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## Research

**Keywords:** Achibo-Sombo, Beneficiation, Coal, Physiochemical characterization, Froth floatation

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## **Beneficiation and Physiochemical Characterization of Coal Deposits in Achibo-Sombo Area, South-Western Ethiopia**

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### **Abstract**

This study was conducted to upgrade the quality of Achibo-Sombo coal deposits by physiochemical beneficiation methods particularly by using froth floatation technique to minimize the ash and sulfur content from coal particles. During the floatation test, proximate, ultimate, calorific value, functional groups, surface morphology and crystal structure analysis of Achibo-Sombo coal was carried out through different methods. The moisture content of a raw sample and treated sample is found in the range of 11.81 to 20.27% and 8.12 to 14.02 % respectively. The volatile matter is ranging from 22.74 to 34.85 % for raw coal and 21.92 to 30.64 % for treated coal samples. The ash content of the raw coal sample is ranging from 22.47 to 36.58 % which became in the range 7.49 to 13.62 % after treatment. The fixed carbon is also changed from 23.85 -38.31% of raw sample to 44.47 - 55.87 % for treated coal samples. The calorific value of a raw sample is found in the ranges of 5838.46 Btu/lb. to 9531.29 Btu/lb. whereas for treated coal samples it is found in the ranges of 9438.12 Btu/lb. to 11756.63 Btu/lb. The sulfur and nitrogen contents of the raw coal sample which were ranged from 0.57 to 1.9 % - 1.22 to 1.44 % are reduced to 0.25 to 0.41% -0.52 to 0.92% respectively after treatment. Generally, the Achibo-Sombo raw coal is falling under the coal grade of lignite B to sub-bituminous B whereas the treated coal sample is grouped under sub-bituminous C to high volatile bituminous C. Accordingly, the results obtained indicate that the Achibo coal streams are upgraded relatively with better calorific value and fixed carbon and with lowest ash contents as compared to samples collected from other sites.

**Keywords:** Achibo-Sombo, Beneficiation, Coal, Physiochemical characterization, Froth floatation

### **1. Introduction**

Coal is an organic fossil fuel and sedimentary rock that contains carbon, oxygen, hydrogen, sulfur, nitrogen, and trace elements as a mineral matter in pore systems of the coal (Wakuma and Assaba 2017; Rao and Anuradha 2019). Coal is formed through the coalification process in which plant debris were transformed from the peat to lignite and then through the higher ranks of coal to anthracite (Speight 2015). According to American society testing and materials (ASTM D-388) standards, coal is classified into four types as lignite, bituminous, sub-bituminous and

anthracite (Ruiz et al.2019). Coal is used as an energy source in industry like cement and metallurgical factories due to its higher energy density than other energy sources (Abraham 2017; Liu et al.2018). Cement industries use coal as an energy source to burn the raw materials with high temperature (1450 °C) in a kiln and metallurgical (steel or iron ) industries are also used coal for the smelting of iron with high temperature (Abraham 2017; Demoze 2016).

It is estimated that nearly two-thirds of energy supplies from coal resources comes from China. (Wakuma and Assaba 2017; Liu et al.2018; Ikwuangu et al.2017). The largest top five coal-producing countries are USA, China, Australia, India and South Africa. Countries like Australia, Indonesia, Russia, South Africa and China use coal exporting to get foreign exchanges (Demoze 2016). Commercially coal is mined in more than 50 countries and about 70 countries are using coal. The world Annual consumption of coal is about 5,800 million tons per year, out of this about 75 % is used for the generation of energy and the rest 25 % is used for the manufacturing of different industrial products (Abraham 2017; Mishra et al.2015).

The availability of coal resources in Ethiopia is estimated to be around 300 million tons. It is widely distributed in the country particularly in Northern (Chilga basin), South-Western (Delbi-Moye, Yayo (Wittete, Achibo-Sombo, Dabaso & kumbabe), Nejo and Arjo basin) and Central Plateau of Ethiopia (Mush valley Basins, Wuchale) (Abraham 2017; Ahmed 2008; Haftu et al. 2019). The Ethiopian coal is characterized by high impurity which does not fulfill the requirements for desired applications. It has high amounts of ash and sulfur which causes environmental pollution, and very low carbon content and calorific values (Demoze 2016; Dejene 2004).

The major environmental impact of coal combustion is related to the emissions of particulate gases into the atmosphere. During combustion, the elements in coal are converted to their particular oxides in the form of gaseous or aerosols (liquid droplets) and adsorbed by particulates (fly ash) and continued through flue gas stream as emissions. The emitted gases include sulfur oxides (SO<sub>x</sub>), water vapor, carbon dioxide (CO<sub>2</sub>), nitrogen oxides (NO<sub>x</sub>), and compounds and elements that are identified as hazardous air pollutants (HAPs) such as mercury and arsenic leading to the major challenges of coal utilization (Ruiz et al.2019; Singh 2016).

Therefore, to solve these problems different researchers have tried to develop beneficiation technology to minimize the impurity of coal so that it fits with its application requirements (Mishra et al 2015; Liu et al.2018; Behera et al.2018). Coal beneficiation is the process of improving the carbon number to increase the calorific value of the coal by removing the inert material (mineral matters). The main purpose of beneficiation of low grade coal is, to reduce the inert particle of mineral matter, to improve the combustion behavior, for reducing slagging and fouling characteristics, to minimize the capital of operating condition, to reduce the particulate pollutants and gas emission, (Behera et al.2018; Meshram et al.2015).

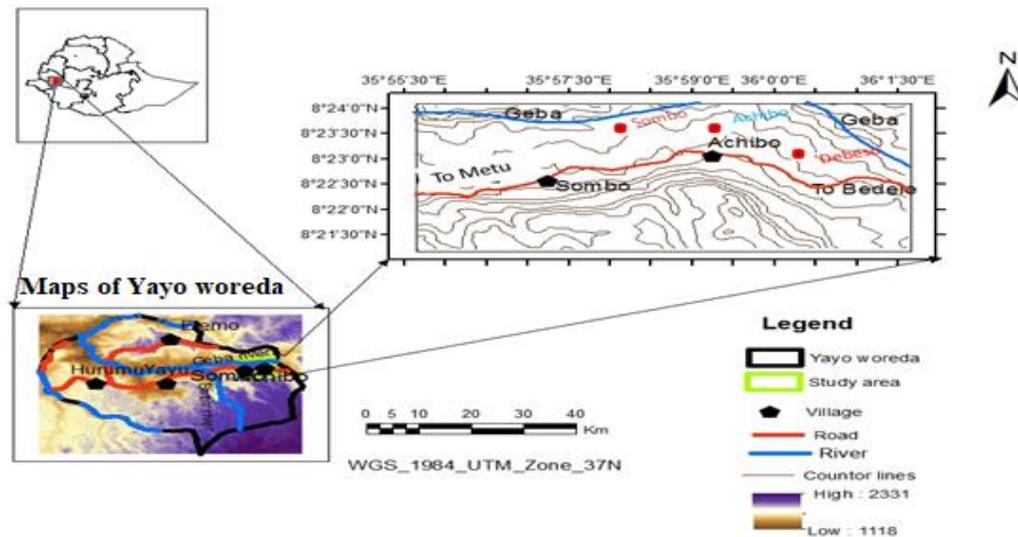
The techniques used for upgrading or beneficiation of low-rank coals are physical, chemical and physicochemical beneficiations (Behera et al.2018; Qu et al.2015; Barma 2019). Physical and chemical beneficiation techniques are more selective for minerals separation and they not economical in terms of cost. These techniques are less efficient and use more expensive reagents that need to be purified before discharge (Liu et al.2018; Chen et al.2018). Physiochemical beneficiation is the most cost-effective and less expensive separation of finely dispersed minerals to

remove the inert materials based on the hydrophobicity of coal particles and hydrophilicity of mineral matters. Among the Physiochemical techniques, Froth floatation is highly economical and efficient with basic principles of surface wettability of fine coal and separation efficiency is based on the attachment of pure coal on the surface of hydrophobic oil reagents and detachment of impurities matters from the surfaces (Barma 2019; Ni et al. 2018; Wen et al.2017; Tia et al.2017). In this study upgrading of local coal (coal collected from Achibo-Sombo) through froth floatation technique was done using diesel oil as collectors and n-octanol as frother to prepare the coal for the desired application after upgrading.

## 2. Experimental part

### 2.1. Description of study area

This study was conducted at Yayo Woreda, specifically in Achibo-Sombo villages. It is located 564 km west of the capital city Addis Ababa on the road to Gambela in Illubabor zone, Oromia Regional States, South Western Ethiopia (Fig. 1). It lies in between 25 km eastern side of Administrative towns (Mattu) and near the village (Chora distinct) 18 km on the western side.



**Fig. 1** Location of the study area.

The targeted area is bounded between the geographic coordinates of 8°21'30" N - 8°24'0" N and 35°55'30" E - 36°1'30" E latitude and longitude respectively covering an area of 9.11 km<sup>2</sup> with elevation of 1118-2331 m above sea level within Geba river streams.

#### 2.1.1. Climate and Vegetation

The climate condition of the study area is characterized by humid and warm tropical rainy climate with a mean minimum temperature of 13.5°C and maximum temperatures of 27.3°C. The rainfall pattern of the area is with a mean annual rainfall ranging from 1243 to 3445 mm. Therefore, such a diverse mixed climatic condition is the most important factor for the availability of a huge wealth of biologically diverse species in the area. The soil condition of the area is fertile or compatible of soil types that covered high diversity and tropical forests (natural vegetation) which are recorded on international level (UNESCO) as named by Yayo forest (IEA 2019; Wagaw 2007)

### 2.1.2. Geology of the area

The coal deposit in the area is mined with surface mining (strip mining) systems that was more used to produce coal recoveries with a higher proportions and high efficiency. In strip mining the giant machines remove the top-soil and layers of rock to expose large beds of coal in **Fig 2**. Once the coal is mined the rocks and dirt things are returned into the area for replanting to be used as a cropland and, habitats of wildlife and recreation



**Fig. 2** The sampling area a) exploration site b) surface mining of coal deposits c) mined coal

### 2.2. Materials and reagents:

The materials that are used to conduct this study are: Muffle furnace, Jaw crusher, Centrifugal mill, Sieve Shaker, Adiabatic Bomb calorimeter, Wedag flotation, Flash CHNS/O analyzer and Kjeldahl Digester. The chemical reagents used were Diesel oil, N-Octanol (99%), Sodium hydroxide (98%), Sulfuric acid (98%), Boric acid (99.5%), Hydrochloric acid (37%), Kjeldahl tablets (0.15gm of  $\text{CuSO}_4$  & 1.5gm of  $\text{K}_2\text{SO}_4$ ), Magnesium oxide, Sodium carbonate anhydrous, Tashoro's indicators (MR& BCG).

### 2.3. Flotation procedures of coal sample

The optimized size of 125-63 $\mu\text{m}$  and Wedag flotation cell of 3L was used to conduct this experiments. 90 g of the fine sample was mixed with 2.5 L of tap water and stirred for 3 minutes with the adjusted impeller speed of 2800 rpm until the coal particles were completely wetted by closing the air valve with scotch. Then 5ml rops of diesel oil were added as a collector and then agitated for again 2 minutes and 40 ppm of N-octanol was added as a frother and an air valve was opened to supply the air in the floatation cell condition to form the bubble particle. When bubble formation was started 0.5 L of water was added to fill the capacity of a cell. The formed bubble particle was started to overflow the cell and the flow particles were collected. The collected and formed foam is continued until the white foam was observed which indicates that all the solid particle in the cell are attached to the oil collectors. Finally, the collected particle was filtered and dried in a hot oven at 80°C and floatation concentrate yield is calculated according to Equation 1.

$$\text{Yield} = \frac{M_c}{M_f} \times 100\% \dots \dots \dots (1)$$

## 2.4. Moisture content determination

The determination of the total moisture was according to the standards of ASTM D-3302 where 2g of the air-dried fine coal sample was taken in silica crucible and gently heated in a hot oven with an experimental temperature of 107 °C for three hours. The heated sample was then taken from the hot oven and put in a desiccator to cool the sample, then the cooled sample was reweighed and moisture content was calculated by using the formula in Equation 2.

$$\text{Moisture content (\%)} = \frac{\text{loss in weight}}{\text{weight of sample taken}} \times 100\% \dots \dots \dots (2)$$

## 2.5. Volatile matter determination

The percentage of the volatile matter was determined according to the testing method of ASTM D-3175 where 1g of an air-dried sample was weighed in a silica crucible which is covered with a lid to avoid oxidation of the sample. Then the sample was heated at a temperature of 950±5°C in a muffle furnace exactly for seven minutes in the absence of air. After heating and putting in desiccator for cooling and the sample was re-weighed again and the volatile matter was determined by using Equation 3.

$$\text{Volatile matter (\%)} = \frac{\text{loss in weight}}{\text{weight of sample taken}} \times 100 \% - \text{moisture\%} \dots \dots \dots (3)$$

## 2.6. Ash content determination

The amount of ash was determined based on the standard procedure of ASTM D-3174. 1g of the sample was weighed in silica crucible and heated in a muffle furnace with a temperature of 850±5°C without lid covers for 3 hours. After burning the residue ash was cooled in a desiccator and re-weighed again and ash content was calculated by using Equation 4.

$$\text{Ash content (\%)} = \frac{\text{weight of ash residue}}{\text{weight of dried coal sample}} \times 100 \% \dots \dots \dots (4)$$

## 2.7. Fixed carbon determination

The percentage of fixed carbon of coal sample was determined according to ASTM D-388 standards. Then fixed carbon was calculated by subtracting the sum of moisture matter, volatile matter and ash content from 100 by using Equation 5.

$$\text{Fixed carbon content (\%)} = 100 - (\% \text{ moisture} + \% \text{ volatile} + \% \text{ ash}) \dots \dots \dots (5)$$

## 2.8. Ultimate analysis

The Ultimate analysis of the coal sample was determined by the using EA 1112 flash CHNS/O analyzer in Addis Ababa University, Department of Chemistry. The working conditions of an analyzer with carrier gas flow rate of 120ml/min, reference flow rate 100ml/min, oxygen flow rate 250 ml/min, furnace temperature of 900°C, oven temperature of 75°C and oxygen injection time was for 5 seconds. For the determination of CHNS, the Flash EA smart Analyzer was operated with the dynamic flash combustion of the powder sample. 3 mg of powder samples

were weighed in a tin container that was introduced into the combustion reactor through the Thermo Scientific MAS plus Auto-sampler in the presence of oxygen. After combustion, the resulting gases were conveyed by a helium flow to a layer filled with copper, then swept through a column that provides the separation of the combustion gases. Finally Thermal Conductivity Detector (TCD) was used for detecting the results.

### 2.9. Determination of nitrogen content by Kjeldahl method

2.00 g. of prepared coal sample was weighed and put into Kjeldahl digestion flask with 20 ml of sulfuric acid with two Kjeldahl tablets as a catalyst. Then the flask was slightly heated in an inclined position adjusted at 390 °C and allowed to boil for 6 hrs. During the digestion, the NH<sub>4</sub><sup>+</sup> ion ((NH<sub>4</sub>)<sub>2</sub> SO<sub>4</sub>) was formed and the mixture was diluted with 100 ml of distilled water for cooling. To neutralize the solution and convert the NH<sub>4</sub><sup>+</sup> ion into an ammonia gas excess of 50ml (50 % (w/v)) of sodium hydroxide was added. Then the flask was connected to the Kjeldahl distillation apparatus followed by boiling and condensation of the gas with 70ml (4 % (w/v)) of boric acid was added that followed by 4-7 drops of Tashoro’s (MR and BCG) indicator in the receiving flasks for 5 min. Finally, the distillate was titrated against 0.1 M hydrochloric acid. The same procedure was repeated for the blank solution and the percentage nitrogen (% N) was calculated using the formula in Equation 6 (Ryemshak et al.2016).

$$\text{Nitrogen in (\%)} = \frac{V2(\text{ ml}) - V1(\text{ml}) \times 0.1\text{M} \times \frac{14.007\text{g}}{\text{mol}}}{\text{Weight of sample taken (g)}} \times 100 \dots \dots \dots (6)$$

### 2.10. Determination of sulphur with Eschka methods

1g of coal samples was weighed in a porcelain crucible and mixed with 3g of Eschka mixtures (2g of magnesium oxide and 1g of anhydrous sodium carbonate). The mixture was covered with another 1g of Eschka mixture that uses for preventing the loss of sulfur as sulfur dioxide. Then the crucible was put in a cold muffle furnace and heated gradually at 800°C for 60 minute. The compounds of Sulphur that evolved during combustion were reacted with Eschka mixture under oxidized conditions; both were converted to Sodium Sulphate and Magnesium Sulphate. Then the product was digested by dilute 1M of HCl solution with intermittent stirring for 45 min. The digested solution was filtered into 400 ml beakers and three drops of methyl oranges were added until the color was turned to neutral. Next, the sample was heated to boiling and 10ml of 10% BaCl<sub>2</sub> was gradually added while stirring in continuous boiling for 30 minute. Finally, the solution was filtered after cooling down by thoroughly washing with hot water and the residue BaSO<sub>4</sub> formed was burned at 500°C and weighed (Speight 2015; Samila et al.2020; Adekunle et al.2015). The formed BaSO<sub>4</sub> was used to calculate the sulfur content in coal samples by using Equation 7.

$$\text{Sulfur (\%)} = \frac{32}{233} \times \frac{\text{Weight of BaSO}_4 \text{ formed (g)}}{\text{Weight of sample taken (g)}} \times 100 \dots \dots \dots (7)$$

### 2.11. Oxygen content

The percentage of oxygen in the given sample was determined by differentiating another ultimate analysis from 100 and oxygen content was calculated as Equation 8.

$$\text{Oxygen \%} = 100 - (C\% + H\% + N\% + S\% + A\% + M\% \dots \dots \dots (8)$$

### 2.12. Determination of calorific value

Approximately 1g of powder coal sample was weighed in nickel crucible and placed in a bomb by supporting over the ring. Then Oxygen supply was purged across the valve into the bomb until the pressure was reached 25-30 atm for completing the combustion. Then the bomb was placed in the nickel bucket of calorimeter that was filled with 2000 ml of distilled water. After the power was switched on the initial temperature was recorded and when bucket temperature was equilibrated with the bomb temperature the bomb was fired. Then the maximum temperature was recorded from the reader and the bomb was removed from the bucket and the knob valve was open gradually to release the residual gas pressure before attempting to remove the cap. All unburned pieces of fuse wire from the bomb electrode were removed, straightened and measured their combined length in cm to know the net amount of wire burned. Finally, the bomb washing was titrated with a standard sodium carbonate solution using a methyl orange indicator. Then the gross heat combustion was calculated as Equation 9.

$$GCV = \frac{(tW - e_1 - e_2 - e_3)}{m} \dots \dots \dots (9)$$

GCV= Gross calorific value in Btu/lb.

t = the temperature change

W = energy equivalent of calorimeter in cal/°C

e<sub>1</sub>= correction in calories for the heat of formation of nitric acid (HNO<sub>3</sub>)

= (ml of standard alkali solution used in acid titration) if 0.0709N alkali was used for the acid titration

e<sub>2</sub> = correction in calories for the heat of formation of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)

e<sub>3</sub>= correction in calories for the heat of combustion of fuse wire

m = mass of coal sample taken in gm

### 2.13. Scanning electron microscopy (SEM)

Scanning electron microscopy analysis using a field emission microscope model (Inspect F50) was used to determine the nature of surface morphology coal samples.. Small amounts of powder sample were put on the sample holder covered with a conductive carbon tape. The operating parameters of SEM were adjusted with a voltage of 15.00 kV, a Working distance of 11.4 mm and different magnification power.

### 2.14. Fourier transform Infrared spectroscopy (FTIR)

The constituent surface functional groups of an original raw and treated coal sample were separately determined by using FT-IR (Perkin Elmer spectrum, USA) with the wavenumber ranges of 4000-400cm<sup>-1</sup>. The final results were collected and reported by transmittance versus wavenumbers (Liu et al.2018).

### 2.15. X-ray diffraction (XRD)

The crystalline structures and mineral matters were identified in both raw and floated samples by using (XRD, SHIMADZU-7000, Japan) equipped with Cu K α radiation (λ = 1.5406 Å) and 2θ ranges from 2 to 70° with continuous scanning. The operating parameters are voltage was at 40 kV, the current was 30 mA and speed of scanning rate was 3.0 deg/min (Qu et al.2015).

### 3. Results and discussion

#### 3.1. Optimized parameters

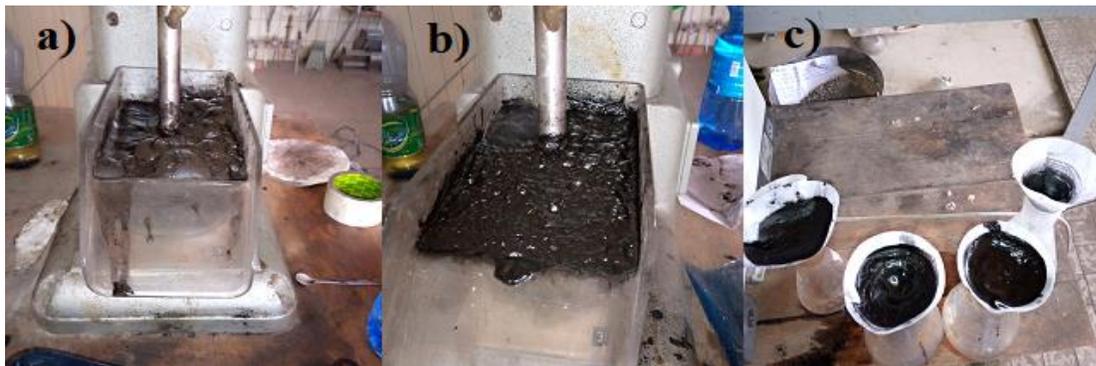
The parameters used during floatation process are to be optimized like particle size, feed rate, dosages of reagents (diesel oil and n-octanol), pulp density, and impeller speed. The optimization of these working parameters in this study was done based on the principles of one variable at a time (OVAT). In this study, the maximum mineral removal efficiency was quantified as the response, together with feed rate, a dosage of collectors and frother, impeller speed and particle size were provided as the main factors. At this optimized condition, the maximum yield of floated coal obtained were 77.5% of Achibo, 73.5% of Sombo and 67.9% of Dabaso (**Table 1**). The recovery coal yield obtained from this experiment was comparable with the result obtained after optimization parameters at impeller speed of 2000 rpm, diesel oil(10 kg/t), MIBC (500g/t) and particle size at 125-74  $\mu\text{m}$  with ash contents of treat coal particle was 8.27% and the yields of coal recovered was 80.7% (Chen et al.2018)

**Table 1:** Optimum values for floatation of the coal

Parameters	An optimized value of the conditions
Feed rate in (g)	90
Pulp density (gm./L)	30
Impeller speed in (rpm)	2800
Particle size in ( $\mu\text{m}$ )	125-63
Collector dosage(diesel oil) in (ml)	5
Frother dosage (n-octanol) in (ppm)	40
Yield of concentrate (%)	77.5% of Achibo, 73.5% of Sombo and 67.9% of Dabaso

#### 3.2. Floatation results

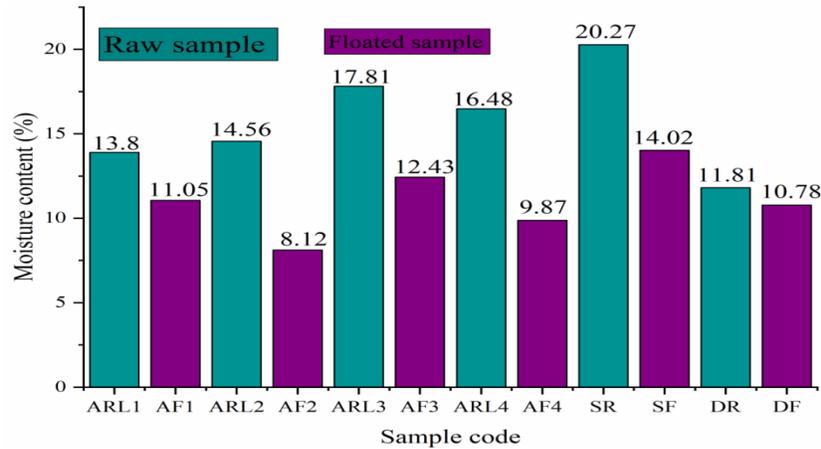
As shown in **Figure 3 a**, the floatation of the coal particle was seen after the addition of collector (diesel oil) at the conditioning time of ( $t=1\text{min.}$ ). The formation of big bubbles was seen and particles were not attached to the surface of the oily bubbles.). But after the addition of n-octanol as frother and increasing the conditioning time to 5 minutes, the bubble was stabilized and small in size as seen in **Figure 3 b**. The formed foam particles were skimmed off and filtered by using Whatman filter paper to separate the residue from its solution as shown in **Figure 3 c**. The filtrate of both tailing (residue) and concentrate (foam) was dried and taken for characterization.



**Fig.3** Froth floatation procedures and resulting coal particles

### 3.3. Inherent moisture content

The obtained results of moisture content for raw coal were ranged from 11.81 % to 20.27% and the moisture content of (treated) floated coal ranges from 8.12 % to 14.02 % with a total average value of 11% as shown in **Fig. 4**. The Sombo streams have the highest amounts of moisture content but Dabaso streams have the smallest moisture contents as compared with other remaining streams. Therefore the highest value of moisture implies that lower carbon contents that lead to lower heating value and higher costs for transportation and storage (Wakuma and Assaba 2017).



**Fig. 4** Moisture contents vs. Sample code

### 3.4. Volatile matter

The experimental result obtained from volatile matter for raw coal samples that were collected from three sampling sites (Achibo, Sombo and Dabaso) was ranging from 22.74 % - 34.85%. But the volatile matter for a treated sample was below 30.64 %. The Achibo stream has the highest volatile matter with both raw and floated samples as described in **Table 2**. Accordingly, as reported by (Habtezgi 2001) results indicated that the obtained results of volatile matter are ranged from 15.4% to 45% and the results have shown that the values of volatile matter lie in ranges of 22.3 % to 38.1%. The higher amount of the volatile matter in coal specifies that the lower rank of the coal and increasing volatile matter also increased the quantities of combustible gases, which directly decreases the amount of fixed carbon and consequently decreases the heating value of the coal. Finally, in this study, the floatation technique is used for treatment of coal to reduce the volatility of the coal and to increase the staying time at storage rooms.

**Table 2** Amount of volatile matter obtained from experimental results

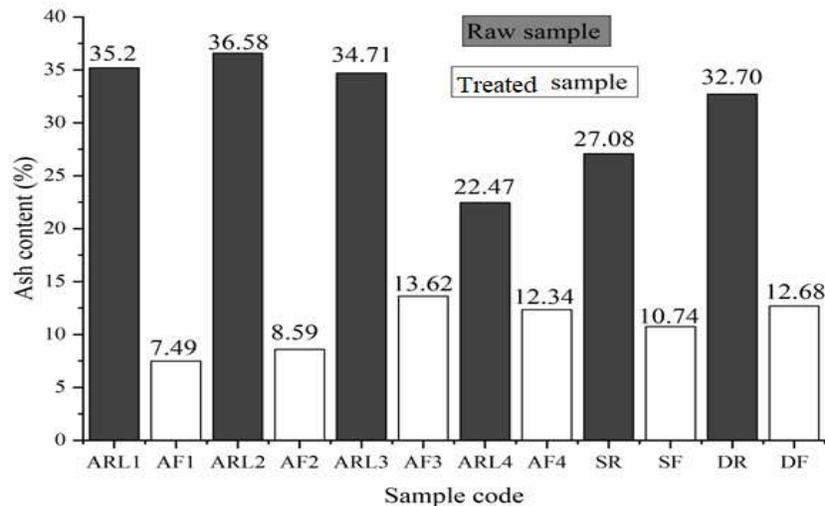
Sample Code	Volatile matter of sample (%)	
	Untreated( raw) sample	Treated (floated) sample
ARL1	34.85	30.64
ARL2	32.57	28.93
ARL3	33.61	29.48
ARL4	22.74	21.92

SR	28.44	25.33
DR	30.84	24.74

### 3.5. Ash content

Ash is an impurity substance that will not be burned. The ash content that was obtained from the experiment on raw coal is ranging from 22.47 % to 36.58 % as stated in **Fig. 5**. The Achibo second layer has the highest value of ash content from the three sampling sites which was 36.58% and the lower ash content was recorded on Achibo fourth layer that was 22.47%. The consequence of increasing ash content led to reducing the burning capacity, affects combustion efficiency, increases handling cost, and causes large slagging. The amount of ash content and its behavior at high temperatures affect the design and type of ash handling system that is employed in the coal-utilization process. At high-temperature coal ash become sticky and eventually forms molten slag. The outcomes of increasing the amounts of ash content in coal lead to decreased quality or rank because volumes of high impurity reduce the coal efficiency. According to Ahmed (1998) the ash content was ranged from 10.2 % to 54.6% and the majority of the sample was attaining less than 38%.

As shown in **Fig. 2** the ash content of the treated coal sample was reduced with ranges of 7.49% to 13.62%. This confirms that the ash content of the Achibo treated coal sample is much more reduced after treatment when compared with others that possessed relatively high fixed carbon and calorific value.



**Fig.5** Ash contents for raw and floated coal sample

The ash content was related to moisture and volatile matter that was shown in **Table 3**, Achibo streams have high amounts of ash and volatile matters but lower in moisture content. But Sombo and Dabaso streams have moderate amounts of ash and volatile matter. Therefore this all proximate parameter was reduced in treated coal and the ash content of three study areas was highly reduced that can be ignited easily and extremely reactive in combustion. Generally, the relationship of proximate value and the increasing amount of moisture content is leading to increasing the ash and volatile matters. Therefore the presence of moisture content was decreased relatively with the volatility of the coal increases, and moisture increases with volatility decreases.

**Table 3:** The relation between Moisture, Volatile matter and Ash content

Sample code	Ash content (%)		Moisture content (%)		Volatile matter (%)	
	Raw	Treated	Raw	Treated	Raw	Treated
ARL1	35.2	7.49	13.8	11.05	34.85	30.64
ARL2	36.58	8.59	14.56	8.12	32.57	28.93
ARL3	34.71	13.62	17.81	12.43	33.61	29.48
ARL4	22.47	12.34	16.48	9.87	22.74	21.92
SR	27.08	10.74	20.27	14.02	28.44	25.33
DR	32.70	12.68	11.81	10.78	30.84	24.74

### 3.6. Fixed carbon content

The Fixed carbon tells a rough estimation of the heating value of the coal. The amount of fixed carbon content for raw coal that was obtained from the experimental result was found in the ranges of 23.15% to 38.31% as described in **Table 4**. All raw coal samples were showing less than 40% of the fixed carbon content which implies that lowering the carbon number highly affected the heating efficiency of the coal. From the studied area Achibo fourth layer was the highest amounts of fixed carbon as compared to others, but the first layers of Achibo and Sombo streams have lower amounts of fixed carbon. The lowering amount of fixed carbon was due to the coal was highly affected by ash, volatile matter, and moisture content that presented in it.

The fixed carbon for the treated coal sample was obtained after beneficiation experiments that were in the ranges of 44.47 % to 55.87 % as shown in **Table 4**. From three studying areas, lower layers of Achibo has shown more carbon content and Achibo third layers have low carbon than all other areas. The upgrading of the fixed carbon in lower layers was due to the depth of the burial increases with increasing the ranks of coal and decreasing the inorganic matter, which leads to increasing the carbon number of that coal. Due to the highest fixed carbon observed in Achibo streams, a lead to the calorific value was also higher than the other sites. Therefore, increasing the fixed carbon increase of heating value and decrease the ash contents. Finally from the observed experimental value of Achibo-Sombo coal, the value of the fixed carbon content for a raw sample was majorly in the ranges 23.85 % to 38.31%, but after treatment, the ranges of fixed carbon contents raised to 55.87%.

**Table 4** The amounts of fixed carbon content for raw and floated coal sample

Sample code	Moisture content (%)		Volatile matter (%)		Ash content (%)		Fixed carbon (%)	
	Raw	Treated	Raw	Treated	Raw	Treated	Raw	Treated
ARL1	13.8	11.05	34.85	30.64	35.2	7.49	23.85	50.82
ARL2	14.56	8.12	32.57	28.93	36.58	8.59	24.29	54.36
ARL3	17.81	12.43	33.61	29.48	34.71	13.62	33.81	44.47
ARL4	16.48	9.87	22.74	21.92	22.47	12.34	38.31	55.87
SR	20.27	14.02	28.44	25.33	27.08	10.74	34.21	52.81
DR	11.87	10.78	30.84	24.23	32.7	12.68	24.89	49.91

### 3.7. Carbon and Hydrogen content

The carbon content of the study area for the sample is ranging from 38.90 % to 44.54 % and for the treated coal sample is ranging from 44.12 % to 61.06% as shown in **Table 5**. The ultimate carbon is differing from proximate fixed carbon, during the treatment of coal under high temperature some carbon contents are lost as volatile in the case of fixed carbon. Therefore lowering ultimate carbon in coal indicated that the coal was categorized under the low grade.

**Table 5** Carbon and Hydrogen content of the coal

Sample code	Carbon content n (%)		Hydrogen content (%)	
	Untreated (raw)	Treated (floated)	Untreated (raw)	Treated (floated)
ARL4	44.54	61.06	5.28	2.98
SR	39.90	44.12	4.85	3.26

The hydrogen content in the study area for the raw coal sample is ranging from 4.85 % to 5.28 % and treated coal hydrogen content was reduced to ranges from 2.98 % to 3.26 %. The higher amount of hydrogen content affects the combustion behavior of the coal and is the characteristic of low-grade coal. But the low value of hydrogen in coal has shown that the coal showed the characteristic of higher grades.

### 3.8. Nitrogen content

The obtained nitrogen content from the experimental value was in the range of 1.22 % to 1.44% for the raw coal sample and after treatment the nitrogen content was reduced and ranged from 0.52 % to 0.92% as described in **Table 6**. Achibo first layers sample showed the highest amount of nitrogen contents, it was closer to organic materials of decomposed plants. The presence of nitrogen in coal samples will pollute the environment by releasing nitrogen oxides into the atmosphere in certain combustion conditions. It also, decreases the heating values of the coal and as the grade of the coal increases (matures) the extractable nitrogen content was decreased. Low-grade coal has high amounts of nitrogen content (Ryemshaket et al.2015).

**Table 6** The Nitrogen content of the coal

Sample code	Nitrogen content (%)	
	Untreated (raw)	Treated (floated)
ARL1	1.44	0.69
ARL2	1.35	0.92
ARL3	1.22	0.83
ARL4	1.28	0.79
SR	1.33	0.64
DR	1.31	0.52

### 3.9. Sulphur content

The investigated results of Sulphur content in raw coal samples are ranging between 0.57 % to 1.9 % and after treated the sulfur content is reduced to the ranges 0.25 % to 0.92% as described in **Table 7**. The first layers of the Achibo site contain the highest amounts of sulfur content than other sites, but the third layers of these sites contain the lowest amounts of sulfur. The high amount of sulfur in coal was lowering the heating ability of the combusted coal due to the formation of high ash of sulfur contents in the forms of barite ( $\text{BaSO}_4$ ), galena ( $\text{PbS}$ ) and gypsum ( $\text{CaSO}_4 \cdot 5\text{H}_2\text{O}$ ). So the higher sulfur led to causes emission of  $\text{SO}_2$  gas that responsible for pollution of the environments that cause acid rain and lung cancer on human health (Ryemshaket et al.2015; Adekunle et al.2016). Then beneficiation of coal by floatation was used to minimize the amounts of sulfur content in the coal sample which leads to increases in the calorific values and qualities of the coal.

**Table 7** Sulfur content obtained in coal samples

Sample code	Weight of $\text{BaSO}_4$ obtained (gm.)		Sulfur contents (%)	
	untreated (raw)	Treated (floated)	Untreated (raw)	Treated (floated)
ARL1	0.1383	0.0299	1.9	0.41
ARL2	0.0422	0.0182	0.58	0.25
ARL3	0.0415	0.0327	0.57	0.45
ARL4	0.0859	0.0669	1.18	0.92
SR	0.0488	0.0240	0.67	0.33
DR	0.0859	0.0277	1.18	0.38

### 3.10. Oxygen content

The oxygen content of the present study area for raw coal samples is ranging from 5.9 to 15.77 % and for treated coal samples is ranging from 12.58 to 16.89 % as shown in **Table 8**. The expected value of oxygen content for treated coal would be lower than that of untreated coal sample, due to highly reduced ash content and increment of carbon number in the lower range. Therefore values of oxygen content for treated coal samples are higher than that of untreated coal in Sombo streams.

**Table 8** Results for ultimate analysis of coal samples

Sample code	Carbon (%)		Hydrogen (%)		Sulphur (%)		Nitrogen (%)		Oxygen (%)	
	Raw	Treated	Raw	Treated	Raw	treated	Raw	Treated	Raw	Treated
ARL4	37.54	61.06	5.28	2.98	1.18	0.25	1.28	0.92	15.77	12.58
SR	39.90	54.12	4.85	3.26	0.67	0.33	1.33	0.64	5.9	16.89

### 3.11. Calorific value

The experimental value of calorific value for both raw and floated coal samples is determined by using the Adiabatic Bomb calorimeter in the central laboratory of the Geological Survey of Ethiopia. As shown in **Table 9**, the obtained

result of the calorific value for the raw sample is ranging from 5838.46 to 8111.29 Btu/lb. for Achibo from first to fourth layers. But after the treatment, the calorific value is ranging from 10285.25 to 11756.63 Btu/lb. for Achibo site. The Sombo and Dabaso site area also increases in their calorific value to 9833 Btu/lb. and 9438.12 Btu/lb. respectively from its untreated types of coal. This shows that the beneficiation by froth flotation method is appropriate approach to increase calorific value of mentioned sites. Specifically, the calorific value of the top layers of Achibo, Sombo and Dabaso liberated very low amounts of heat; for containing high impurity materials and ash which affect the combustion efficiency of the coal. Top layer is also accounted for lower carbon contents and high amounts of volatile matter than the remaining layers. Even though top layers of the sample sites after treatment show low calorific values, Achibo top layer has better quality than other sites. On the hand, the results from **Table 9**, prove that as the burial depth of Achibo increases the organic carbon content increases which is responsible for an increase in the calorific value. The obtained results confirm that quality of a coal increases when depth increases (Ahmed 2008)

**Table 9** Calorific values of raw and floated coal samples.

Sample code	Calorific value					
	Cal/gm.		MJ/kg (x by factors of 4.18		Btu/lb.(x by factor of 1.8	
	Untreated (raw)	Treated (floated)	Untreated (raw)	Treated (floated)	Untreated (raw)	Treated (floated)
ARL1	3913.89	5714.03	16.36	23.88	7045	10285.25
ARL2	3243.59	5942.87	13.56	24.81	5838.46	10697.17
ARL3	4448.81	6392.13	18.6	26.72	8007.85	11505.83
ARL4	4506.25	6531.46	18.84	27.30	8111.29	11756.63
SR	5295.27	5462.78	22.13	22.88	9531.49	9833
DR	4975.2	5243.4	20.8	21.92	8955.25	9438.12

The obtained proximate value is given rough estimation information of liberating heat value. The obtained value of fixed carbon in Achibo of third and fourth layers was higher than other sites so they liberated more calorific value. Then after treatment, the calorific value of the coal is increased due to an impure material is separated with the help of chemical reagents. In Achibo streams due to increments of the burial depth than all other sitting areas the amounts of fixed carbon was highly changed from its value of 38.31% to 55.87% in lower layers that led to more coal particle was attached on the surface of the reagents and the more calorific value was recorded by changing from 8111.29 Btu/lb. to 11756.63 Btu/lb. The calorific value of the Sombo raw sample is increased from the value 9531.49 Btu/lb. to the treated value of 9833 Btu/lb. These increments of heating value were due to the change of fixed carbon from 34.21 % to 52.81% and the reduction of ash content from its value of 27.08 % to 10.74%. Generally, the relationships between fixed carbon and ash with calorific value have a direct relation. The increasing of fixed carbon increases the calorific value and decreasing the ash contents, but the fixed carbon decreases the calorific value is decreasing with increasing ash content of the coal as shown in **Fig. 5**

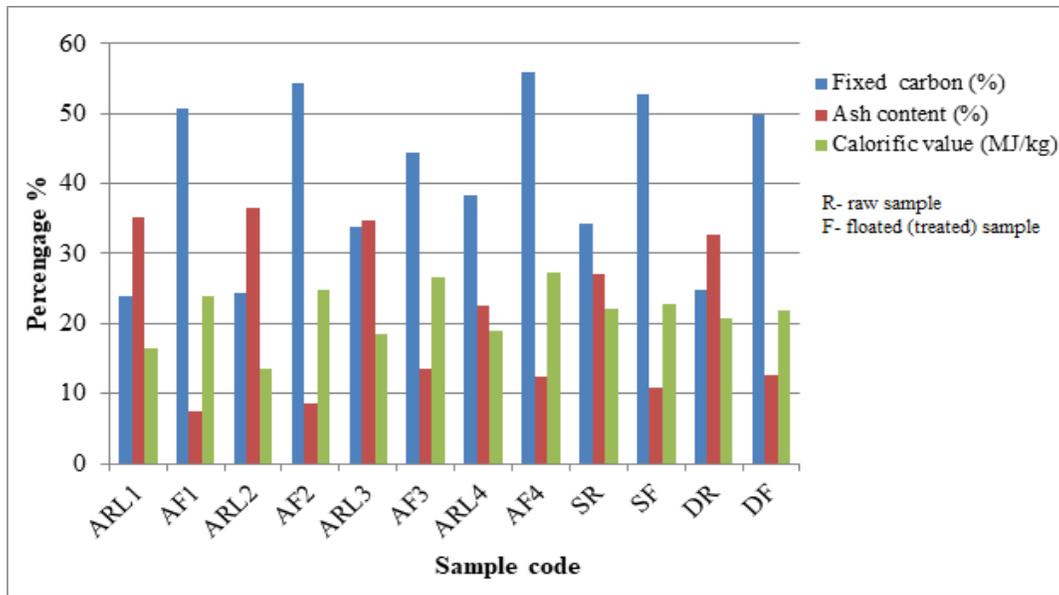


Fig. 3 Relationship between calorific value with proximate analysis

### 3.12. Coal grade determination

ASTM D-388 is used for the classification of coal grades or ages based on the amounts of fixed carbon and calorific values. As shown in **Table 10** of this work, the gross calorific value and fixed carbon results of raw samples from sampled sites are used to classify the grade of raw coal that was found in grade between lignite B and sub-bituminous B. According to the obtained results Sombo and Dabaso sites have better calorific value than Achibo site. Although the two sites *viz* Sombo and Dabaso did not show significant improvement in calorific value after treatment, Achibo site layers shown remarkable change in it. Generally, the gross calorific value (Btu/lb.) is increased from its lower value to higher and this enables to grade the coal in the range of sub-bituminous B and high volatile bituminous C after treatment for four layers of Achibo Site. Although raw coal from top layers of Sombo and Dabaso valleys have been graded under sub-bituminous B and C with higher calorific value, they did not show significant change in calorific value after treatment when they compared with layers of Achibo. This could be justified for presence of high impurities in the layers.

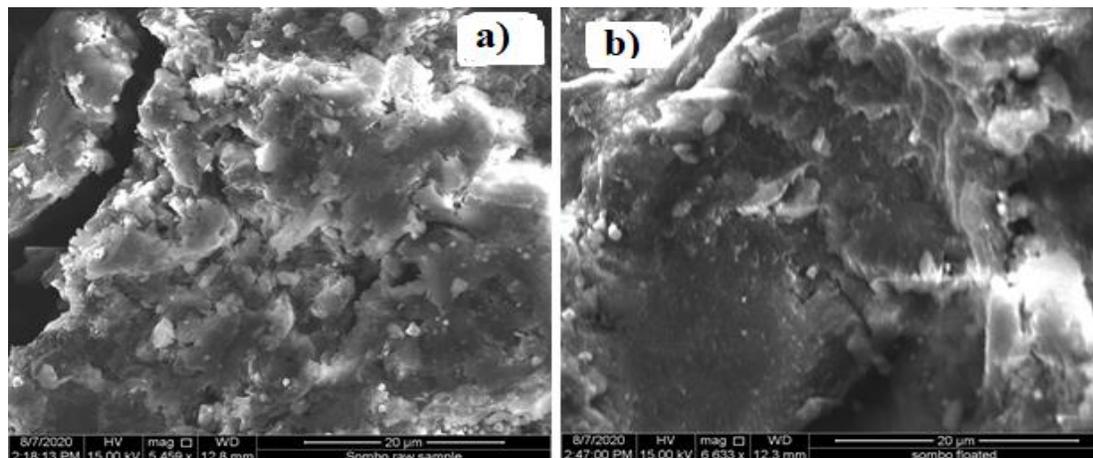
**Table 10** Coal Grade Determination.

Sample code	Fixed carbon (%)		Calorific value Btu/lb.		Grade (groups) of coal	
	Raw	Treated	Raw	Treated	Raw	Treated
ARL1	23.85	50.82	7045	10285.25	Lignite A	Sub-bituminous B
ARL2	24.29	54.36	5838.46	10697.17	Lignite B	Sub-bituminous A

ARL3	33.81	44.47	8007.85	11505.83	Lignite A	High volatile bituminous C
ARL4	38.31	55.87	8111.29	11756.63	Sub-bituminous C	High volatile bituminous C
SR	34.21	49.91	9531.49	9833	Sub-bituminous B	Sub-bituminous B
DR	24.98	52.81	8955.36	9438.12	Sub-bituminous C	Sub-bituminous C

### 3.13. Surface Morphology

The surface morphology of coal particles for the raw and floated sample was revealed in **Figure 6** (a) and (b). The SEM result of the surface of raw coal particles indicated by having large holes and crack formation that increases the surface roughness. So the holes on the surface will adsorb more collector reagent rather than spreading on the surface of the coal particles that decreases the performance of the floatation process. However; after floatation, the surface of the coal demonstrated several physical and chemical alterations by decreasing the holes and cracks of the particle. The SEM image after treatment indicates that the particles are more compacted and homogeneity of particles is increased. This might be due to the removal of impurities and the increment of carbon content in a sample.



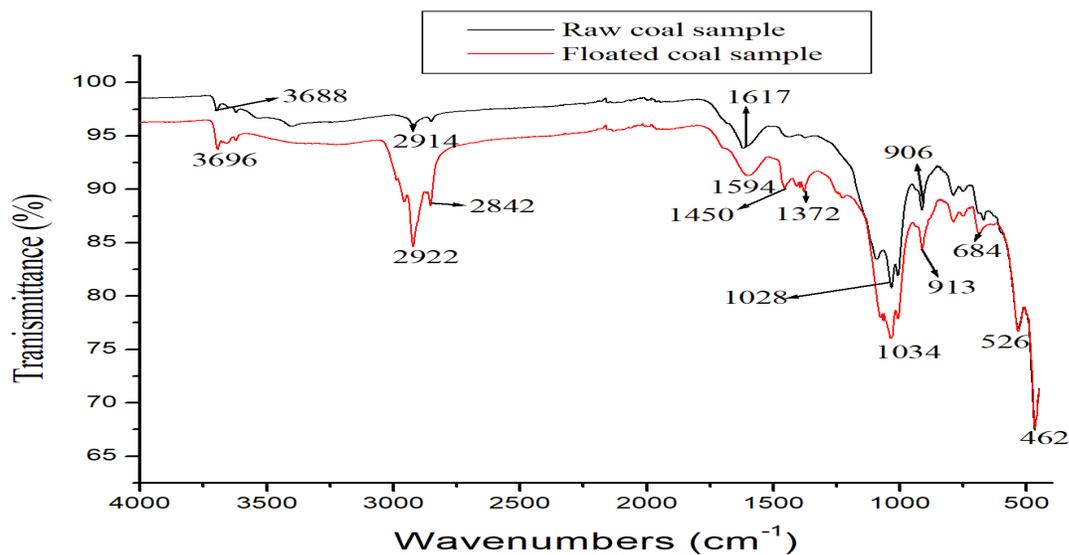
**Fig.4** SEM image of raw (a) and treated coal (b)

### 3.14. Functional Groups

FT-IR analysis was made to understand the nature of the bonds and to recognize various functional groups which exist on the surface coal sample. **Figure 7** shows the FT-IR spectra of coal surface and the peaks at  $2842\text{ cm}^{-1}$  and  $2922\text{ cm}^{-1}$  correspond to C-H stretching of alkane. The peaks at  $1594\text{ cm}^{-1}$  and  $3696\text{ cm}^{-1}$  are representing C=C stretching of aromatic hydrocarbon of benzene rings and O-H stretching of an alcohol group respectively as shown in **Table 11**. The spectra of ash forming minerals in coal analysis were illustrated in the fingerprint region such as the peaks at  $526\text{ cm}^{-1}$  and  $462\text{ cm}^{-1}$  are corresponding to aromatic pairs of sulfide -SH and  $\text{Fe}_2\text{S}$  respectively. To summarize, the results of FTIR confirm that the intensity of the C-H and C=C stretching are increased which may indicate an increase of carbon contents in treated sample (Roya et al. 2020; Zhu et al. 2020; Yang et al.2020)

**Table 11** The characteristic of FT-IR coal surface functional groups

Wavenumbers (cm <sup>-1</sup> )	Functional groups	Compound class	Intensity
684	C=C bending	Alkene	Strong
913	O-H bend	Carboxylic acids	Medium
1034	S=O stretching	Sulfoxide	Strong
1372	S=O stretching	Sulfonate	Strong
1450	C-H bending	Alkane	Medium
1594	C=C stretching vibration	Aromatic hydrocarbon on a benzene ring	Strong
1617	C=C stretching	$\alpha$ , $\beta$ unsaturated ketone	Strong
2842	C-H stretching	Alkane	Medium
	N-H stretching	Amine salt	Strong
2922	C-H stretching	Alkane	Medium
3696	O-H stretching	Alcohol	Sharp, medium

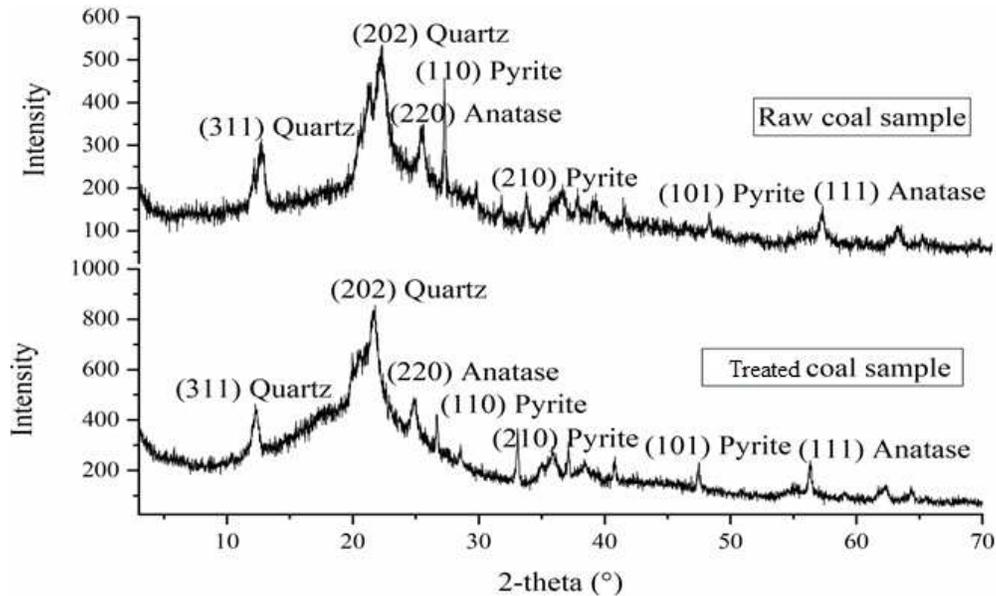
**Fig.5** FT-IR spectra coal sample before and after treatment

### 3.15. Crystal structures and mineral Identification

The crystalline structure and mineral compositions for both raw and floated coal particles are examined by using XRD as shown in **Figure 8**. The peaks at 2-theta values of 12.55 and 22.14° were recognized as the (310) and (528) planes of SiO<sub>2</sub> respectively (JCPDS card no. 027-0605 Silicon oxide (Quartz)). But the peaks at 2-theta values of 26, 33 and 47° were corresponding to (449), (185) and (139) shows the planes of FeS<sub>2</sub> respectively (JCPDS card no. 042-1340 Iron Sulfide (Pyrite)). Whereas the peaks at 2-theta values of 24 and 56° were corresponding to (487) and (232)

the planes of  $\text{TiO}_2$  respectively (JCPDS card no. 021-1272 Titanium oxides (Anatase, syn)). Hence, the XRD result of both samples shows that the coal particle contained the minerals of quartz, pyrite and anatase.

The peaks of the pyrite minerals at floated samples were minimized which shown that the removal of sulfide forms ash and shows slightly shifts of the peaks on floated coal samples. XRD results confirm that quartz ( $\text{SiO}_2$ ), pyrite ( $\text{Fe}_2\text{S}$ ) and anatase ( $\text{TiO}_2$ ) which are responsible for ash increment in the sampled coal are common and the results are similar with work of (Chen et al.2018). In addition to this the XRD image proves that the minerals content is decreased after treatment.



**Fig. 6** XRD Analysis of both raw and treated coal samples

#### 4. CONCLUSION

The Achibo-Sombo coal deposit is treated by physiochemical beneficiation via floatation method to increases the calorific value by using diesel oil and n-octanol to obtain the coal recoveries of 77.5% yield of Achibo, 73.5% yield of Sombo and 67.9% yield for Dabaso. The amount of ash and sulfur content of the area is highly reduced from its maximum value of 36.58% of second layers of raw Achibo to a minimum value of 7.49% of first layers of Achibo treated sample. The grades of raw coal in the studying area fell under lignite B coal to sub-bituminous B groups with calorific value ranged from 5838.46 to 9531.49 Btu/lb. But after treatment of the samples the grades of the coal are ranging under sub-bituminous B to high volatile bituminous C with the calorific values ranging from 9438.12 to 11756.63 Btu/lb. The results of FTIR confirm that the intensity of the C-H and C=C stretching are increased which is a clear indication of an increase of carbon contents in treated samples. The SEM image after treatment indicates that the particles are more compacted and homogeneity of particles is increased. This might be due to the removal of impurities and the increment of carbon content in a sample. The XRD obtained result confirms that the quartz ( $\text{SiO}_2$ ), pyrite ( $\text{Fe}_2\text{S}$ ) and anatase ( $\text{TiO}_2$ ) which are responsible for ash increment in the sampled coal are reduced

after treatment. Finally, the overall finding of this study confirms that the calorific value of Achibo-Sombo coal is upgraded to the expected level of the desired application by the froth floatation technique implemented.

## Abbreviation

A, Ash content; BCG, Bromocresol green indicator; Btu/lb., British thermal unit per pound; ASTM, American society for testing and materials; e1, correction in calories for heat of formation of nitric acid (HNO<sub>3</sub>); e2, correction in calories for heat of formation of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>); e3, correction in calories for heat of combustion of fuse wire; Hg, gross calorific value; JCPS, Joint Committee on Power Diffraction Standards; m, mass of coal sample taken in gm; M, Moisture content; MR, Methyl red; PR china, Peoples Republic china; V1, Volume of hydrochloric acid consumed for blank; V2, Volume of hydrochloric acid consumed for sample; t, temperature changes of bomb calorimeter; w, energy equivalent of calorimeter in cal/°C;

## Conflicts of interest/Competing interests

The authors have no conflict of interest

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# Figures

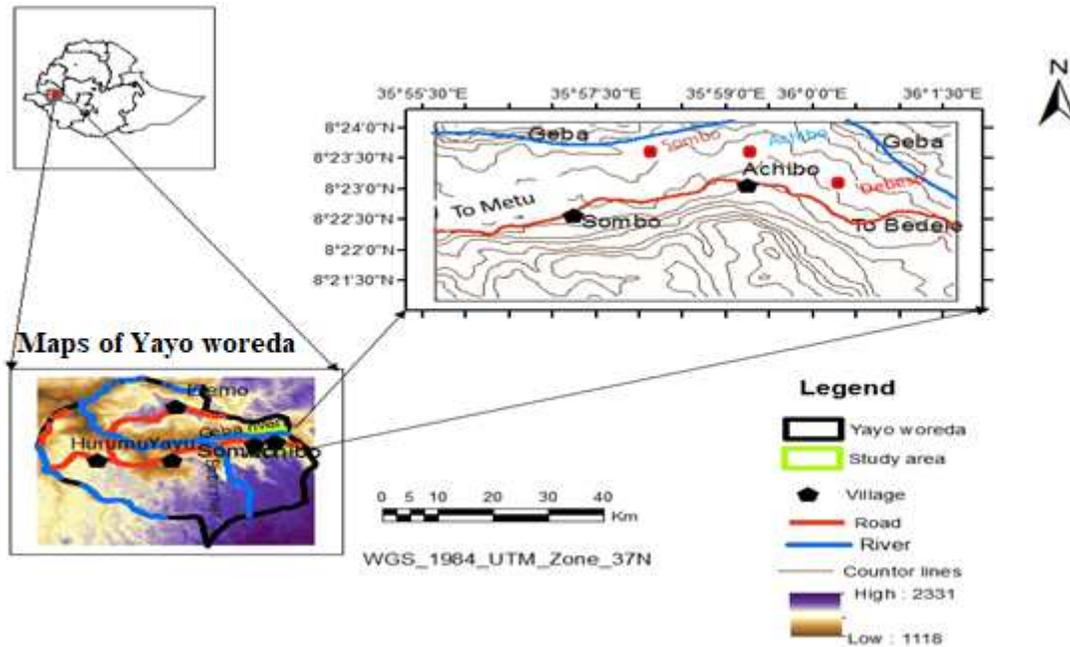


Figure 1

Location of the study area Note: The designations employed and the presentation of the material on this map do not imply the expression of any opinion whatsoever on the part of Research Square concerning the legal status of any country, territory, city or area or of its authorities, or concerning the delimitation of its frontiers or boundaries. This map has been provided by the authors.



Figure 2

The sampling area a) exploration site b) surface mining of coal deposits c) mined coal



Figure 3

Froth flotation procedures and resulting coal particles

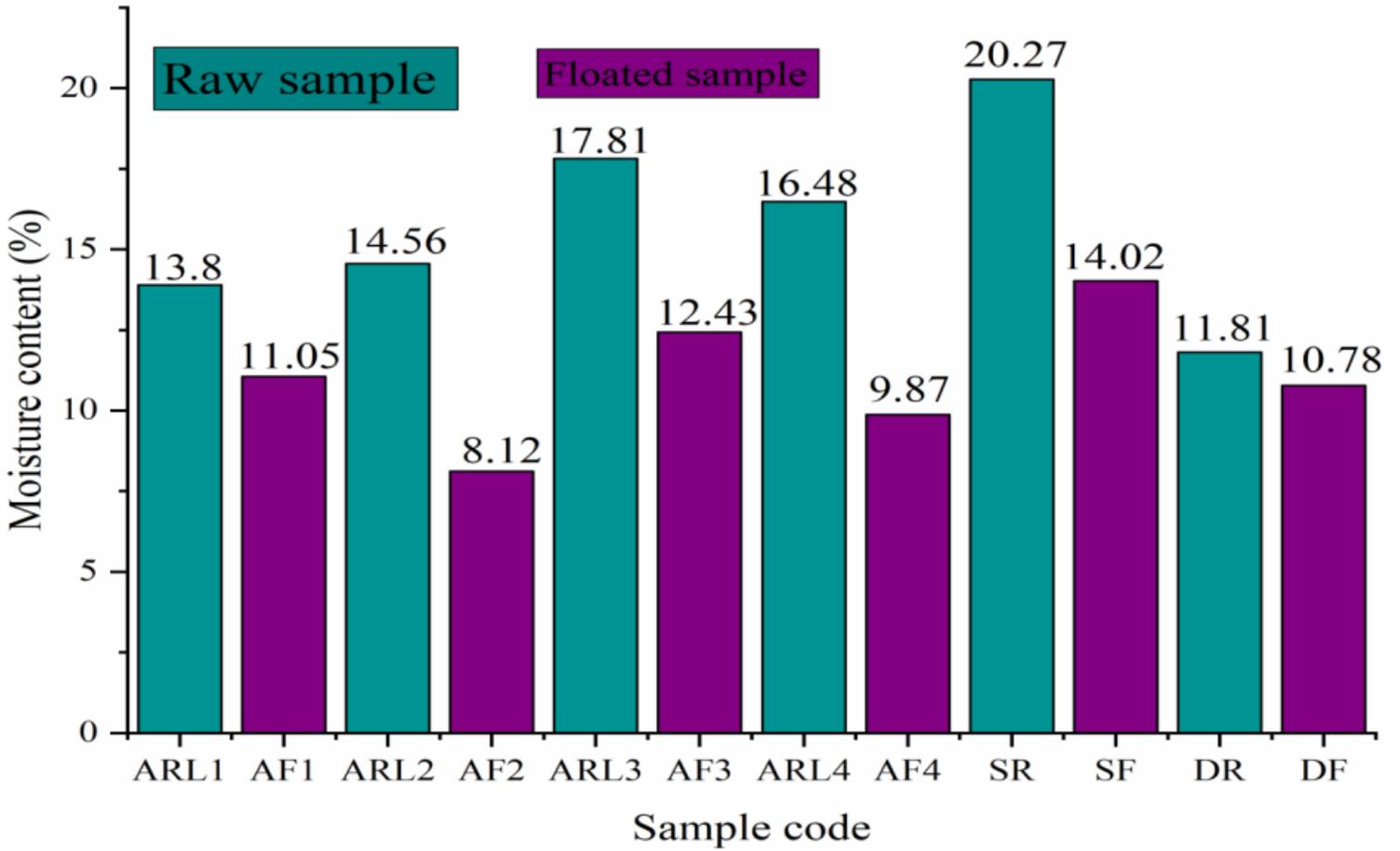


Figure 4

Moisture contents vs. Sample code

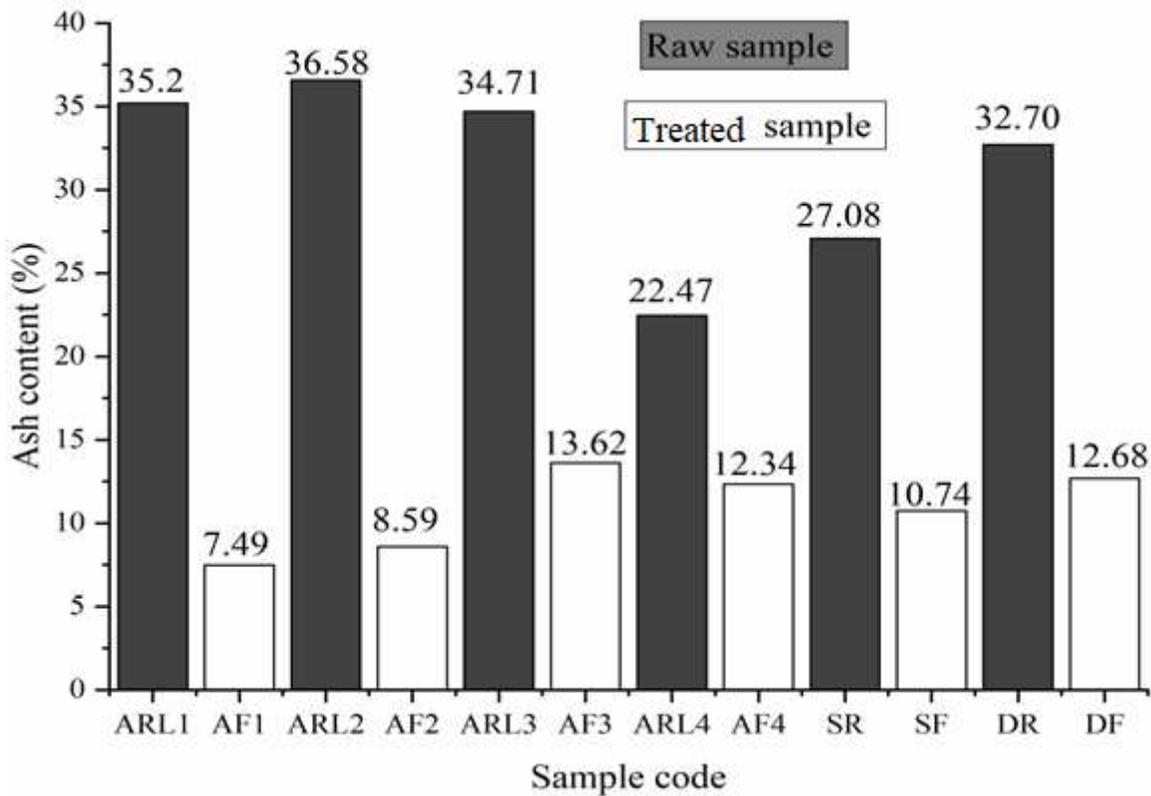


Figure 5

Ash contents for raw and floated coal sample

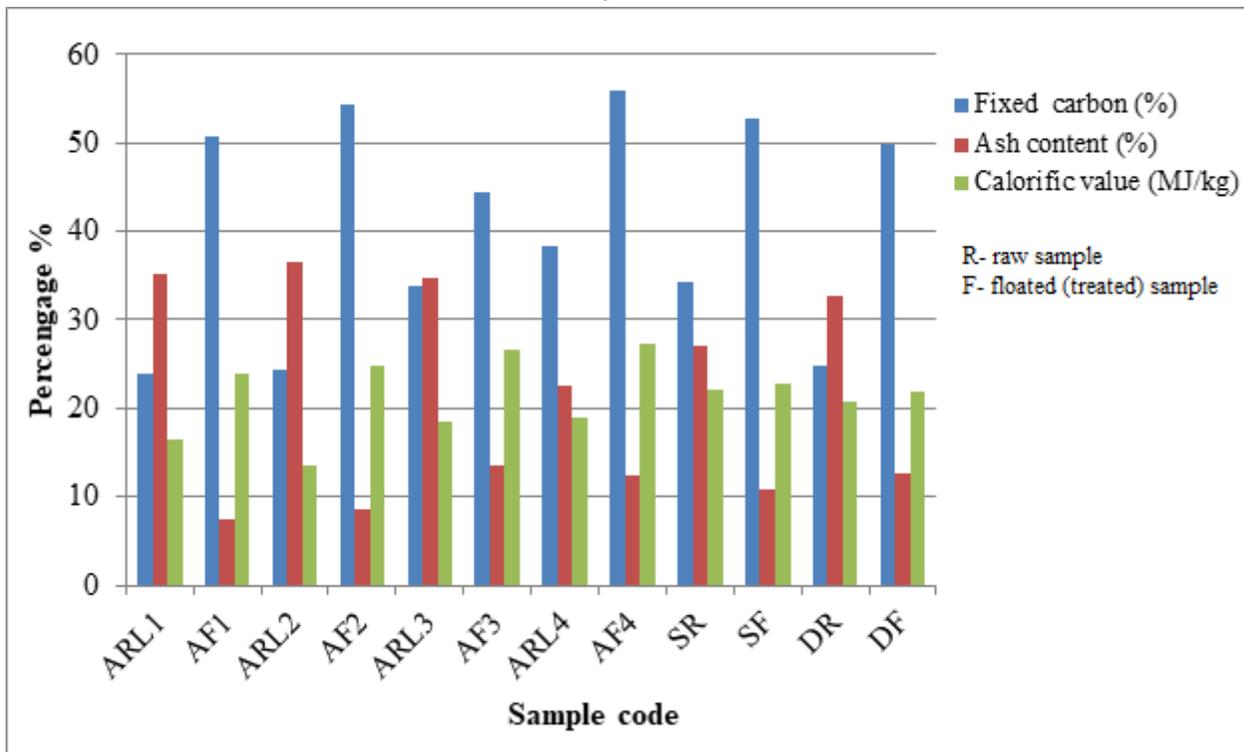


Figure 6

Relationship between calorific value with proximate analysis

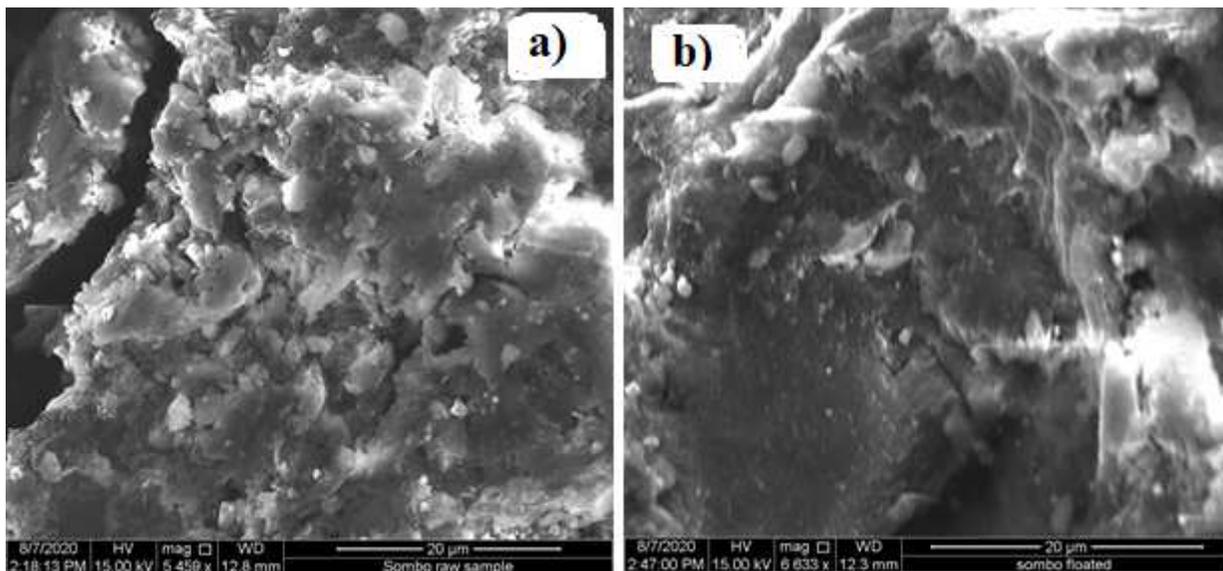


Figure 7

SEM image of raw (a) and treated coal (b)

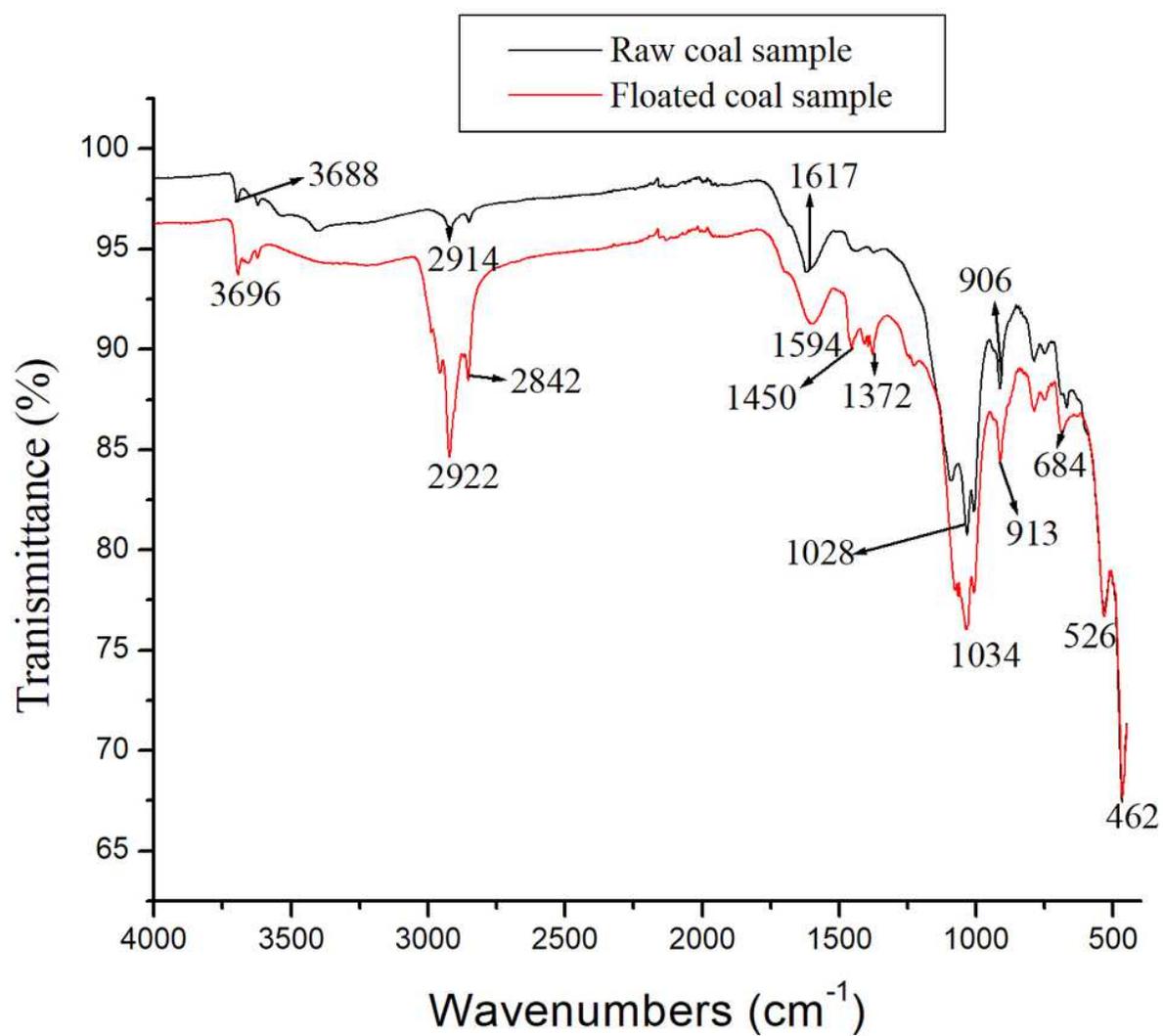


Figure 8

FT-IR spectra coal sample before and after treatment

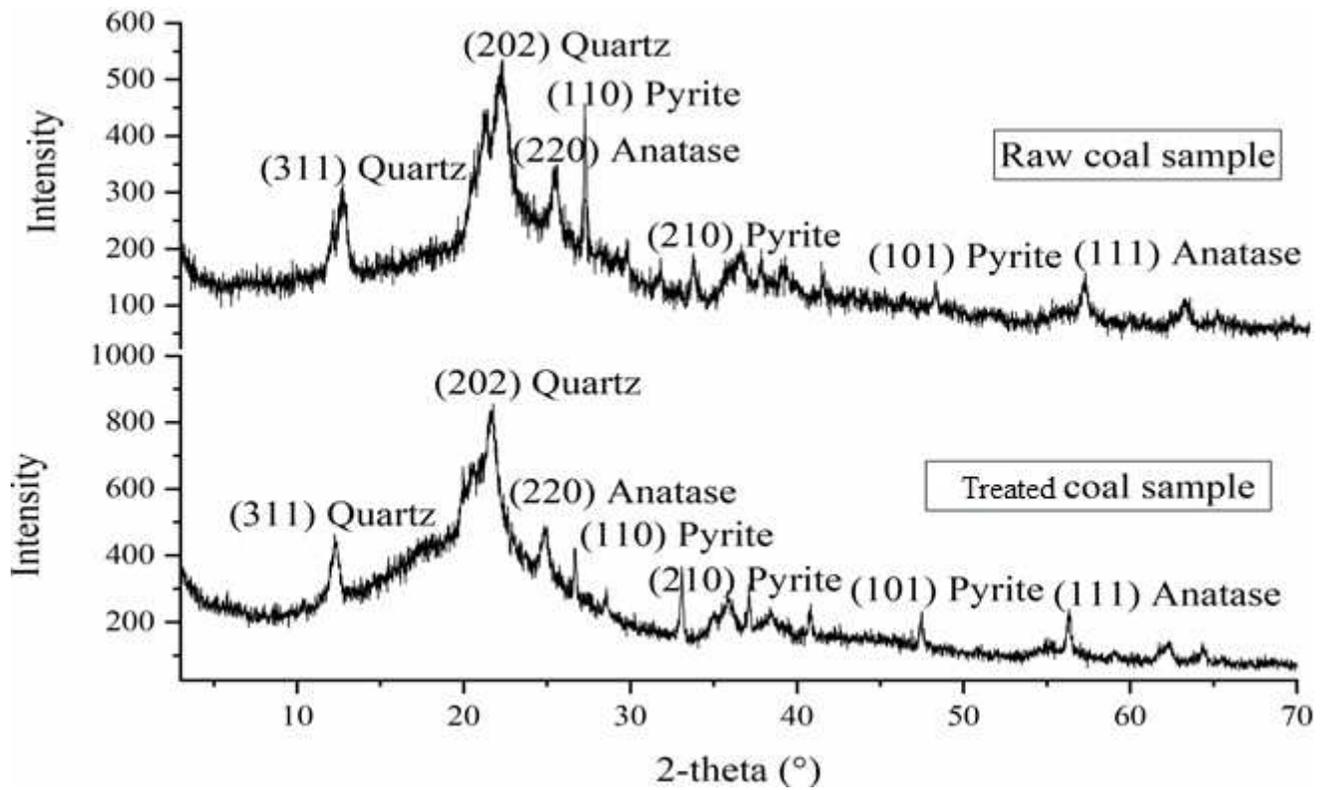


Figure 9

XRD Analysis of both raw and treated coal samples