REDUCTION OF SULFUR AND ASH CONTENTS FROM LOW-RANK COAL USING FROTH FLOTATION TECHNIQUE

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This research attempted to reduce sulfur and ash content for enhanced coal recovery by the froth flotation technique. Dukki district (Pakistan) coal was upgraded using a flotation technique followed by acid leaching. Tests on flotation concentrate were carried out to improve the grade of coal further. Tests were carried out to investigate and determine the effect of critical parameters like particle size, pH, and pulp density on reducing ash and sulfur content from coal. The particle size was observed to have the most significant role in coal ash and sulfur reduction, followed by the pH, and pulp density, respectively. The optimum particle size, pH, and pulp density values were 74 µm, 9 and 10%, respectively. The results from the flotation study on a laboratory scale at optimized conditions were a 54.1% increase in total carbon, a 42.12% reduction in ash, and a 38.32% reduction in sulfur. Final ash and sulphur contents in clean coal were 15.2% and 1.65%, respectively. Thus, clean coal can preferably be used in power plants and the cement industry in the country.

Keywords: froth flotation; acid leaching; pulp density; sulfur reduction

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1. Introduction

Coal is one of the most significant fossil fuels used worldwide, accounting for 60 to 80 % of the total commercial energy sources consumed. It has been assured that 909 billion tons of coal are reserved worldwide. Coal is the most abundant resource, which provides a diverse raw energy source for industrial processes. Coal helps deliver more economical and stable electricity to reduce the energy crises and increase economic growth. Pakistan is one of those countries with a considerable amount of coal in each province, estimated at 186,282 billion tonnes, of which 459 million are in Baluchistan. Unfortunately, in Pakistan, industrial development is badly affected by severe energy issues and crises due to a lack of integrated planning and proper utilization of its resources [1]. Despite ample coal resources, Pakistan has limited coal utilisation, typically for power generation and other purposes, due to technical issues. That's why energy crises are becoming more challenging. In Pakistan, the major portion of coal has not been appropriately utilised in power generation, cement manufacturing, and steel making because Pakistan's coal does not meet the requisite standard without its cleaning and upgrading [2]. To encounter this problem, coal must be upgraded or cleaned by exploiting physicochemical characteristics. The problem is attributed to the high content of sulfur, ash, and other impurities. These impurities limit coal utilization, which needs to be removed or decreased using coal cleaning or coal up-gradation techniques [3]. The inherent impurities are negligible in proportion/parts, and these impurities are also very hard to reduce unless some unique separation/cleaning techniques, such as alkali leaching methods, are utilized thus, we focus primarily on reducing extraneous impurities [1-2, 4].

Different methods are being practiced for the cleaning of coal [2, 5–8]. The physical coal cleaning process helps us in coal cleaning based on the difference in the physical properties of pure coal and impurities [6]. Froth flotation is a widely used metallurgical technique in mineral processing, wastewater treatments, etc. [6]. This is one of the best methods to enhance the surface properties, i.e., hydrophobicity, to reduce coal ash and sulfur content by optimized flotation parameters. This technique is found to be very environmentally friendly and economical for the separation of pyrite sulfur from coal. The simplicity of this method and low operating cost should be the additional features of the froth flotation. Physical treatment includes flotation, magnetic separation, or the use of hydrocyclones and mainly removes inorganic sulfur, whereas elimination of organic sulfur requires, in most cases, chemical and/or microbial treatment [9].

An essential criterion of separating minerals by the froth floatation method is that the size of the particles of the ores or coal must be minimally equivalent to powder form [7]. This is very important because the heavier and bigger particles would require a greater adhesive force, without which they would no longer attach to the froth and settle down in the bottom [7–9]. Thus, separation will not be possible.

The current study aims to enhance coal quality by reducing ash and sulfur using froth flotation and post-chemical cleaning methods. The flotation effect will be studied based on varying parameters, i.e., particle size, pH, and pulp density.

2. Materials and Methods

2.1. Materials

Coal samples for this research were collected from different mines of the Dukki coal field located about 300 km from Quetta near Loralai. About 45 kg of coal was collected to cover the entire area, about 300 sq. km. About 1 kg of coal was collected from each mine in air-proof bags properly labeled at different locations. The collected samples were subjected to crushing, grinding, sieving, and flotation to minimize the effects of oxidation and moisture adsorption.

2.1.1 Preparation of reagents

One molar solution of NaOH and HCl was prepared as per ASTM E 200-97 standard for modifying or controlling the slurry's pH range [11]. First, a beaker of 1000 mL was cleaned with distilled water, and the 40-gram solid NaOH was poured into it [5]. 700 ml of water was poured to avoid a violent reaction. The beaker was then filled up to the scale of 1000 ml, shaken well, and labelled as a 1M NaOH solution. The same procedure was followed for the HCL solution.

2.1.2 Sample sizing

The comminution or size reduction of the head sample was carried out primarily through the laboratory Denver Jaw Crusher. The product of coal obtained from the jaw crusher was then subjected to further size reduction by the Laboratory Rod Mill located in the Mineral Processing Laboratory. After the size reduction of coal samples, sieving was carried out through a sieve shaker by following the ASTM D5709 [5]. Sieve analysis of powdered (size less than 1mm) coal sample was carried out to evaluate the particle size division. The coal samples of 74um, 125um, and 500um sizes were selected for proximate and ultimate analysis and flotation.

2.2. Characterization

To characterize the coal samples, proximate and ultimate analysis methods were used.

2.2.1 Proximate Analysis

Proximate analysis was done as per ASTM D3174-3 and D3177 standards [1, 8, 12].

2.2.2 Ultimate analysis

The carbon, nitrogen, and sulfur contents in coal were determined as per ASTM D3176 [13] using ELTRA CHS 580 (Verder Scientific, Germany-Netherlands).

2.2.3 Calorific Value

The standard calorimeter and method were used to calculate the calorific values of the coal samples.

2.3. Flotation tests

At first, the flotation cell tank, with a capacity of approximately 1.2 liters, was cleaned with distilled water. An accurate amount (100 g, 150 g, and 200 g) of coal sample with a specific particle size (74 um, 125 um, 500 um) was added to the tank. Then, 1 litre of distilled water was added to the water tank. The flotation cell was switched on, and the speed of the impeller was set at a fixed rpm of 1000 for all tests. For conditioning, 10 minutes was provided before adding reagents and pH regulators. After complete agitation of 10 minutes, the pH of the pulp was modified and controlled by adding a solution of NaOH and HCl. Then, kerosene oil was added as a collector to enhance the coal sample's hydrophobic properties according to ASTM D2234 standards. The slurry was further agitated for 3 minutes, and then pine oil was added as a frother to stabilize the air bubbles and stirred for 2 minutes more. The air valve remained closed during agitation. The air intake valve was then opened slowly until the froth formed. The machine was stopped, and the froth was collected with the help of a scraper in a tray as a concentrate and thus dried in the furnace at 90 degrees centigrade for 5–6 hours. The tailings that remained in the liquid phase were filtered and dried in an oven. The dried samples (froth and tailings) were subjected to proximate and ultimate analysis. The effect of particle size, pulp density, and pH was studied for optimum impurities reduction and recovery.

The ash and sulfur content recovery was determined using the following equation [14].

$$\text{Recovery (\%)} = cC/fF \times 100 \quad (1)$$

where c is sulfur or ash content in concentrate, C – concentrate weight (g), f – sulfur or ash content in the feed, and F – feed weight (g).

The reduction of ash and sulfur was determined using the following equation:

$$\text{Reduction (\%)} = 100 - \text{Recovery} \quad (2)$$

3. Results and Discussion

3.1. Proximate and Ultimate Analysis

It was thought that different size fractions from coal prepared by the same sieving method as used in this study might not necessarily have the same composition, and larger particles might have associated with them more mineral matter. Results of proximate, ultimate analysis and calorific values of Dukki Coal are presented in Table 1. The coal samples with the sizes 74 µm, 125 µm, and 500 µm were analyzed.

Table 1 Results of proximate, ultimate analysis, and calorific values of Dukki coal samples

Size (µm)	Moisture (%)	Volatile matter (%)	Ash (%)	Fixed Carbon (%)	Sulfur (%)	Calorific value (MJ/kg)
74	6.32	37.62	14.0	42.08	4.53	25.665
125	6.48	36.81	15.7	40.93	4.61	25.365
500	6.55	37.20	15.9	40.31	4.65	25.402

Sample 1, which has the smallest size of 74μm has the lowest contents of moisture 6.32 %, volatile matter, 37.62 %, and ash, 14.0 % and has the highest content of fixed carbon, 42.08 and calorific value of 25.665 MJ/kg. In contrast, sample 3, which has a comparatively large size, i.e., 500 μm, contains the highest contents of moisture, volatile matter, and ash but has the lowest contents of fixed carbon and volatile matter. The variations in ash values among different size fractions are believed to be due to the different varieties of the minerals [15]. However, to increase the coal samples' efficiency and energy value, the sulfur and the ash values should be minimal. It was also observed that total volatile yields increased slightly with decreasing particle size due to the unavoidable maceral enrichment effects and different grind abilities during sample preparation [4].

3.2. Effect of Particle Size

The effect of particle size on the reduction of sulfur and ash contents from the coal samples was assessed by keeping the pH and pulp density constant and varying the particle size as 74 μm, 125 μm, and 500 μm, respectively. The effect of particle size on ash and sulfur reduction from coal with a fixed pulp density can be significant. Smaller particle sizes of coal tend to result in more complete combustion and lower ash content in the resulting combustion by-products. This is because the finer particles are more easily mixed with air or oxygen, resulting in more thorough combustion and less unburned material remaining. Smaller particles also tend to have a higher surface area-to-volume ratio, which allows for more complete combustion [4]. In terms of sulfur reduction, smaller particle sizes can also be beneficial. This is because the finer particles tend to have more surface area, allowing for more thorough coal mixing with air or oxygen [13]. This improved mixing can result in more efficient combustion, reducing sulphur compounds' formation and ultimately lowering sulfur emissions.

3.2.1 Flotation at pH 5, pulp density 10%

In the first set of experiment the pH and pulp density were kept constant at 5 and 10%, respectively, while particle size was varied accordingly as 74 μm, 125 μm, and 500 μm. The results showed that decreasing particle size will increase the reduction rate. The highest reduction of ash and sulfur content were found as 39.40 % and 33.50 %, respectively, at the particle size of 74 μm (Table 2).

Table 2 Effect of particle size on reduction of ash and sulfur at pH 5 and pulp density 10%

Parameters		Ash reduction (%)	Sulfur reduction (%)
Constant	Particle size (μm)		
pH: 5 Pulp density: 10%	74	39.40	33.50
	125	38.17	33.41
	500	23.39	14.59

The variations in ash values among different size fractions are believed to be due to the different friability of the minerals [15].

3.2.2 Flotation at pH 7, pulp density 15%

In the second set of experiments, the effect of different coal particle size (74 μm, 125 μm, and 500 μm) on fixed pH (7) and pulp density (15%) was analysed. Table 3 shows the effect of particle size on ash and sulfur reduction at pH 7 and pulp density 15%.

Table 3 Effect of particle size on reduction of ash and sulfur at pH 7 and pulp density 15%

Parameters		Ash reduction (%)	Sulfur reduction (%)
Constant	Particle size		
pH: 7 pulp density:15%	74 μm	39.25	29.24
	125 μm	35.65	25.25
	500 μm	26.73	21.42

The result observed that the decrease in the particle size will increase the reduction rate. The highest removal of ash and sulfur content was found to be 39.25 % and 29.24 %, respectively, at the particle size of 74 μm. Smaller coal particles tend to have more surface area during flotation, allowing for greater reagent absorption and increasing sulfur removal [16]. This implies that the reaction takes places at the surfaces of the particles [16]. This conclusion is in agreement with the microscopic structure of pyrite, which is generally nonporous [15].

3.2.3 Flotation at pH 9, pulp density 20%

In the third set of experiments, the effect of particle size was analysed at pH 9 and pupl density 20% while the coal particle seze was varied as74 μm, 125 μm, and 500 μm (Table 4).

Table 4 Effect of particle size on reduction of ash and sulfur at pH 9 and pulp density 20 %

Parameters		Ash reduction (%)	Sulfur reduction (%)
Constant	Particle size (μm)		
pH: 9 pulp density: 20%	74	41.73	35.82
	125	36.70	34.54
	500	21.57	19.52

The results indicate that with decrease in the particle size there is an increase in the reduction rate of sulfur and ash. The highest reduction of ash and sulfur content were found to be 41.73 % and 35.82 %, respectively, at the particle size of 74 μm. However, the highest reduction is found to be achieved at pH 9.

3.3. Effect of pH

To observe the effect of pH on the sulfur and ash reduction, further experiments were performed. In three sets of experiments, the pulp density and particle size of the coal samples were kept constant and the pH of the slurry was varied to 5, 7, and 9, respectively.

3.3.1 Flotation at size 74 µm, pulp density 10%

In the first set of experiment, effect of pH on coal flotation was calculated by keeping the particle size and pulp density constant at 74 µm and 10%, respectively. Results are shown in the Table 5. The result indicates that the increase in the pH towards alkaline side is increasing the reduction rate of ash and sulfur. The highest reduction of ash and sulfur content was 42.12 % and 38.32 %, respectively at the pH of 9; the highest removal of ash and sulfur by froth flotation at a certain pH revealed the optimum conditions for the parameters. A similar trend was observed in previous research conducted by [17], where the authors successfully reduced the sulfur and ash contents from coal samples at pH 7. This due to a change in the hydrophobic nature to the hydrophilic nature of pyrite sulfur [17].

Table 5 Effect of pH on reduction of ash and sulfur with particle size 74 µm and pulp density 10%

Parameters		Ash Reduction (%)	Sulfur reduction (%)
Constant	pH		
Size: 74 µm Pulp density: 10 %	5	39.40	33.50
	7	40.02	37.84
	9	42.12	38.32

3.3.2 Flotation at particle size 125 µm, pulp density 15%

In the second set of experiments, the pH of the slurry was varied to 5,7 and 9, while the coal particle size and the pulp density were kept constant as 125 µm and 15%, respectively. The result indicates that an increase in the pH from 5 to 9 increased the reduction rate. The highest ash and sulfur content reduction were found at 39.69 % and 28.12 %, respectively, at a pH of 9. Table 6 thus shows the effect of pH on ash and sulfur reduction.

Table 6. Effect of pH on reduction of ash and sulfur with particle size 125 µm and pulp density 15%

Parameters		Ash Reduction (%)	Sulfur Reduction (%)
Constant	pH		
Size: 125 µm Pulp density:15%	5	38.38	32.50
	7	39.15	32.50
	9	39.69	28.12

However, the reduction of ash and sulfur is lower when compared with the particle size 74µm and pulp density 10%, because, along with the increase in pH the particle size of the coal has a large impact on the reduction of ash and sulfur.

3.3.3 Flotation at size 500 µm, pulp density 20%

To further confirm the effect of pH on specific particle size, another set of experiments is performed by keeping the coal particle size 500µm and pulp density 20%. Results are shown in Table 7. The highest ash and sulfur content reduction were found to be 26.75 % and 16.61 %, respectively, at a pH of 9. Similar trend of increase in the reduction of ash and sulfur was observed with an increase in the pH of the slurry. However, the reduction is lowest when compared with the previous results. This is because the highest coal particle size gives a lower reduction in terms of sulfur and ash.

Table 7. Effect of pH on reduction of ash and sulfur with particle size 500 µm and pulp density 20%

Parameters		Ash reduction (%)	Sulfur reduction (%)
Constant	pH		
Size: 500 µm Pulp density: 20 %	5	18.23	12.92
	7	24.78	14.56
	9	26.75	16.61

3.4. Effect of Pulp Density

The effect of pulp density on the sulfur and ash reduction from coal samples is another important factor. However, this parameter has not been thoroughly investigated. In this research, the effect of pulp density was assessed by varying it to 10, 15, and 20 %, respectively, and keeping the pH and the coal particle size constant.

3.4.1 Flotation at pH 5 and particle size 74 µm

At first, the pulp density was varied by keeping the pH and the coal particle size as 5 and 74 µm. Results are listed in Table 8. The highest reduction of ash and sulfur content was found at 39.40 % and 33.50 %, respectively, at a pulp density of 10%. The results indicate a decreased slurry ratio increased the reduction rate [15]. Excessive solids would cause a competing effect for coal and collector interactions; therefore, the cleaning effect for high solids ratios would not be very effective [18].

Table 8 Effect of pulp density on reduction of ash and sulfur with particle size 74 µm and pH 5

Parameters		Ash reduction (%)	Sulfur reduction (%)
Constant	Pulp density (%)		
pH: 5 Size: 74 µm	10	39.40	33.50
	15	38.76	26.04
	20	32.35	24.97

3.4.2 Flotation at pH 7, particle size 125 μm

In the second set of experiments the pH and coal particle size were kept constant as 7 and 125 μm, respectively, while the pulp density was varied as 10%, 15% and 20%, respectively. The result observed that decreased pulp density increased the reduction rate (Table 9). The highest reduction of ash and sulfur content was found to be 40.69% and 33.22%, respectively, at a pulp density of 10%.

Table 9 Effect of pulp density on reduction of ash and sulfur with particle size 125μm and pH 7

Parameters		Ash reduction (%)	Sulfur reduction (%)
Constant	Pulp density (%)		
pH: 7 Size: 125 μm	10	40.69	33.22
	15	38.18	31.14
	20	34.03	27.79

3.4.3 Flotation at pH 9 and particle size 500 μm

Figure 10 shows the effect of pulp density on reducing ash and sulfur from coal at pH 9 and particle size 500 μm. The pulp density was varied from 10 % to 15% and 20%, respectively. The results indicate decreased ash and sulfur reduction with an increased pulp ratio. The highest removal of ash and sulfur content were 26.42 % and 20.45 %, respectively, at a pulp density of 10%. A similar kind of trend was observed in previous research [15].

Table 10 Effect of pulp density on reduction of ash and sulfur with particle size 500 μm and pH 9

Parameters		Ash reduction (%)	Sulfur reduction (%)
Constant	Pulp density (%)		
pH: 9 Size: 500 μm	10	26.42	20.45
	15	23.09	19.18
	20	17.50	14.89

4. Conclusions

The enhanced chemical method was successfully applied to reduce the ash and sulfur contents of the coal samples from Dukki, Pakistan. The following result shave been drawn out of this study.

1. The effect of major parameters such as pH of the slurry, particle size, and pulp density was studied to remove ash and sulfur associated with coal. All these three factors are important for the maximum reduction of ash and sulfur, but the particle size has a significant impact other than pH and pulp density.

2. Because of the properties of washability and hydrophobicity, large coal particles exhibit low flotation characteristics. The increase in particle size lowers the reduction rate of ash and sulfur. It has been shown that the size of 500 μm has a low ash and sulfur reduction rate.

3. Pulp density and pH adjustment were found to have the most significant impact on ash and sulfur reduction. The pH 5, 10% pulp density and a particle size of 74 μm resulted in a high sulfur and ash content reduction.

4. Hence, according to the result obtained from the experiments of froth flotation in which the parameters were adjusted as 74 um particle size, pH 9, and pulp density 10%, the highest reduction of sulfur and ash, which has been considered the best fitting conditions of parameters for the up gradation of Dukki coal. It has also been observed that sulfur and ash were removed at 42.12 % and 38.32 %, respectively, under the pre-obtained parameters. This study reveals that froth flotation reduces ash and sulfur from fine coal.

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