

Cleaning of Thar Coal by Froth Flotation and Water Washing

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Abstract

The demand for a constant supply of energy is increasing day by day. To meet these increasing demands, natural resources must be utilized smartly. Pakistan having 185 billion tons of coal have huge potential of energy and chemical production from it. However, 175 billion tons of it, found in Thar region of Sindh province, need pre-cleaning of coal in terms of sulfur and ash removal before its effective utilization.

In this work, cleaning of Thar coal was carried out using froth flotation and water washing techniques. Three different particle sizes $(250 \ \mu m, 149 \ \mu m, and 105 \ \mu m)$ were employed to study the effect of froth flotation on ash and sulfur reduction. Experiments were carried out in laboratory flotation cell under different set of conditions (i.e., flotation with and without collector and frother and by simple water washing). Kerosene oil was used as a collector and pine oil as a frother. Characterization of raw and treated Thar coal samples was carried out by ASTM standards. The results exhibited an inverse relationship of particle size towards efficiency of the cleaning process; thus, the optimal combustible recovery was attained at 105 μ m particle size. Furthermore, FTIR results disclosed the presence of aromatic and aliphatic structure in treated coal samples. Thermo-gravimetric analysis was also performed to determine the weight loss in raw and treated samples and increased combustion efficiency and reactivity was observed in treated coal samples.

Keywords: Coal Cleaning, Froth Flotation, Collector, Frother, Thermogravimetric Analysis, Deashing, Desulfurization, FTIR Analysis

1. Introduction:

Over the past few years, Pakistan has faced major energy crisis due to depleting natural gas, inflating prices of crude oil and increasing debts [1]. Imported crude oil creates adverse effects on Pakistan's foreign exchange reserves and environment. On the other hand, Pakistan's natural gas reserves are reducing rapidly, due to their low availability and excessive use without definitive future plans [2]. Over the last 10 years, these reserves are decreasing at the average rate of 2.45% [3]. Thus, neither crude oil nor natural gas is an appropriate fuel for the continuous supply of energy.

On contrary, coal can be used as a promising fuel due to its huge availability in Pakistan. It is estimated that in Pakistan, oil and natural gas reserves will end up in the coming 60 and 40 years,

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respectively. However, coal reserves may last for 150 years, though the share of coal in energy supply is merely 6% [4]. To meet the energy demand and supply gap, coal can be an efficient, plentiful and inexpensive fuel [5] and hence it becomes inevitable to extensively explore and develop coal as a primary source for power generation. In Pakistan, the production of coal has been 7.2 million tons in 2020 [6]. The total coal reserves available in Pakistan are estimated to be 185 billion tons. About 94% of coal reserves are found in Thar region at Sindh province [7]. A small part of these reserves are being used to produce electricity but that is merely 1300 MW [8, 9].

Thar coal is lignite by rank and it has comparatively less ash and sulfur but high moisture content [9, 10]. The use of coal even with low percentages of ash and sulfur, may cause many environmental problems, such as slagging and fouling (in boilers and furnaces), acid rain, global warming, CO_2 emissions, and many more [11]. Thus, it is necessary to process it before utilization. Various physical and chemical methods have been developed for coal processing and beneficiation. Physical methods include water washing, dense media separation, froth flotation, jigging, and magnetic separation etc. Whereas chemical methods include chemical leaching, agglomeration, flocculation etc. [12].

Froth flotation is a highly effective physiochemical method for cleaning low grade fines, on the basis of difference in wettability of coal and mineral matter [13]. It mainly relies on the surface characteristics of particles (i.e. hydrophobic or hydrophilic nature). In froth flotation technique air is bubbled through the coal water suspension, so that hydrophobic coal particles getattached with air bubbles and comeout as froth. While the mineral matter being hydrophilic in nature, easily soaks and settles down as tailings [14]. A number of chemical reagents (collectors, frothers, and modifiers) are used to facilitate the separation process. The collectors are usually hydrocarbon molecules used to increase the hydrophobicity of coal particles. On the other hand, frothers are the long chain organic molecules which help to stabilize air bubbles. The shape and size of air bubbles directly influence the quality and

quantity of the froth collected during flotation [15]. Modifiers are the oxidizing and reducing agents (acids and bases) used to depress the flotation of undesired particles such as pyrite [16].

Another physical coal cleaning technique used in this study is water washing is based on the difference in densities of coal and mineral impurities and their settling rates. The specific gravity of coal ranges from 1.2 to 1.4 while that of mineral impurities varies from 2 to 2.6. The coal being lighter, floats at the surface of water while the impurities settle down. Clean coal is then filtered using some filtration device [17-19].

The purpose of this study is to explore the potential of froth flotation (with and without the collector and frother) and water washing techniques in terms of beneficiating fine sized coal samples. The impact of treatment type and particle size is evaluated on separation efficiency, combustion analysis and characteristics. A comparative evaluation of these two different cleaning methods is carried out based on heating values, biochemical content, and functional groups of raw and treated Thar coal samples.

2. Experimental:

2.1 Sample Collection And Preparation:

The sample was collected from Thar mines (block II) located in Sindh province. Afterwards, it was crushed by roll crusher and ground by laboratory mills, as described in previous literature [20]. The desired sizes (i.e., 250 µm, 149 µm, and 105 µm) were obtained by passing the ground sample through Tyler Sieve Shaker. As the decrease in particle size maximizes the use of hydrophobic/hydrophilic nature of coal particle for impurity separation in physical coal cleaning methods and the particle size of 250 µm is used for major characterization of coal as per ASTM standards; so size of 250 µm and lower (i.e 149 µm and 105 µm) were studied for efficiency of sulfur and ash removal. Laboratory grade pine oil and kerosene oil were purchased by local stores.

3.2 Pre-treatment Characterization:

The proximate analysis of representative sample (250 μ m) was determined by ASTM D-3127. Whereas GCVs of coal sample was calculated by digital bomb calorimeter (LECO AC500), as per

ASTM D-5865. Moreover, CS analyzer (model: LECOSC-144DR) was used to examine total carbon and sulfur contents by following ASTM D-5016. All of the analyses were repeated three times and their mean values are reported. Besides, FTIR analysis was done using Fourier Transform Infrared Spectrometer (model: Nicolet iS10 Mid Infrared, Thermo Fisher Scientific, Austria) to identify functional groups in coal samples. In this technique about 0.5 mg of sample was examined via FTIR and absorption graphs were obtained [21]. The graph represented different groups (e.g., O-H, N-H, C-H etc.) and labeled by comparing the results with available library.

Thermo-gravimetric analyzer (LECO-TGA-701) was used to determine the weight loss with respect to temperature in nitrogen environment. Sample was heated from 25° C to 110° C with a stay time of 10 minutes (1st Step). The temperature of the furnace was elevated from room temperature to a maximum of 900 °C (2^{sd} Step) with 10 °C per minute heating rate. The flow rate of nitrogen was kept at 3.5 L/min. The weight percent of each sample was reported at the end of each analysis step.

Table 1: Detailed characterization of raw Thar coal (250 μ m), including proximate analysis, GCV,

Test	Results			
Proximate Analysis;				
Total Moisture (%)	32.89			
VM (%)	40.20			
Ash (%)	4.96			
FC (%)	21.95			
GCV (Kcal/kg)	4750.01			
Total Carbon (%)	59.01			
Total Sulfur (%)	0.80			

total carbon and sulfur.

2.3 Froth Flotation:

2.3.1 With collector and frother

Froth flotation of three particle sizes (i.e., $250 \mu m$, $149 \mu m$, and $105 \mu m$) was carried out in a laboratory flotation cell. 50 g of coal sample and 1 L of water was taken for each run of experiment. The impeller speed was varied up to 1000 rpm. Subsequently, the coal water slurry was stirred for 5 min. After 5 min of conditioning, 2-3 drops of kerosene oil were added as collector and conditioned for further 90 s.

Afterwards, 2-3 drops of pine oil were used as frother and conditioned for 30 s with the airflow turned on forming bubbles. The froth was collected as a top layer while tailings were allowed to settle down. The froth and tailings were filtered, dried, and weighed and yield was calculated by using Eq. (1);

$$Yield\% = \frac{Wc}{WC + Wt} \times 100$$
(1)

Where,

Wc = weight of the concentrate (clean coal or froth) Wt = weight of the tailing.

2.3.2 Without collector and frother:

Experiments without collector and frother were operated on all three particle sizes (i.e., $250 \mu m$, $149 \mu m$, and $105 \mu m$), by keeping the solid liquid ratio and impeller speed same as 3.3.1. However, the coal water slurry was conditioned for 10-15 min. After conditioning time, the air flow was turned on and froth was collected in the dishes until the change in color was observed. The froth and tailings were filtered, dried, weighed and their yields were calculated as Eq. (1).

2.4 Water Washing:

Water washing of Thar coal was carried out using all three particle sizes $250 \,\mu\text{m}$, $149 \,\mu\text{m}$, and $105 \,\mu\text{m}$) in glass beakers. A suspension was made by adding 50 g of coal sample in 1 L distilled water and magnetically stirred for 24 h. Afterwards, the solution was filtered and dried till a constant weight was obtained. The dried sample was weighed and the yield was calculated by using Eq. (2);

 $Yield\% = \frac{Weight of coal obtained dafter water washing}{Weight of raw coal added} \times 100 \quad (2)$

2.5 Post-Treatment Characterization

The highest yield of concentrate is obtained at 105 μ m particle size for both treatments (i.e., water washing and froth flotation), therefore further characterization was made only for 105 μ m. Post treatment characterization include proximate analysis, carbon and sulfur determination, Thermogravimetric analysis, FTIR analysis and heating value determination (as described in section 3.2)

3. Results and Discussion:

3.1 Percentage Yield Analyses With Respect To Particle Sizes and Treatment Types

As the process of froth flotation is based on surface

chemistry, decrease in the particle size increases the surface area and hence facilitates interaction of particles to act as hydrophilic/hydrophobic. The addition of collector increases the hydrophobicity of coal and facilitates the separation of organic part of coal from mineral particles [22]. While frother stabilizes the air bubbles to carry as much coal as possible [23]. Due to the addition of these chemical reagents, the hydrophobic/hydrophilic nature of feedstock increased and resulted an improved interaction of alike particles which triggered a sharp separation of hydrophobic coal particles from hydrophilic minerals. Maximum separation is obtained in case of 105 µm particles and hence highest yield of concentrate (see Table 2). It is in agreement with the findings of Davaadorj, T., et al., according to whom ultrafine particles catch the

collector efficiently and float well [24].

Similar results were obtained without collector and frother, where the highest yield and stable froth is obtained at same particle size (i.e., 105μ m). However, the yield obtained from these experiments is less as compared to actual froth floatation process where collector and frother are employed. In the case where collector and frother are were not used and only air flow is involved, the air bubbles may have lifted the lighter particles only. While the coarser particles may have broken the air bubbles and settled down and ended up in tailings. Likewise, water washed sample also showed maximum impurity removal at 105 µm. It may be due to the attainment of mesh of liberation at particle size of 105 µm, specifically for Thar coal.

Treatment Type	Prod	ucts		
Froth Flotation (with Collector & Frother)	Concentrate (%)	Tailings (%)		
250 μm	21.00	77.87		
149 μm	34.80	64.11		
105 μm	65.60	30.40		
Froth Flotation (without Collector & Frother)	Concentrate (%)	Tailings (%)		
250 μm	15.20	82.76		
149 μm	29.87	68.88		
105 μm	41.20	57.10		
Water Washing	Yield (g)	Yield (%)		
250 μm	48.60	97.20		
149 µm	47.21	94.42		
105 μm	45.00	90.00		

Table 2: Percentage yields of products obtained at different mesh sizes

4.2 Results Of Detailed Proximate Analyses With Respect To Treatment Types:

Table 3 displays a reduction in the sulfur and ash content along with increase in VM and heating value of coal. The purpose of coal flotation is to decrease ash content to maximize combustible recovery. Thus, it is reasonable to consider both factors in evaluating the efficiency of flotation process [25]. The surface of coal is hydrophobic because it is mainly containing nonpolar hydrocarbons. However, water is polar by nature and shows a little propensity to wet nonpolar materials e.g., oil. On contrary, oils possess a strong affinity for nonpolar sides. The numerous ash minerals are strongly polar compound and water wets them easily, but oils do not. This primary difference in structure facilitates the cleaning of coal by froth flotation. Therefore, it is evident that both collector and water participate in froth flotation. In general, a reduction in sulfur is also observed and showed agreement with the results of Ehsani, M.R. and F. Eghbali [26].

The total carbon analysis shows that the mass fractions of carbon content in froth is relatively high. A fuel with high carbon content is helpful for combustion because of its increased heating value [27]. Same findings were reported in previous literature [28]. In addition, gross calorific value of raw Thar coal was only 4750 kcal/kg but it rises to 5585 kcal/kg after flotation. The reason for higher GCV after floatation could be due to higher carbon content and lower content of ash as given in Table 3. This is also consistent with the observation of Khan, M. [29]. Hence, froth flotation can be considered a significant way to accomplish Thar coal upgradation.

Similar trends appear in case of froth flotation without collector and frother and water washing of

coal. The ash and sulfur content are reduced with the increase in heating value of coal, which supports the classification and utilization of Thar coal in substituting fuel source.

Table 3: Product yield and detailed proximate analysis with respect to treatment types for 105 μm particle size. **Froth Flotation** (with Collector & Frother)

Products	Yield	Moisture	VM	Ash	FC	С	S	GCV
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(Kcal/kg)
Concentrate	65.60	8.82	76.18	4.00	11.00	64.48	0.78	5585.28
Tailings	30.40	9.80	76.47	9.80	3.93	62.39	0.76	5360.29
Froth Flotation (without Collector & Frother)								
Concentrate	41.20	12.07	69.07	4.70	14.16	57.30	0.69	5428.65
Tailings	57.10	15.64	65.32	10.65	6.39	52.98	0.97	4812.76
	Water Washing							
Clean Coal	97.20	18.58	60.42	3.33	17.67	51.29	0.61	4852.14

4.3 FTIR Analyses

FTIR analysis gives the information about the functional groups present in the coal structure. The peaks are compared with standard charts reported in previous literature [30-32]. Fig. 1 shows the FTIR spectra of raw Thar coal along with water washed sample. Raw sample reveals medium secondary amine (N-H) stretching at wavelength of 3349.82 cm⁻¹. Similarly, there is the stretching of C-H observed at region of 3000-2500 cm⁻¹, showing a medium stretching of alkane and alkene compounds. The highest peak between 1500-1000

cm⁻¹ represents strong C=C stretching, indicating á, â unsaturated ketone group. Whereas, a mild O-H (phenol group) bending and C-N (amine) stretching becomes visible at 1500-1000 cm⁻¹.

The FTIR graphs of water washed sample is almost similar to raw Thar sample but only a little variation in the stretching and bending of functional groups are observed (see Fig. 1 right side) below 1500 cm^{-1} . The zone $1500-1000 \text{ cm}^{-1}$ shows C-H bending and represent the aldehyde groups. The other peak in the same zone denotes the stretching of alkyl aryl ether group (C-O).



Figure 1: FTIR spectra of raw (left side) and water washed (right side) sample for $105 \,\mu$ m particle size.

The FTIR results of concentrate with collector and frother showed in Fig. 2 and found somewhat similar trends as raw Thar coal. For instance, the zone 3500-3000 showed a medium N-H stretching and doom-shaped peak at 3226.43 cm⁻¹ wavelength. Similarly, the 3000-2500 cm⁻¹ zone showed C-H stretching. The peak at 2000-1500cm⁻¹ illustrates the bending of N-H amine compounds. The tailings of 105 μ m size with collector and frother have shown the stretching of O-H group representing strong and broad spectra of carboxylic group centered on 3000 cm⁻¹. The peaks at 3000-2500 cm⁻¹, displayed medium stretching of C-H group. Moreover, the zone of 1500-1000 cm⁻¹ showed two different trends; one is the medium bending of C-H (alkane) group (at 1375.81 cm⁻¹) and the other is C-O stretching, representing alkyl aryl ethers.

The band at 3226.43 cm⁻¹, showed a strong and broad stretching of O-H group in concentrates (without collector and frother). The zone 3000-2500 cm⁻¹ illustrated two sharp peaks of medium stretching of C-H group. The highest peak at

1593.95 cm⁻¹, presented medium bending of N-H (amine) groups. The reduced peak of C-H bending in the zone of 1500-1000 cm⁻¹, might be due to the presence of methyl and sulfonamide (S=O) group. On the other hand, a medium stretching of secondary amine was observed at 3500-3000 cm⁻¹ in tailings (without frother and collector). Similar to concentrate, the peaks at 3000-2500 and 2000-1500 cm⁻¹ showed medium stretching of C-H (alkane) and N-H groups, respectively. However, the reduced peak in the zone 1500-1000 cm⁻¹ showed a sharp stretching of sulfonate (S=O) and C-O (alkyl aryl ether) groups



Figure 2: FTIR spectra of concentrates (left side) and tailings (right side) obtained with and without collector and frother for 105 μm particle size

4.4 Thermogravimetric Analyses

The TGA is a technique which measures the weight loss of a substance as a function of temperature. TGA curve gives the information about thermal degradation behavior of raw and treated coal samples as well as thermal stability information of intermediate products formed. While DTG (difference gravimetric ratio dw/dt) curve gives the information about rate of mass loss at any temperature [33]. Raw and treated Thar coal samples were subjected to thermo-gravimetric analysis as a function of reaction temperature at hating rate of 10 °C/min. The TGA curve gives three peaks showing onset temperature (where degradation of VM starts), peak temperature (where maximum mass is lost) and offset temperature (where decomposition of VM ends). The DTG curve gives three reaction orders first is the moisture loss, secondly de-volatilization and the small peaks represent the removal of fixed carbon or oxidation of char [34].

Fig. 3 shows the thermogravimetric analysis of raw Thar coal, where DTG peak at 110.497 °C attribute moisture removal from coal. The other peaks between 200 to 400 °C, represent the release of carbon containing volatile matter. The reduced peaks of DTG either indicate the removal of fixed carbon or the oxidation of char [35].



Figure 3: TG and DTG curves of Raw Thar coal obtained in nitrogen environment.

DTG curves of the treated Thar coal in Fig. 4 showed a removal of moisture at the temperature range of <120 °C, which correlated with increased reactivity of treated coal. Since, Thar coal has high volatile matter content, so it is easily ignited. It is observed from the results that maximum weight loss was attained between 200-400°C. It might be due to rapid volatilization along with the formation of carbonaceous residue. The peak value of tailings lies almost in the same temperature range. DTG curve of tailings with ?4% of fixed carbon, shows only one low peak which is similar to reaction temperature interval of residual carbons. It proves that TGA peaks of tailing fractions are strongly associated with residual carbon content [27].





Fig. 5 and 6 display TGA analysis of concentrate and tailings obtained without collector and frother. The DTG of concentrate showed a peak at $100 \,^{\circ}\text{C}$ which was attributed for the removal of moisture. The other peaks between 200 to 400 $^{\circ}\text{C}$ represent the release of volatile matter. The reduced peaks of

DTG after 400 °C, indicate the combustion of fixed carbon. However, the tailings showed moisture loss at > 100 °C whereas the maximum weight loss has also been observed at 478.672 °C, it may be due to higher ash content which delayed ignition. Ash may cover the carbon surfaces in coal which hinders the



Figure 5: TG and DTG curves of concentrate and tailings obtained from froth floatation without frother and collector for 105 µm particle size.

DTG curves of the water washed coal showed removal of moisture at the temperature <120 °C represent the improved reactivity of treated coal. The maximum weight loss for froth was noted to be 277 °C, whereas third peak of DTG curve correspond to combustion of residual carbon.



Figure 6: TG and DTG curves of product from froth floatation with water washing only for 105 µm particle size.

As observed in TGA results for all treated coal samples (also in Fig. 6), the removal of moisture gets completed nearly at 100 °C. As the temperature is increased from 400 °C, a sharp mass loss has been observed which correlate a rapid coal combustion as stated in Tang, L., et al. [34]. Thus, clean coal samples own high volatile matter content which may discharge rapidly during ignition and combustion. High VM lowers ignition temperature and combustion stability of coal, however it may increase the reactivity and porosity of coal.

4. Conclusion:

This study shows that froth flotation and water washing can add value to Thar coal and obtain relatively clean coal with improved combustion properties. Smaller size sample of coal results in increased hydrophobicity and higher combustible recovery and hence maximum yield was obtained at 105 μ m for all treated samples. The treatment reduced the ash content in the product coal and hence an increase in heating value was observed after treatment. A significant change was also observed in other constituents of coal including moisture, volatile matter and fixed carbon showing that the treatment process also affected the overall coal structure in all aspects.

The cleaning of Thar coal using water washing showed similar trends, however, froth flotation enhanced the separation efficiency of clean coal and showed maximum ash reduction.

The FTIR analysis confirmed presence of different aliphatic and aromatic structures in all treated samples. DTG curves of raw Thar, and treated samples show peaks shifted to low peak temperature after froth flotation and water washing. This behavior indicates that beneficiation improves coal reactivity.

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