

Parametric Study on Desulfurization and De-ashing of Lakhra Pakistan Coal by Froth Floatation Column

Method

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Abstract: Herein, we report the desulfurization and de-ashing of lignite coal of Lakhra (Sindh, Pakistan) by froth flotation column method. The effects of various parameters during froth flotation such as a solution, feed composition, separation time, and airflow rate were determined. Poly acetal alcohol was used as wetting reagent and crecylic acid as frothing agent. The results indicated that of coal sample solution, time of separation, velocity of air and feed composition have significant influence on the desulfurization, de-ashing and efficiency of pyritic sulfur. The sulfur and ash components were reduced by 66% and 53% by controlling afford mentioned parameters.

Key words: desulfurization, de-ashing, Froth floatation, pyrite sulfur, wetting agent and frothing agent

1. Introduction

Pakistan is a coal rich country. The total coal resources of Pakistan is 185 billion tones. The average sulfur and ash content of Pakistani coal is relatively high have deterioration effect on environment and human health. Coal contains mainly carbon, sulfur, oxygen, hydrogen and nitrogen elements, simultaneously some radioactive materials [1]. Especially sulfur is one of the most toxic and corrosive chemical substance in coal resources, which is carcinogenic, corrosion causative agent and responsible for acid rain as well [2]. Ash contents has adverse effects on power plants and it costs. Increase in

ash contents directly decrease specific energy and also responsible for erosion of boilers parts and ducts resulting in increase of costs and loss of availability. The effects of 1% increase in ash content the plant cost US\$ 0.2 million per year and capital costs by 4% [3, 4].

Lignite contains low carbon and it is brownish black with high sulfur content. Lignite coal is known as low grade coal and is used for the generation of power and electricity. Its heating value is 8 to 18 million BTU per ton [5]. Many techniques (extraction, microwave energy, oxidation, heat and electrochemical treatment and froth floatation) are available in the literature for the desulfurization of coal. During heat treatment of coal, it is observed that the coal with low rank is more hydrophobic while the coal with higher rank is more hydrophilic. Desulfurization has been investigated carried out by a number of methods like emulsification

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technique at elevated temperature [6, 7], electrochemical reduction [8, 9], demineralization of coal using acid base solutions [10, 11], microwave assisted desulfurization through HI [12, 13], air oxidation process using sodium butaoxide [14], Microbial desulfurization [15, 16] , and froth flotation has also been reported [17]. In the ordinary flotation process, agitator is used to agitate the pulp in a flotation column to attain suspension of particulates. The functioning of agitator favors air in to the cell. This air spread out as tiny bubbles ^[18]. During the mechanism of flotation, the bubble comes upward to the pulp surface. Solids and water are recycled into consolidate from the froth height equilibrium. Majority of the solids are hydrophobic particulates, and these particulates de-attached from the surfaces of bubbles. In the froth mechanical entrainment was used to recycle the hydrophilic mineral particulates, which is known as slime. This slime is moved upward out of cell, in the water bubbles retained. The recovery of consolidate in enhanced via entrainment, while consolidate grading is minimized [19]. Due to this disadvantage and problem entrainment is not used for flotation of extremely small particles.

In comparison to conventional flotation, column froth flotation method is one of the best techniques to resolve this problem, column froth flotation give higher grading concentrate. Column froth flotation also give low operating and maintenance cost, high efficiency, high recovery and better control of the process [20]. Some particles stay over in liquid phase which do not stick to the bubble of air. The additional features of this technique are suitability and quite low cost of operation [21]. Characteristics of coal surface can be changed by proper wetting agent. The active surface substance makes variation in wet surface on elective mixture of coal [22]. Froth floatation is favorable physical technique for contaminations and desulfurization of chosen lignite coal among all the others described techniques. Here, we have presented for the first time, the experimental outcomes of desulfurization and

de-ashing of Lakhra coal Pakistan and the effects of various parameters like pH, separation time and velocity of air.

2. Experimental

2.1 Coal Sample Collection

The sample of coal was collected from the Lakhra coal mines Sindh, Pakistan. The quality of Lakhra coal mines is lignite-A. The coal was crushed to a size of 190 *um* in size and dried at room temperature. Sulfuric acid potassium hydroxide and potassium cyanide were used as pH regulators. The hydrophobicity of coal samples were minimized by treating coal sample with one mole of sulfuric acid and one mole of potassium cyanide at room temperature for 15 minutes. Crecylic acid was used as a frother while poly acetal alcohol was used for as a wetting agent.

2.2 Materials

Sulfuric acid (H_2SO_4), potassium cyanide (KCN), Crecylic acid or tricrecol ($CH_3C_6H_4OH$), and Poly acetal or acetal ($C_6H_{14}O_2$, POM) were purchased from Sigma Aldrich. All the chemical were used directly as received.

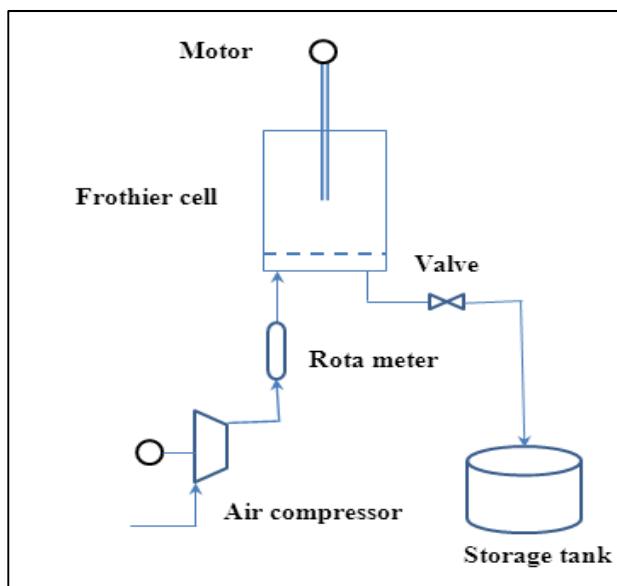
2.3 Froth Flotation Column

Lakhra Coal Pakistan has relatively high contents of Coal and Sulfur as shown in Table 1. The coal sample was added to column and continued stirring in the Poly acetal alcohol solution for 50 minutes to improve the bubbles effect followed by addition of Crecylic acid at prescribed amount rate of 4.55 ml per liter of solution. The temperature, particle, quality of samples, density of pulp and rate of feed are the fundamental and major parameters that influence the froth flotation. The Schematic diagram of froth flotation column is shown in the Fig. 1.

The coal samples were crushed to minimize hydrophobic characteristic of coal samples. One mole of potassium cyanide and one mole of sulfuric acid solution was used for the preprocessing of feed.

Table 1 Composition of Lakhra Coal Pakistan.

Moisture Heating Value (AR Btu./Lb. 5.774%)	46.77%
Heating Value(Dry)Btu./Lb 10,898	
Volatile matter	23.42%
Fixed Carbon	16.66%
Ash	6.24%
Sulphur	1.16%
Heating Value(AR Btu/Lb)	5.774%
Heating Value(Dry)(AR Btu/Lb)	10.898%

**Fig. 1 Schematic diagram of froth flotation column.**

Polyacetal and Crecyclic acid was fed into the column. The frothing was composed and collected at the top and air is supplied from the bottom of the column. The coal was collected at the basement of the column and then dried out. The calculation of efficiency of coal samples and pyrites sulfur was separated from one another and the sulfur removal percentage was calculated.

3. Results and Discussions

3.1 Effect of pH of Solution on Separation Performance

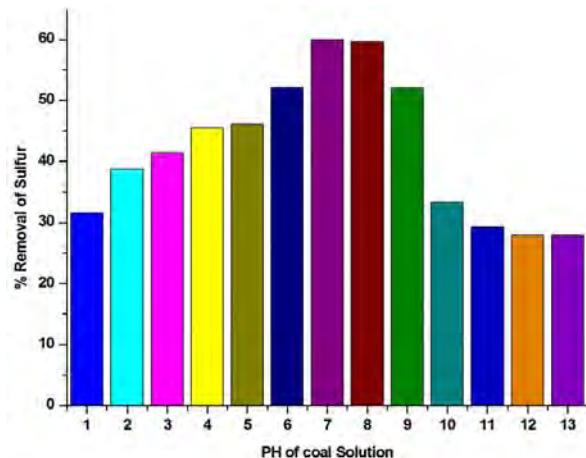
80% (wt%) of feed was used according to previous literature review [22], the time of separation 50 minutes and velocity of air is 20 cubic feet per hour. All the experiments were performed under vigorous stirring at 40 revolutions per minutes (RPM).

At acidic medium pH is equal to 1, some impurities

and contaminations were removed but it very low level. This in turn indicates that the pyrite sulfur hydrophobicity in acidic medium was active. When the pH of solution was gradually enhanced toward the neutral value of pH (pH = 7), the results was sufficiently better because the pyrite sulfur of hydrophobic nature was changed to hydrophilic nature. Coal samples surface characteristics were entirely changed, hydrophobicity increased and hydrophilicity was decreases at pH = 7. Hence, it is remarkable that the best possible level of pH is 7, where efficiency of sulfur removal becomes high, as indicated in Fig. 2 and Table 2. Similarly, with the removal of sulfur, ash removal increases and decreases, as demonstrated in the Fig. 3. Ash removal was also high at pH value of 7.

Table 2 Effect of pH on removal of contamination from coal samples.

pH	Sulfur (% in wt)	% of Sulfur removal	Wt.% of Ash	% of Removal of Ash
1	9.6	31.55	24	21.55
2	9.1	38.66	23.8	22.88
3	8.9	41.40	23.7	23.42
4	8.6	45.45	23.3	25.31
5	8.5	46.08	23	26.16
6	8	52.09	22	30.85
7	7.2	60.01	19	45
8	7.4	59.60	20.89	36.13
9	7	52.08	23	24.28
10	8.5	33.33	23.50	21.93
11	8.8	29.27	23	19.67
12	8.9	27.92	24.40	17.24
13	8.99	27.93	24.50	17.25

**Fig. 2 Effect of pH of solution on removal of sulphur.**

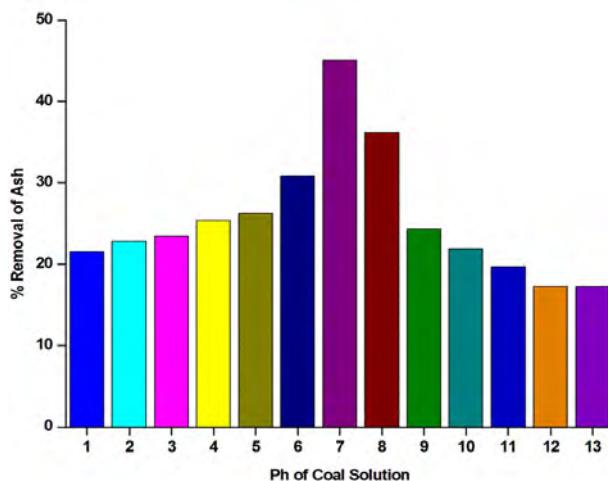


Fig. 3 Influence of pH of solution on removal of ash.

3.2 Effect of Time of Separation on Separation Performance

The consequence of time of separation time on separation efficiency was studied. The experiments were conducted for the frothing agent crecylic acid and polyacetal alcohol as wetting agent under constant operating conditions of feed composition, pH and velocity of air. The effect on the removal of sulfur of separation time, at constant velocity of air at 13 standard cubic feet per hour (SCFH) and pH = 7 is shown in Table 3.

The experimental results exhibited that the removal of sulfur and separation performance is increased due to increase in time of separation. It was observed that the pyrite sulfur separation efficiency enhances after a time interval of 4 minutes. It was also examined that the recovery of sulfur was improved, as illustrated in the Figs. 4 and 5. At the time of separation (70 minutes), the impurities removal were optimum.

3.3 Effect of Velocity of Air on Separation Performance

The effect of velocity of air on separation efficiency of sulfur pyrite was observed at constant separation time, composition of feed and pH as shown in Table 4. The experiments were carried out under constant operating parameters (pH = 7 and Time = 1 hour 20 minutes).

Table 3 Effect of time of separation on contamination removal.

Time (min)	% of Sulfur Removal	Wt. % of Ash	% of Ash Removal
50	64	23	49.01
54	64.5	22.7	49.95
58	65.0	22.6	50.42
62	65.5	22.5	50.89
66	66.0	22.3	51.82
70	67.5	22	53.20

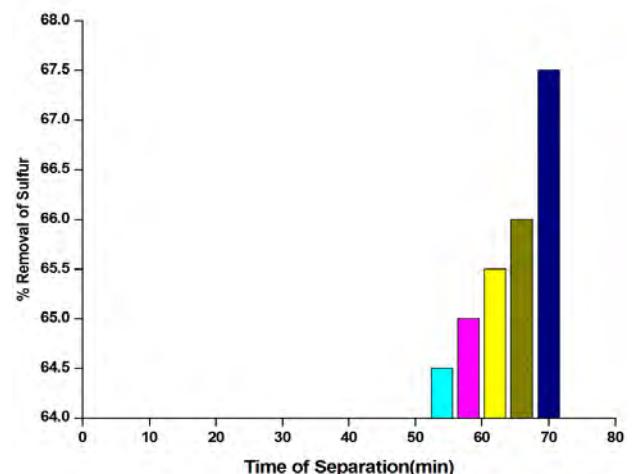


Fig. 4 Effect of time of separation on removal of sulphur.

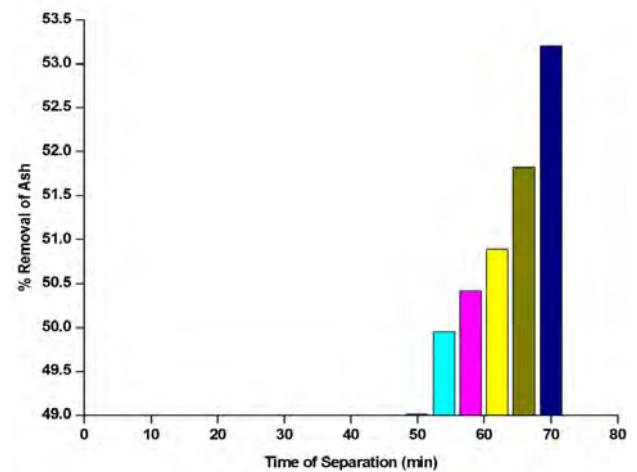
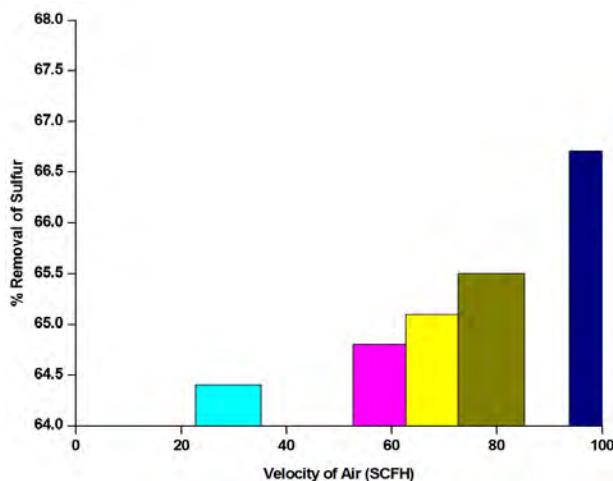
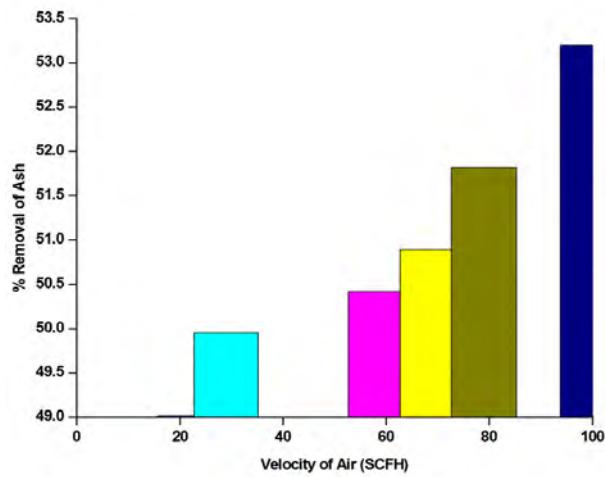


Fig. 5 Effect of time of separation on removal of ash.

The Figs. 6 and 7 indicate that the recovery of sulfur was enhanced with the enhancement of air velocity. The sulfur was recovered more across a flow rate of 100 standard cubic feet per hour. High velocity of air contribute for turbulence which cause corrosion in the

Table 4 Influence on removal of different impurities of air velocity.

Velocity of air (SCFH)	% Removal of Sulfur	% wt. of Ash	% Removal of Ash
22	64	23	49.01
29	64.40	22.66	50
59	64.80	22.61	50.51
69	65.10	22.56	50.75
79	65.50	22.54	50.84
100	66.70	22.36	51.68

**Fig. 6** Influence of velocity of air on removal of sulphur.**Fig. 7** Influence of velocity of air on removal of ash.

column, so due to which very low quantity of sulfur pyrite was capture which results in low sulfur recovery in froth column. In the same way, with the enhancement of flow rate of air removal of ash also increased.

4. Conclusion

The study was conducted on desulfurization and de-ashing of Lakhra Coal. The conclusions are as follows:

- 1) The effect of pH of a solution, feed composition, separation time, and airflow rate were investigated.
- 2) The capacity of Polyacetal alcohol a novel wetting reagent and Crecyclic acid a novel frothing agent were investigated.
- 3) We concluded that separation time, feed composition, volumetric flow rate of air and pH are the elementary and fundamental factors for the removal of pyrite sulfur and ash from the sample of coals.
- 4) The maximum contaminations removal was obtained at pH value 7.
- 5) The medium of time of separation designate that desulfurization and ash removal increases with the separation time. At constant feed composition, time of separation and PH of coal sample solution, the impact of air velocity on separation performance of pyritic sulfur was under observation. Hence, by using the above mention three conditions, such as ash and sulfur constituent were deduced to 53% and 66%.
- 6) Even though, poly acetal alcohol as wetting agent showed better results among other chemical synthetic frothing agent. Poly acetal alcohol is used to repress and control the surface properties of material from coal mixture and sulfur pyrite. This experimental work revealed that crecyclic acid is best for the removal pyrite impurities from mixture of coal. For emulsion the crecyclic acid is used as emulsifier. Separation was obtained in acidic neutral medium, which represents that the hydrophobicity of the sulfur pyrite was minimized from coal samples. Increasing separation time high removal efficiency of pyrite sulfur has been obtained.

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