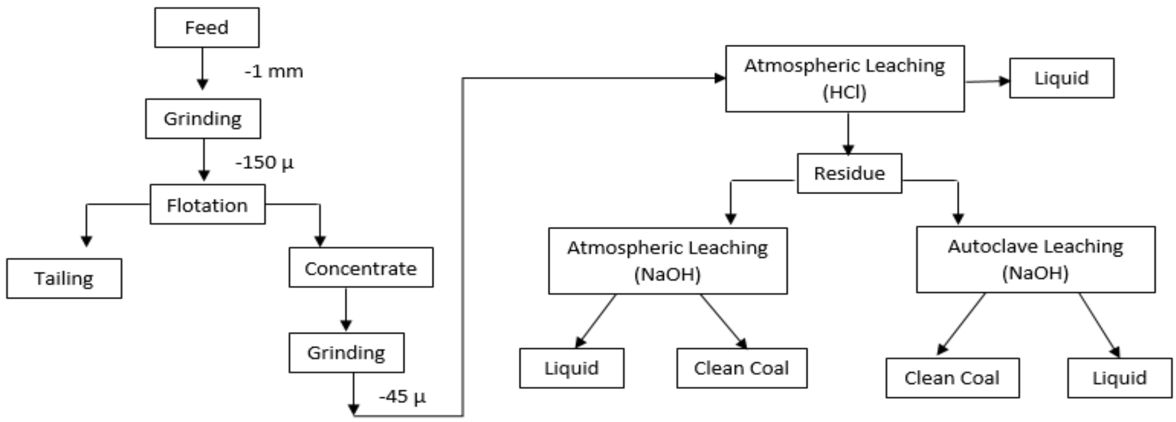

Fig. 2. Flowsheet of the experimental procedure

3. Results and discussion

3.1. Flotation experiments

The flotation experiments were carried out at several parameters to obtain a clean concentrate with high recovery. The results of the flotation experiments conducted with different experimental parameters are shown in Figs. 3(a), (b), (c), and (d).

The first group of flotation experiments were performed to determine the effect of solid/liquid ratio (w/w) on the properties of the coal concentrates. As can be seen from Fig. 3(a), the increasing solid/liquid ratio (w/w) caused an increase in the ash content of the coal concentrate. As the aim of the study was to produce the cleanest concentrate with acceptable yield values, 10% of solid/liquid ratio (w/w) was chosen as the optimal solid/liquid ratio (w/w) for further flotation experiments.

The second group of experiments were performed to find out the optimal collector concentration. The results of the experiments are shown in Fig. 3(b). As can be seen from Fig. 3(b), the combustible recovery increased until a plateau value around 300 g/Mg kerosene, and did not increase by increasing the amount of kerosene to 550 g/Mg.

The third group of flotation experiments were conducted to determine optimal depressant concentration. As shown in Fig. 3(c), it was observed that the increasing concentration of Na_2SiO_3 helped to reduce the ash content of the coal concentrate but also decreased yield values.

The last group of experiments were performed to find out the effect of using different amount of frother in the flotation process as seen in Fig. 3(d). The results suggested that the use of the increasing amount of frother in flotation increased the combustible recovery along with the ash content.

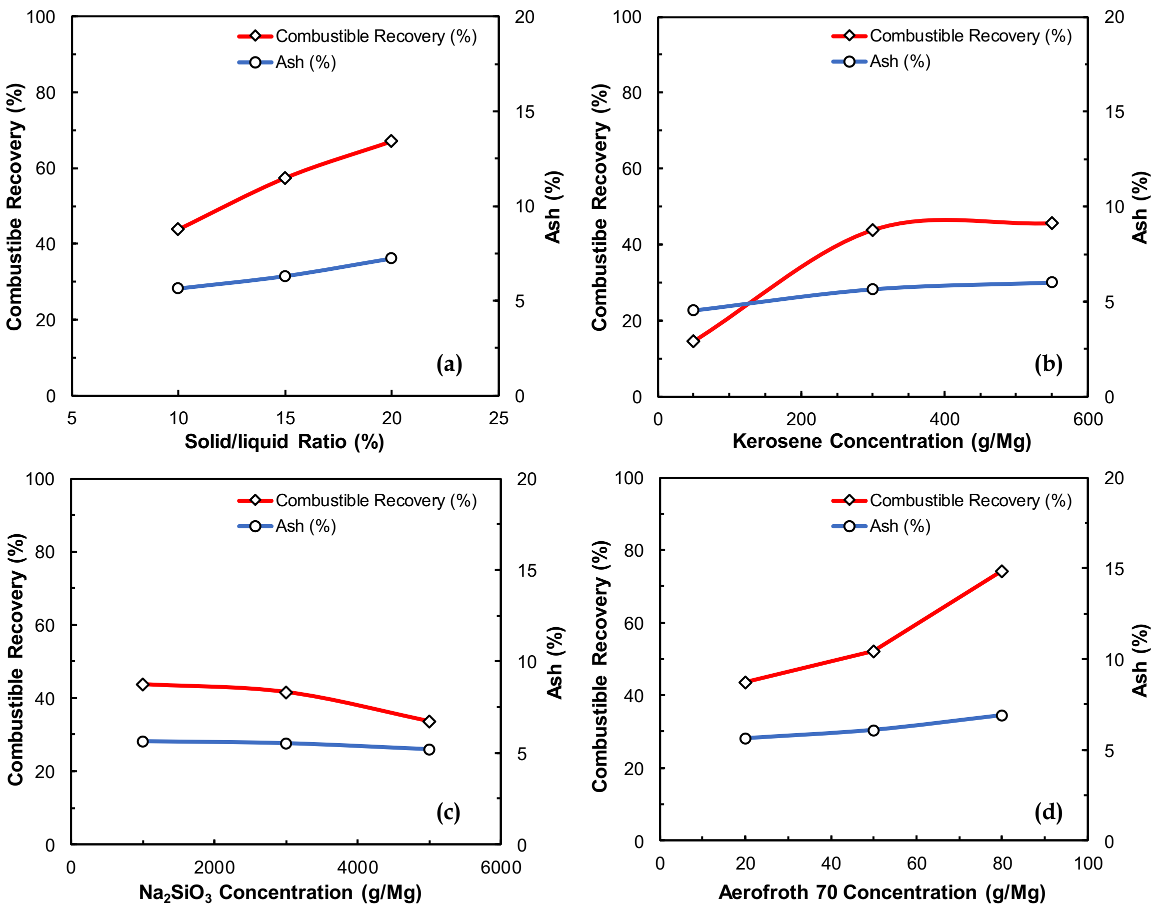

Fig. 3. Effect of parameters on flotation recovery and ash content (a) Solid/liquid ratio (300 g/Mg kerosene, 1000 g/Mg Na_2SiO_3, 20 g/Mg Aerofroth 70), (b) Kerosene concentration (1000 g/Mg Na_2SiO_3, 20 g/Mg Aerofroth 70 with 10% solid/liquid ratio), (c) Na_2SiO_3 concentration (300 g/Mg kerosene and 20 g/Mg Aerofroth 70 with 10% solid/liquid ratio), (d) Aerofroth 70 concentration (300 g/Mg kerosene and 1000 g/Mg Na_2SiO_3 with 10% solid/liquid ratio)

According to the results obtained from the flotation experiments, the optimal flotation conditions for producing the cleanest coal concentrate by flotation were determined as 10% solid/liquid ratio (w/w), 300 g/Mg kerosene, 1000 g/Mg Na_2SiO_3, and 20 g/Mg Aerofroth 70. Under these optimal conditions, a concentrate with 5.63% of ash with 41.57% recovery was obtained. The properties of the clean coal concentrate are presented in Table 1, and the XRD results of the flotation products are shown in Fig. 4. As can be seen from Fig. 4, the amount of ash-forming minerals such as clay, dolomite, calcite, pyrrhotite, and quartz minerals in the flotation concentrate decreased while the amount of these minerals increased in the flotation tailing.

Table 1. Properties of the clean coal concentrate produced under the optimal flotation conditions

	Weight (%)	Ash (%)	HHV*(kcal/kg)	Combustible Recovery (%)
Concentrate	41.57	5.63	8130	43.69
Tailing	58.43	14.17	7273	56.31
Run of mine	100	10.62	7715	100

* Higher Heating Value

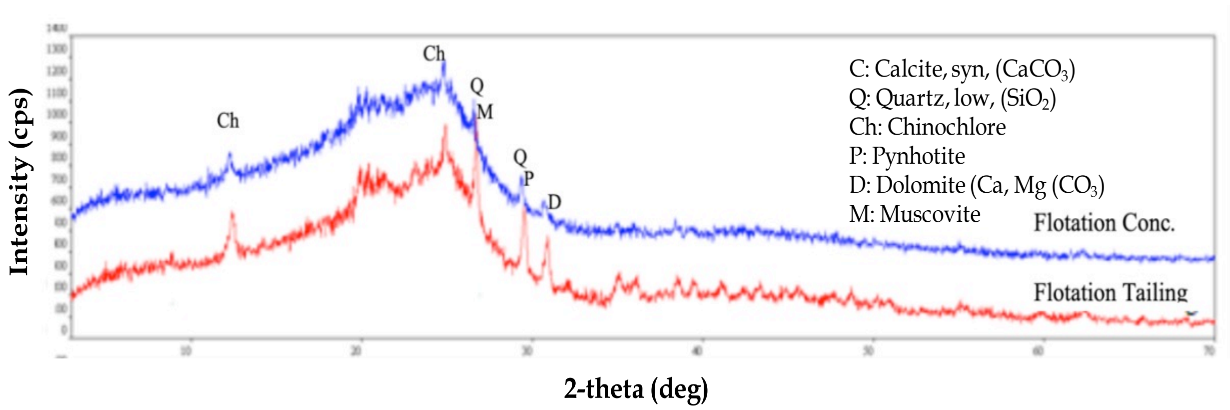

Fig. 4. XRD results for the flotation products

3.2. Leaching experiments

The results of the flotation experiments indicated that only a limited reduction in the ash content of coal samples could be obtained by the flotation experiments. Therefore, the required amount of flotation concentrate was produced under the optimal conditions of flotation to use in the leaching experiments.

3.2.1. HCl leaching

HCl leaching method was utilized to remove the carbonates and iron oxides from the coal body. The experiments were conducted with 5% solid/liquid ratio (w/w). The effect of different parameters such as HCl concentration, temperature, and leaching time was investigated. The results are shown in Figs. 5(a), (b), and (c).

The results of the HCl leaching experiments showed that only a limited increase in the ash removal efficiency was achieved by increasing the concentration and leaching time in the range of the experimental conditions applied. However, as shown in Fig. 5(c), the temperature showed a significant effect on the removal of ash from the coal. Under the best conditions (3 h – 2 mol/dm^3 HCl at 80℃), 32% of ash was removed from the coal concentrate, and a product with 3.7% ash and 0.38% S was obtained by the flotation followed by HCl leaching.

3.2.2. NaOH leaching

Before starting the leaching experiments with NaOH, the required amount of clean coal product was produced from the run of mine coal by applying the optimum conditions of flotation and HCl leaching sequentially. Atmospheric and pressure NaOH leaching techniques were used to remove SiO_2 and sulfur content of the flotation concentrate.

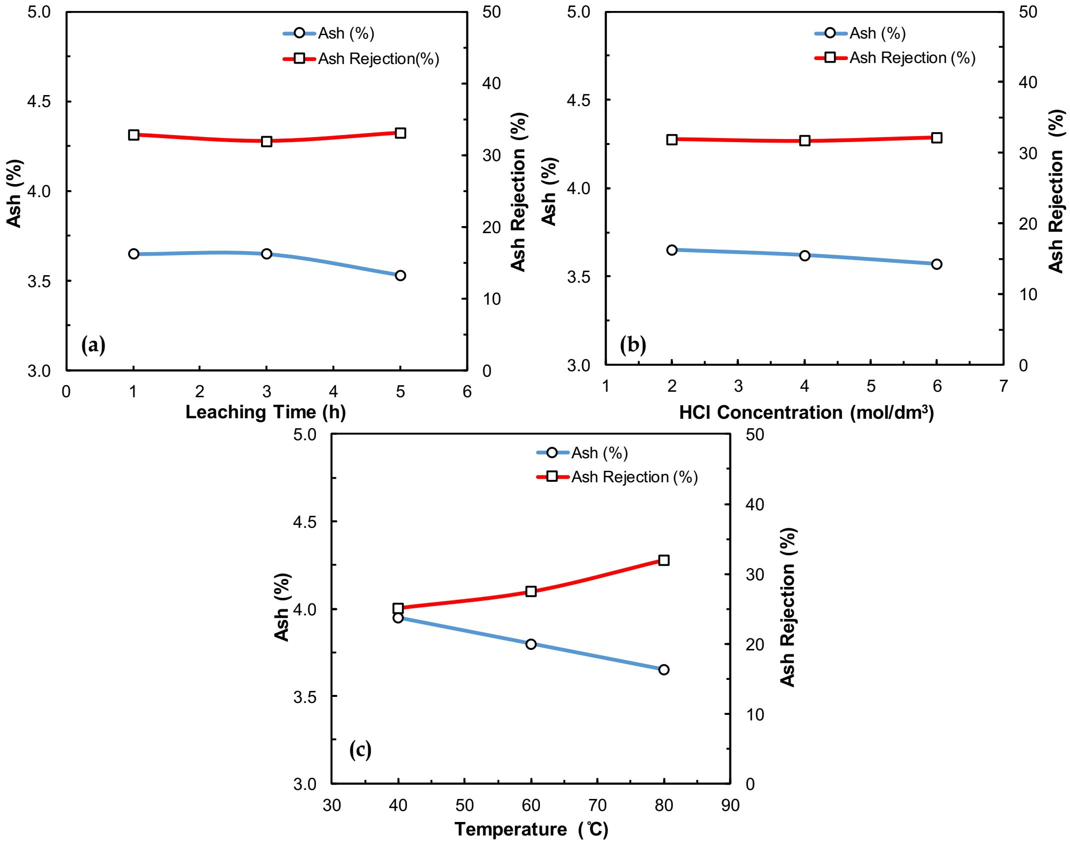

Fig. 5. Effect of parameters on ash rejection and ash content: (a) Leaching time (2 mol/dm^3 HCl at 80^{o}C), (b) HCl concentration (80^{o}C temperature and 3 h leaching time), (c) Temperature (2 mol/dm^3 HCl and 3 h leaching time)

3.2.2.1. Atmospheric NaOH leaching

Mineral matter removal from HCl treated Zonguldak coal flotation concentrate by NaOH leaching with different concentrations of NaOH solutions under atmospheric conditions was investigated at 80^{o}C temperature and 3 h of leaching time (Fig. 6). As seen from Fig. 6, a coal concentrate containing 2.7% of ash and 0.37% of sulphur was obtained with a higher calorific value of 8230 kcal/kg.

3.2.2.2. Autoclave NaOH leaching

In this part of the study, the extent of ash reduction in HCl-treated Zonguldak coal flotation concentrate was investigated by applying pressure NaOH leaching. The effect of NaOH concentration and temperature at 1 h leaching time was investigated thoroughly. The results are shown in Figs. 7(a), (b), and (c). Under the optimum conditions, a coal concentrate containing 1% of ash and 0.37% of sulphur was obtained with a higher calorific value of 8390 kcal/kg. The NaOH leaching experiments carried out at 220°C temperature and 0.5 mol/dm^3 solution concentration, the ash content of the concentrate was 1.16%, at 130°C in 4 mol/dm^3 NaOH solution, the concentrate ash content was 1.29%. In addition to that at 180°C temperature in 1 mol/dm^3 NaOH solution, the concentrate ash content was 1.04%. These experimental conditions were chosen as the optimum conditions due to low NaOH consumption and temperature values. The results showed that the high-pressure autoclave leaching was very effective to obtain a super clean coal concentrate.

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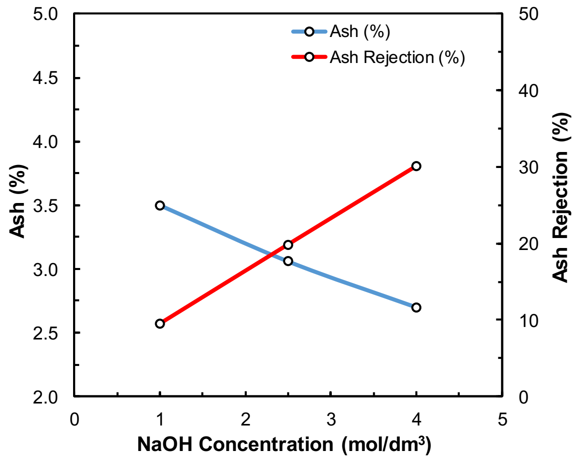

Fig. 6. Effect of NaOH concentration at 80ºC temperature and 3 h of leaching time

3.3. Discussion

As it was stated earlier, the presented study aimed to develop a sequential process to decrease the mineral matter content of the coal sample. For this purpose, flotation, HCl and NaOH leaching (including atmospheric and pressure leaching techniques) methods were applied sequentially. The overall results are presented in Table 2. The results show that it is possible to produce a low ash coal product from Zonguldak coal containing around 1% ash by applying the sequential process suggested in this study.

Table 2. Summary of the final results of the study

Product	Weight (%)	Ash (%)	S (%)	HHV (kcal/kg)
Feed coal sample	100	10.62	0.39	7715
Flotation concentrate	29.35	5.63	0.38	8130
HCl leaching product	28.59	3.7	0.38	8206
Atmospheric NaOH leaching product	27.28	2.7	0.37	8237
Autoclave NaOH leaching product	25.78	1	0.37	8390

Additionally, the chemical composition of the final product analyzed via the XRF method at ARGETEST laboratory services. Based on the results from the study, the amount of SiO_2 in ROM ore reduced from 56% to 38% in the final product. On the other hand, CaO% in ROM ore was 8.5% while it was 2.7% in the final product. In addition to these results, another significant decrease was obtained on Fe_2O_3, in ore body amount of Fe_2O_3 was 8.5% while it was 4% in the super clean coal concentrate.

In the literature, it can be found that various researchers worked on producing super-clean coal concentrates from Zonguldak coal by applying various methods. Ozbayoğlu and Mamurekli (1994) worked with Zonguldak coal applying heavy media separation and flotation sequentially and produced a coal concentrate with 2.16% ash and 0.42% total sulfur from a feed coal containing 12.11% ash and 0.41% total sulphur. Hacifazlioglu (2016) studied the chemical leaching with aqueous HF, HCL, HNO_3,

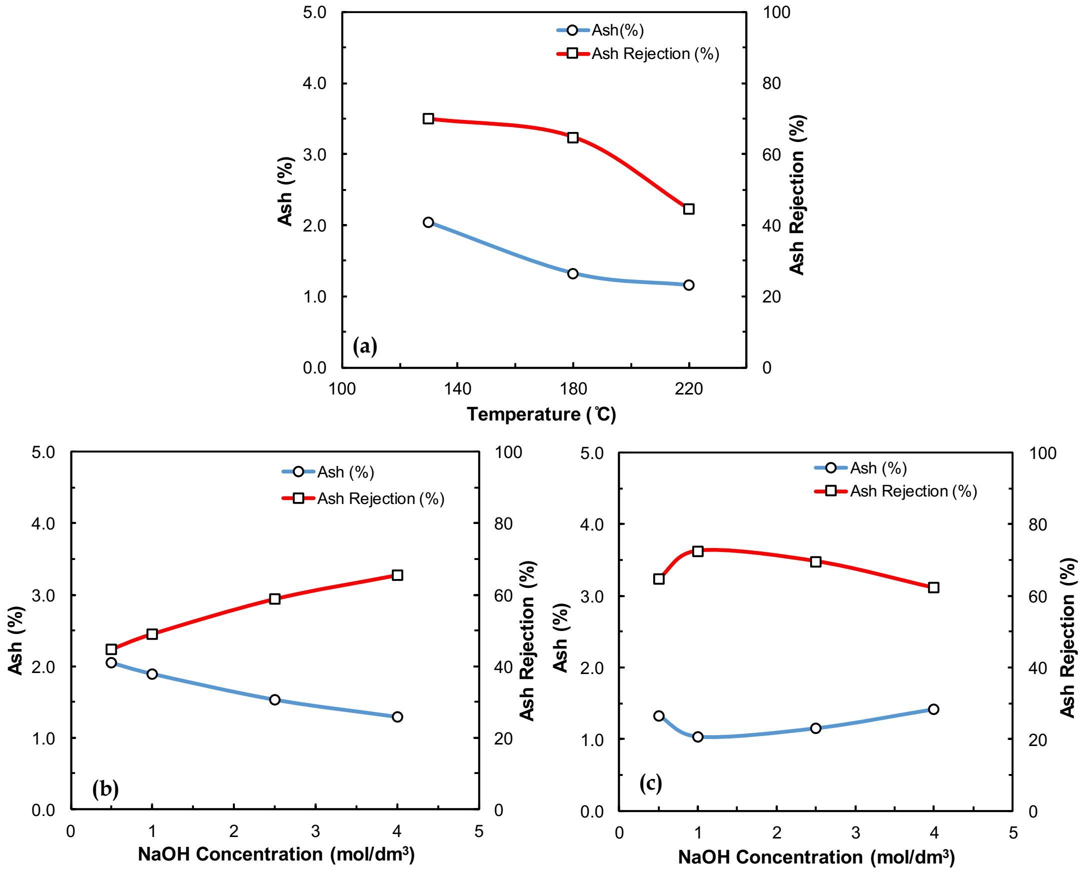

Fig. 7. Effect of parameters on ash rejection and ash content: (a) Temperature (0.5 mol/dm^3 NaOH and 1 h of leaching time), (b) NaOH concentration (130^{o}C temperature and 1 h of leaching time), (c) NaOH concentration (180℃ temperature and 1 h of leaching time)

and citric acid (CA) to produce ultra-clean coal (UCC) from Zonguldak bituminous coal. As a result of a leaching operation of 240 min with a concentration of 25% HF and at 80°C, a clean coal product containing 0.82% produced from Zonguldak coal with 8.84% ash content. Dikici and Hacifazlioglu (2019) used vegetable oils (sunflower and eucalyptus oil) and organic acids (citric and formic acid) to produce super-clean coal concentrate using flotation and leaching sequentially. A clean coal concentrate with 2.92–2.98% ash was obtained by flotation followed by citric acid and formic acid leaching at 65°C for 3 h from Zonguldak coal containing 29.67% ash.

In the presented study, a three-stage sequential process including flotation with kerosene followed by acid and basic leaching steps (atmospheric and pressure leaching) was successfully implemented. The developed process converted 25.78% of the feed coal into a low ash product with 1% ash. The tailing of the process contains about 14% ash and it can still be used in various sectors as fuel. Although the processes and chemicals utilized are different, the results of the study can be considered in agree with the data found in the literature.

Coal water slurry as a substitute for fuel oil provides high combustion efficiency, low ash discharge and can benefit from lower SO_2 and NOx properties compared to raw coal. Thus, coal water slurry may help a transition economically, away from Turkey's limited resources of oil and natural gas, to Turkey's most abundant resource of energy in an environmentally friendly way. The coal products should contain less than 2% ash to be used in coal-water slurries for burning in diesel engines and the produced low ash coal concentrate can be used in the coal-water slurry production.

4. Conclusions

In this study, flotation and two different NaOH leaching techniques were applied with a three-stage beneficiation process to produce a super clean coal concentrate from Zonguldak, Kozlu coal. As a result of the process applied in the study, the ash content of the run of mine coal was reduced from 10.62% to

2.7% with 27.2% of weight recovery with atmospheric leaching. Moreover, the ash content of the run of mine coal was reduced from 10.6% to 1% with 25.78% of weight recovery by the autoclave leaching. Although no significant decrease was observed in the sulphur content of coal, the sulphur value (0.39%) remained within acceptable limits. The results of this study indicate that it is technically possible to obtain a super clean coal concentrate from Zonguldak hard coal by using a multistage beneficiation process. The ash content of the produced low ash coal meets the requirements for the production of coal-water slurry to be utilized as a fuel in diesel engines, gas/oil boilers and coal boilers.

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