

Flotation and Leaching Studies on Processing of El-Magharah Coal, Sinai, Egypt

¹Mostafa A. Metwally*; ²W. M. Fathy; ¹M. G. Farghaly; ²A. M. Ramadan; ²A. M. Saleh; ²M. R. Moharam

¹Al-Azhar University, Faculty of Eng., Mining and Petroleum Dept. Gena, Egypt

²Al-Azhar University, Faculty of Eng., Mining and Petroleum Dept. Nasr City, Cairo, Egypt

*Corresponding Author: ibnanwar2@yahoo.com

Abstract— Coal is a heterogeneous, complex and non-crystalline macromolecules containing a percentage of ash and sulfur, which play an important role in almost of all coal utilization systems. Cleaning of coal before using is an inevitable to achieve clean fuel and reduce the environmental impacts. This study applies the flotation and leaching techniques on El-Magharah coal for enhancing ash reduction and desulphurization. A low rank coal sample of El-Magharah coal (22.5% ash, and 3.54% total sulphur) was subjected to a flotation process to reduce ash and total sulphur. Subsequently, a clean coal of 13% ash and 2.14% total sulphur was obtained. The obtained clean sample from flotation was subjected to a leaching process using NaOH to reduce the residual total sulphur, followed by leaching with 10% HCl to reduce ash to a minimum. In this regard, the effect of sodium hydroxide as a leachant, particle size, solid/liquid ratio, temperature, and leaching time were examined. The final product was found to contain total sulphur 1.10% with 54.77% sulphur reduction and ash 8.1% with 59.27% ash reduction.

Keywords— Low rank Coal, Demineralization, Desulfurization, Flotation, Leaching, Sodium hydroxide, Hydrochloric acid.

I. INTRODUCTION

Coal is the most abundant fossil fuel available on the Earth which accounts for about 55% of the World's electricity generation. Apart from this, coal finds its application in many other fields such as metallurgical coke manufacture (in steel industries), polymers, fibers, dyes, alumina refineries. In the recent time, it has found many applications towards advanced carbon nano tubes, carbon electrode, carbon fibers ... etc. [1]. Run-of-mine coal generally has an ash content of 5-40% and a sulfur (S) content of 0.3-8% depending on the geologic conditions and mining technique used [2].

Existence of sulfur compounds in coal limits its industrial application due to environmental as well as technical problems. Sulfur is present in coal in three forms: pyrite, organic, and sulfate. The organic sulfur directly bound to the coal matrix is in the form of thiols, sulfides, disulfides, thiophenes, and cyclic sulfides. Pyritic sulfur (FeS₂) occurs in mineral phases as agglomerates of pyrite and marcasite crystals. The sulfate exists mostly as sulfates of iron and calcium. Silica in different forms such as quartz, crystallite; clay minerals such as kaolinite, illite; carbonates such as

calcite, dolomite, siderite; sulfate and sulfides, are the major ash forming minerals in coal [3]. However, high sulfur coals can be upgraded by desulphurization through physical, chemical and biotechnological processes. One of the most suitable physicochemical methods is flotation for removal of pyritic sulfur from coal [4]. In this technique, separation of fine coals relies upon the wetting ability differences between the coal-rich and mineral-rich particles in an aqueous solution [5, 6]. Leaching is one of the common methods employed for coal desulfurization. Coal is mixed with acid or alkali and its sulfur is extracted while being heated or stirred. The type of leachant depends upon the type of sulfur in the coal. The pyritic sulfur (iron sulfide) is usually reduced using the direct leaching method with acid. This method is the most effective method in eliminating metal compounds from coal [7].

The main objective of this paper was to study the flotation and leaching techniques on processing El-Magharah coal for enhancing ash reduction and desulphurization. The quality of the flotation and leaching products were evaluated in terms of the percentage of combustible matter recovery, sulphur reduction and ash reduction percentages.

II. EXPERIMENTAL WORK

The experimental work was planned in two steps: clean of El-Magharah coal by flotation technique, and leaching the clean coal by using NaOH to reduce the residual total sulphur and ash to a minimum.

A. Experimental Setup

The experimental setup involved a laboratory-scale Denver flotation cell and a three neck conical flask. Although this type of flotation machine has a self-inducing aerator in which the air flow rate is dependent on the impeller speed, the flow rate of compressed air to the machine was controlled by an air flowmeter. The liquid (local tap water) volume in the tank was maintained at 1 L for all experiments.

B. Parameters Investigated

The investigated parameters and their tested ranges are tabulated in Table 1.

C. Materials and Methods

A 50 kg of El-Magharah low rank coal sample (22.5% ash, 3.54% total sulphur) was received as -4 mm crushed and screened sample. It was well mixed and split into 1 kg for X-ray diffraction (XRD), X-ray fluorescence (XRF), and size analyses. The sample was

measured using XRD in the laboratories of the Egyptian Mineral Resources Authority (EMRA). The results of XRD analysis showed that the major phases of minerals were Graphite, Quartz, Sulphur, pyrite, and kaolinite. The head sample and feed size fractions were also analyzed using XRF, in the laboratories of the Egyptian Mineral Resources Authority (EMRA), the results of which are tabulated in tables 2, 3, and 4. Based on these results, the bulk sample was screened on a 0.5 mm wedge wire screen using a vibrating shaker. The oversize material was ground by a disc grinder and screened on a 0.5 mm wedge wire screen until all material has passed through the 0.5 mm.

Table 1: Parameters investigated:

Parameter	Range
Flotation stage	
Collector dosage, (kg/t).	1 : 4.
pH.	2 : 10.
Collector type.	Kerosene, Oleic Acid, and Fuel
Solid/liquid ratio, (%).	5 : 20.
Agitation speed, (rpm).	800 : 1400.
Particle size, (μm).	-500 : -180
Leaching stage	
Alkali concentration, (M).	0.5 – 3.0.
Particle size, (μm).	-500 : -180
Solid/liquid ratio, (gm/ml).	1:4 to 1:10
Temperature ($^{\circ}\text{C}$).	40 : 100
Leaching time, (min).	30 : 120

Table 2: Chemical analysis of the head sample.

Constituents	Assay %
SiO ₂	9.86
TiO ₂	0.48
Al ₂ O ₃	4.85
Fe ₂ O ₃	3.09
MnO	<0.01
MgO	0.17
CaO	0.72
Na ₂ O	0.09
K ₂ O	0.17
P ₂ O ₅	0.04
Cl	0.01
SO ₃	6.29
LOI	74.03

Approximately, 20 kg of the under size material (500 μm) was collected as feed for flotation experiments. Kerosene, fuel oil and oleic acid (from Al-Nasr Pharmaceutical Chemicals Company) were investigated as collectors. Other reagents used were hydrochloric acid and sodium hydroxide for pH control, and pine oil was used as a frother. Local tap water was used in all experiments. Batch flotation tests were performed in a 1 L cell. For all the flotation experiments, the float as well as the sink material were filtered, dried, weighed, and analyzed for ash content. Quick assessment of flotation experiments was obtained by

assaying the dried float and sink material for their percentage of combustible matter recovery.

The percentage of combustible matter recovery was calculated as shown in equation no. (1). [6]

$$\text{Combustible Matter Recovery, \%} = \frac{M_c * (100 - A_c)}{M_f * (100 - A_f)} * 100 \quad (1)$$

Where:

M_f = mass of feed.

M_c = mass of concentrate (float).

A_f = ash of feed.

A_c = ash of concentrate (float).

At the end of the first stage, the clean coal was collected and considered as a feed for the leaching process using NaOH, followed by leaching with 10% HCl.

The percentage of sulphur and ash reductions were calculated as shown in equations (2) & (3) respectively. [7]

$$\text{Sulphur reduction, \%} = \frac{[x_1 - x_2 \left(\frac{m_2}{m_1}\right)]}{x_1} * 100 \quad (2)$$

$$\text{Ash reduction, \%} = \frac{[y_1 - y_2 \left(\frac{m_2}{m_1}\right)]}{y_1} * 100 \quad (3)$$

Where:

m₁ = weight of clean coal dry sample before leaching.

m₂ = weight of dried sample after leaching.

x₁ = sulphur percentage in the clean coal sample.

x₂ = sulphur percentage in the coal obtained from leaching.

y₁ = ash percentage in the clean coal sample.

y₂ = ash percentage in the coal obtained from leaching.

Table 3: proximate & ultimate analyses of the head sample:

Parameters	Value, %
Proximate analysis	
Ash	22.5
Moisture	2.60
Volatile matter	43.60
Fixed carbon	31.30
Ultimate analysis	
Carbon	56.30
Hydrogen	4.8
Nitrogen	1.13
Sulphur	3.54
Oxygen, (by diff.)	9.13

Table 4: Sieve analysis, sulphur%, ash content%, moisture%, volatile matter%, and fixed carbon% of the head sample.

Size in Microns	Wt. % retained	Sulphur %	Ash Content %	Volatile matter %	Moisture %	Fixed Carbon %
-2000+1400	7.48	2.91	32.30	39.60	2.00	26.10
-1400+1000	15.80	2.70	18.10	46.90	1.80	33.20
-1000 +710	17.69	2.64	15.60	47.40	2.00	35.00
-710 +500	10.84	2.93	17.10	47.20	1.90	33.80
-500 +355	7.42	3.09	17.50	46.60	1.50	34.40
-355 +250	6.52	3.12	18.20	45.50	2.00	34.30
-250 +180	6.29	3.26	19.90	44.70	2.40	33.00
-180	27.96	3.68	30.30	40.30	2.70	26.70
	100	3.10	22.10	44.38	2.14	31.38

III. RESULTS AND DISCUSSION

A. Flotation stage

1. Effect of collector dosage

The effect of kerosene dosage as a collector on the floatability of clean coal from its tailing is illustrated in Fig. 1. The concentration of collector is varied in the range of 1 - 4 kg per ton of coal.

From this figure, it can be seen that, an increase of collector dosage resulted in an overall increase in combustible matter recovery. No change in ash of concentrate was observed. This may be attributed to, at low concentration of reagents, only coal grains of higher hydrophobicity and low ash content float. When the reagent concentration increases, the surface of the coal grains and /or composite grains of less hydrophobicity, especially the coarser and the finer fractions of lower floatability, become more hydrophobic and float. This agrees well with other investigators [8].

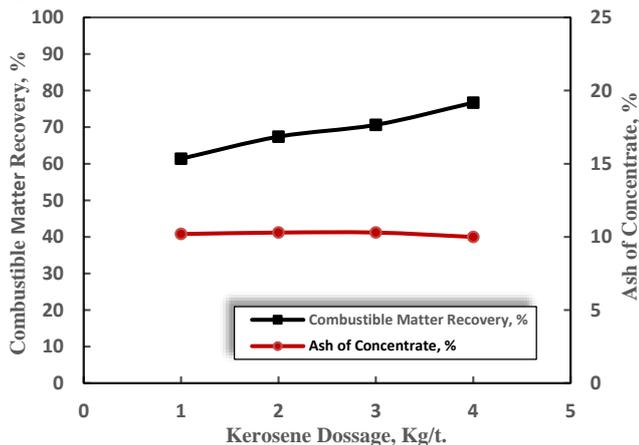


Fig. 1: Effect of collector dosage (Kerosene) on the floatability of clean coal from its tailing at different collector dosages.

[Test conditions comprised: collector type kerosene; pH 6±0.1; size -500 µm; pulp density 10 %; agitation speed 1000 rpm; Air flow rate 20L/min.; pine oil 28 ml at 0.25 % by volume.]

2. Effect of pH

Delicate pH control is very important in selective flotation process. Figure 2 shows the effect of pH value on the floatability of clean coal from its tailing at pH values ranging from 2-10. It is clear that gradually increase of combustible matter recovery is observed by increasing the pH value from 2-8. After that, inflection point was observed by increasing the pH over 8.

This may be due to the dispersion of clay particles in the system. At natural slurry pH of 5.8, coal particles carry a higher negative charge while clay particles remain positively charged till pH of 7. So under natural pH conditions, clay particles must be attracted towards the coal particles and thus imparting the hydrophilic character to coal particles. Increasing the slurry pH to 7 creates a negative charge on the clay particles and electrostatic repulsion takes place between clay and coal particles. Dispersion of clay particles in the solution allows the collector to interact with coal particles surface and thus better flotation recovery was achieved at pH 7 [9].

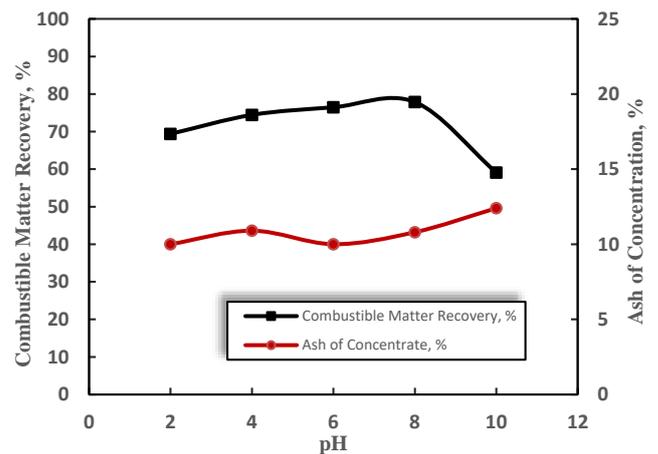


Fig. 2: Effect of pH value on the floatability of clean coal from its tailing.

[Test conditions comprised: collector type and dosage kerosene, 4 kg/t; size -500 µm; pulp density 10 %; agitation speed 1000 rpm; Air flow rate 20 L/min.; pine oil 28 ml at 0.25 % by volume.]

3. Effect of collector type

Figure 1 shows the effect of collector type on the floatability of clean coal from its tailing at different collector dosages. It is obvious that the best result ($R_c = 80\%$) among all types of collectors was obtained with kerosene at 3-4 kg/t. At the same dosage the fuel oil and the oleic acid were appeared less effectiveness. This figure indicates that kerosene has more effect to the floatability of coal surface than fuel oil and oleic acid. To understand the effect of kerosene on the surface of coal, the system mechanism should be clear. The surface of unoxidized coal is naturally hydrophobic because coal is mainly composed of nonpolar

hydrocarbons. Since water is a polar liquid, it has little tendency to wet nonpolar materials such as coal. Oils, on the other hand, have a strong affinity for nonpolar surfaces and wet them easily which in turn, increasing the hydrophobicity of these surfaces. The various ash minerals, such as silicates and clays, are composed of strongly polar compounds, therefore water wets them but oils do not. This basic difference in structure is then responsible for the ease of cleaning coal by flotation. So, it is clear that both oils and water are participated in flotation process [10].

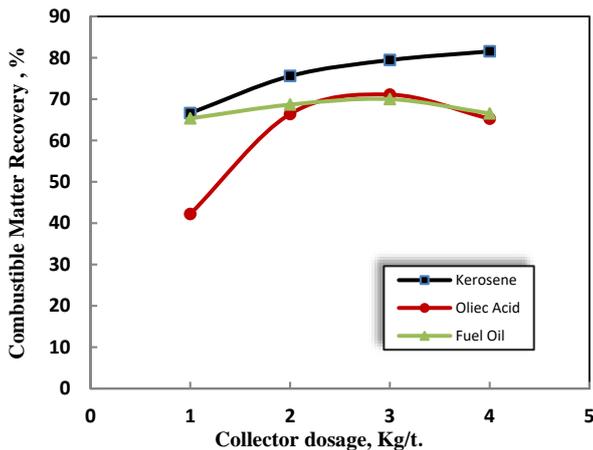


Fig. 3: Effect of collector type on the floatability of clean coal from its tailing at different collector dosages.

[Test conditions comprised: pH 7 ± 0.1 ; size $-500\ \mu\text{m}$; pulp density 10 %; agitation speed 1,000 rpm; Air flow rate 20L/min.; pine oil 28ml at 0.25% by volume.]

4. Effect of pulp density

The effect of variation of pulp density on the floatability of clean coal from its tailing is illustrated in Fig.4. The pulp density is varied in the range of 5.0 – 20.0% solids by weight. From figure 4, it can be seen that, the best pulp density was found at 12.5% where a maximum of combustible matter recovery was obtained and a minimum ash of concentrate. In coal flotation, the optimum pulp density was reported to be generally lower, approximately around 12% by weight [11]. This may be regarded, as a consequence of reduced mechanical entrapment of gangue in the froth [12].

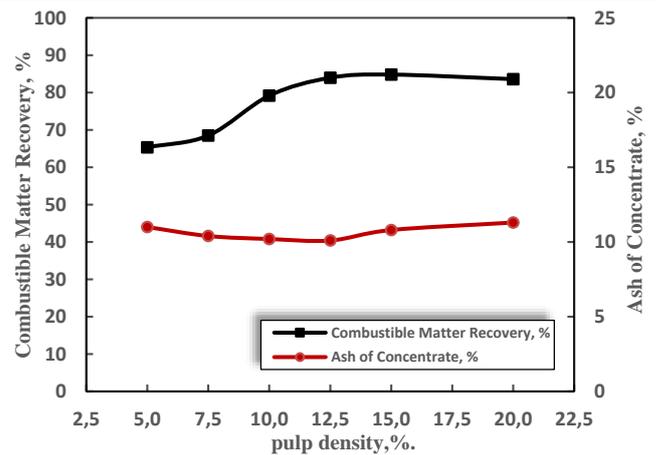


Fig. 4: Effect of pulp density on the floatability of clean coal from its tailing.

[Test conditions comprised: collector type and dosage kerosene, 3kg/t; pH 7 ± 0.1 ; size $-500\ \mu\text{m}$; agitation speed 1000 rpm; Air flow rate 20 l/min.; pine oil 28ml.at 0.25 % by volume.]

5. Effect of agitation speed

Figure.5 shows the effect of agitation speed on the floatability of clean coal from its tailing. The agitation speed is varied in the range of 800 – 1400 rpm.

At an agitation speed of 1200-1400 rpm, the combustible matter recovery % was decreased as a result of the predominant turbulent flow regime. From figure 5, it can be seen that, the best speed of agitation was found at 1000rpm, where a maximum combustible matter recovery and a low ash of concentrate were found. The low combustible matter recovery at low agitation speed ($<1000\ \text{rpm}$), may be attributed to, insufficient mixing, insufficient dispersion of an oily collector and poor bubble-particle contact. Excessively high speeds can decrease effective air dispersion and destroy fruitful bubble-particle contact [13].

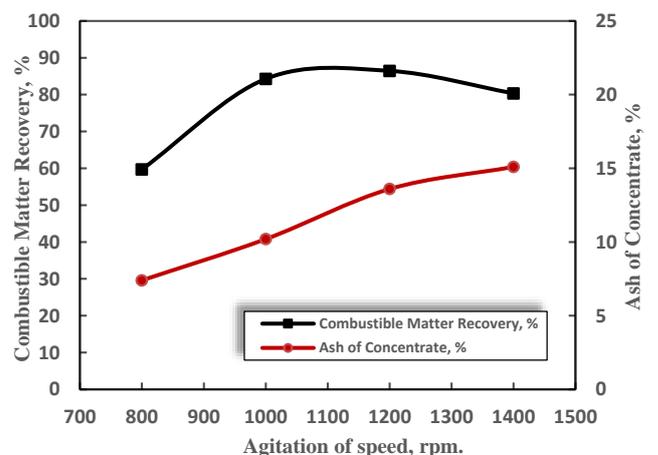


Fig. -5: Effect of agitation speed on the floatability of clean coal from its tailing.

[Test conditions comprised: collector type and dosage kerosene, 3 kg/t; pH 7 ± 0.1 ; size $-500\ \mu\text{m}$; pulp density 12.5 %; Air flow rate 20 L/min.; pine oil 28ml.at 0.25 % by volume.]

6. Effect of particle size

The effect of variation of particle size the floatability of clean coal from its tailing is illustrated in Fig. 6. The particle size is varied in the range of -180 to -500 μm .

From Fig. 6 it can be seen that a decrease of particle size resulted in an overall increase in combustible matter recovery, and an increase in ash of concentrate. As particle size increased (coarse particles), combustible recovery decreased rapidly [14]. Generally, the coarse coal has good selectivity, but the flotation rate is slow. When the particle size is too big, the bubbles can't carry over and hence it is lost in the tailings. Conversely, for fine coal particles the selectivity is poor and flotation rate is high. By comparison, it can be predicted that medium-size coal particles have good floatability and represent a better choice [15].

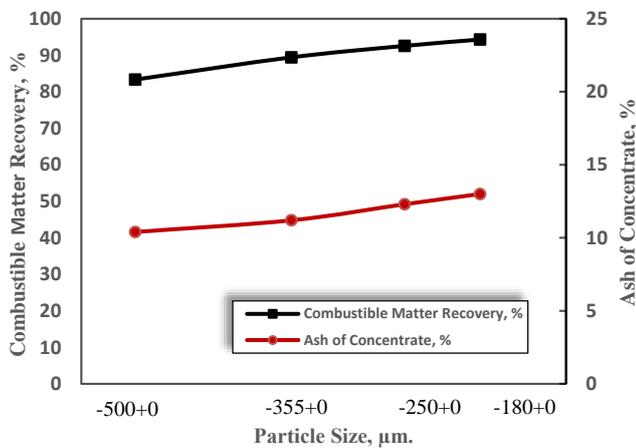


Fig. 6: Effect of variation of particle size on the floatability of clean coal from its tailing.

[Test conditions comprised: collector type and dosage kerosene, 3 kg/t; pH 7 ± 0.1 ; pulp density 12.5%; agitation speed 1000 rpm; Air flow rate 20 L/min.; pine oil 28ml.at 0.25 % by volume.]

B. Leaching stage

Carried on flotation concentrate at optimum conditions. In this stage, the suitability of sodium hydroxide solution to leach sulphur from clean coal (flotation concentrate), was studied by the agitation leaching technique. The specification of clean coal sample taken as a leaching feed is shown in table 5.

Table 5: Specifications of clean coal (Flotation concentrate at optimum conditions):

Size, μm	Ash, %	Carbon, %	Hydrogen, %	Nitrogen, %	Sulphur, %
-	13.0	65.88	5.44	1.33	2.14

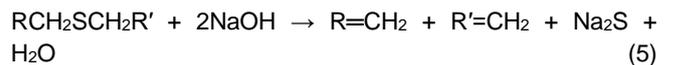
The leaching process was carried out as follows: 10gm. of clean coal was suspended in 100 ml. of a sodium hydroxide solution of a different concentration, and then stirred at 400 rpm in 500ml three neck conical flask for 60 min at 100°C . The slurry was filtered and the filtrate was analyzed in the laboratories of the

Egyptian Mineral Resources Authority (EMRA), to determine the sulphur content.

The alkali removes different forms of sulfur, sulfate, pyritic, as well as organic sulfur. Alkali may react with pyritic sulfur forming soluble sulfates as per the following reaction:



Alkali may also convert some of the organic functional groups such as thiols, disulfide, etc., present in the coal to soluble salts:



Where: R =aryl group.

1) Effect of an Alkali concentration on sulphur reduction

The effect of NaOH concentration on sulphur removal from clean coal samples at different concentration (0.5, 1.0, 2.0, and 3.0 M), is illustrated in Fig. 7. By gradual increasing the alkali concentration from 0.5M to 3M, the removal of sulphur increased until it reaches to 48.6% at 2M alkali concentration. By increasing the alkali concentration beyond 2M, sulphur reduction was found to slightly increase until it reaches to 53.27% at 3M alkali concentration. Therefore, the concentration of 2M NaOH was chosen as the best concentration. This may be due to more solvent was available to interact with sulphur present in clean coal. These results agree well with many investigations [16].

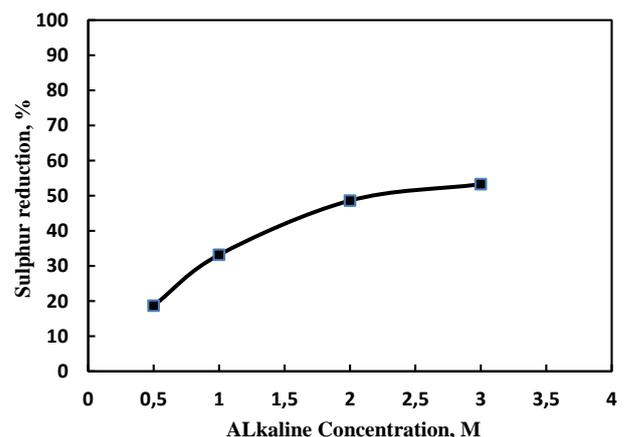


Fig. 7: Effect of NaOH concentration on sulphur reduction.

[Test conditions comprised: size fraction -180 μm ; solid/liquid ratio 1:10; agitation speed 400rpm; temperature 100°C ; agitation time 60 min.]

2) Effect of particle size on sulphur reduction.

The particle size of coal is an important parameter in the sulfur reduction. The effect of particle size on sulphur removal from clean coal samples (flotation

concentrate), of the various particle sizes (-500+0, -355+0, -250+0, and -180+0 μm) is illustrated in Fig. 8. From figure 8, it is clear that the rate of sulphur removal increases as the particle size decreases. The smaller the size, the greater is the interfacial area between the solid and liquid, and therefore the higher is the rate of transfer of material and the smaller is the distance the solute must diffuse within the solid as already indicated. These results agree well with many investigations [17].

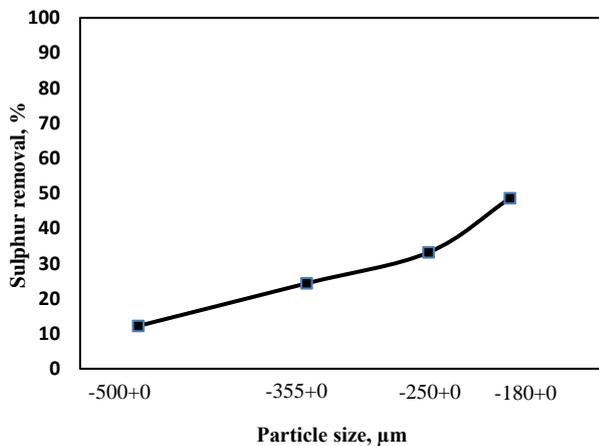


Fig. 8: Effect of particle size on sulphur reduction.

[Test conditions comprised: NaOH concentration 2.0M; solid/liquid ratio 1:10; agitation speed 400 rpm; temperature 100°C; agitation time 60min.]

3) Effect of solid/liquid ratio on sulphur reduction.

The effect of the solid/liquid ratio on sulphur removal from clean coal samples at different solid/liquid ratio (1:4, 1:5, 1:6 and 1:10 S/L ratios, gm/ml), is illustrated in Fig. 9. From the result obtained, it is clear that the removal of sulphur increases with decreasing the pulp density. This may be due to the amount of alkali present in the liquid be enough to leach the sulphur. This is because the active surface area for the reaction is increased with increase in the liquid/solid ratio.

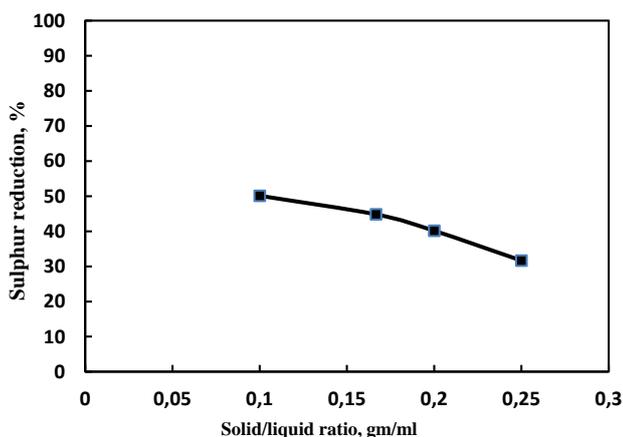


Fig. 9: Effect of solid/liquid ratio on sulphur reduction.

[Test conditions comprised: NaOH concentration 2.0M; particle size -180 μm ; agitation speed 400 rpm; temperature 100°C; agitation time 60min.]

4) Effect of temperature on sulphur reduction.

The effect of temperature on sulphur removal from clean coal is illustrated in Fig. 10. Clean coal samples were leached at (40, 60, 80, and 100°C). From Fig. 10, it is clear that rate of sulphur removal increases as the temperature increases. This may be due to more easy diffusion of solvent into coal particles to extract sulfur from coal. These results agree well with many investigations [18].

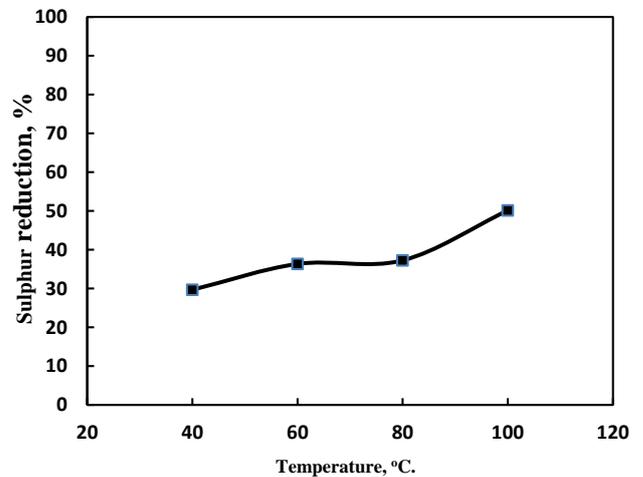


Fig. 10: Effect of temperature on sulphur reduction.

[Test conditions comprised: NaOH concentration 2.0M; particle size -180 μm ; agitation speed 400rpm; solid/liquid ratio 1:10; agitation time 60min.]

5) Effect of leaching time on sulphur reduction.

The effect of leaching time on sulphur removal from clean coal is illustrated in Fig. 11. Clean coal samples were leached for (30, 60, 90, and 120 min).

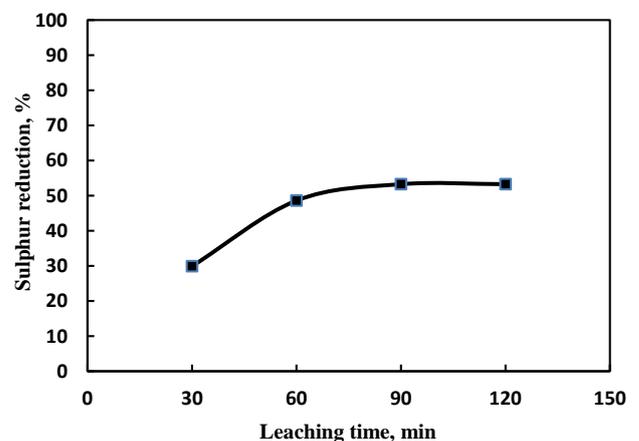


Fig. 11: Effect of agitation time on sulphur reduction.

[Test conditions comprised: NaOH concentration 2.0M; particle size -180 μm ; solid/liquid ratio 1:10; agitation speed 400rpm; temperature 100°C;]

From Fig. 11, It is clear that the rate of sulphur removal increases as leaching time increases, but after specific leaching time (60min), the rate of leaching have slight change. This may be attributed to, more time was available to interact the solvent with sulfur present in coal.. After (60 min,) it change slightly. Initially, the most easily removed sulfur was eliminated,

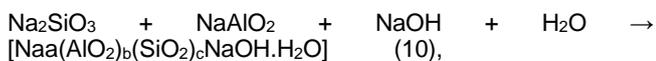
after which the rate of removal was relatively slow. These results agree well with many investigations [17].

6) Effect of HCl on ash reduction.

The behavior of ash removal from coal with alkali treatment is different from sulfur removal from coal. The ash of sample of the best condition, (NaOH 2M; -180 µm; 1:10 S/L ratio; 100°C; 60min), gain from 13% to 17.5%. Alumina and Silica are the two major components of mineral matter present in the coal. NaOH reacted with these two impurities to form soluble aluminates and silicates respectively.



In the presence of NaOH, silicates and aluminates react with each other and form sodium aluminosilicates precipitates [16]. By the following reaction;



(Sodium aluminosilicates precipitates) These precipitates can only dissolve with acid treatment after alkali leaching.

After leaching the sample of the best condition with 10% HCl for 60min at 100°C, ash reduced to 8.1% with 59.27% ash reduction and 54.77% sulphur reduction.

IV. CONCLUSIONS AND RECOMMENDATIONS

- Two steps demineralization and desulfurization were considered as a reasonable approach regarding the preparation of clean coal. Only chemical cleaning cost is too expensive, therefore, demineralization and desulfurization of El-magharah coal was investigated using aqueous caustic leaching following flotation treatment.
- It was found that kerosene as a collector has more desirable hydrophilic effect to the coal surface than fuel oil and oleic acid.
- Flotation technique can be reduce the ash content of 22.5%; and sulphur content of 3.54% to the ash content of 13%; and sulphur content of 2.14%, respectively.
- Chemical cleaning by leaching with aqueous caustic, following by flotation, can effectively remove much of sulphur.
- The increased of alkali concentration, solid/liquid ratio, reaction temperature, treatment time, and reduction in clean coal particle size enhanced the desulphurization rate.
- Coal concentration obtained from flotation was leached using NaOH, and the total sulphur content was 1.10%, and the ash content was 17.5%.
- The clean coal of leaching residue (using NaOH), followed by leaching with 10 % HCl and the total sulphur 1.10% with 54.77% sulphur reduction and ash 8.1% with 59.27% ash reduction.

- The other leaching operation variables should be investigated, such as, solvent type and agitation speed.

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