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Optimization of Concentration Parameters for the Flotation of Alawa Graphite to Pencil Grade

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ABSTRACT

This work presents optimization of concentration parameters for the flotation of Alawa graphite to pencil grade. The graphite samples obtained from Alawa, Rafi local government area of Niger State, Nigeria were crushed, pulverized, coned and quartered to yield a representative sample. The optimum particle size, collector, frother, depressant volume and pH value were determined as 50 µm, 2 ml, 2 ml, 3 ml and 8.5 respectively. Fresh flotation experiment using optimum concentration parameters obtained was examined and this yielded clean graphite concentrate of 95.4% fixed carbon with recovery of 75.2%. Therefore, the result of the study has shown that the recommended grade of graphite for pencil making has been achieved, which requires 95-98% fixed carbon and a maximum size of 50 µm.

1. INTRODUCTION

Graphite, a major additive to many coating systems, is known for its multifarious functions, such as, refractory, lubricant, thermal conductor, electrical conductor, UV shield, electromagnetic pulse shield, corrosion shield and pigment (Indian Mineral Review, 2015). It is also used as moderator in nuclear reactors. Traditional uses of graphite are found in crucibles, foundries, pencils, etc. (Indian Mineral Review, 2015). Graphite is one of the main resources of graphene. Graphene obtained from high-grade graphite usually has good quality and is highly conductive (Jagiello *et al*., 2014). Generally, graphite production from ores is achieved by a combination of careful crushing, milling, screening, tabling, flotation, magnetic separation, electrostatic separation, and leaching (Jean, 1927; Andrews, 1992; Kaya and Canbazoğlu, 2009; SGS Minerals Services, 2012).

Froth flotation is a process used for selectively separating hydrophobic materials from hydrophilic ones. Various factors, such as the type and quantity of chemicals added (Evans *et al*., 1995; Ralston *et al*., 2001), bubble size (Gorain *et al.,* 1995), stator and rotor configuration (Forrester *et al.,* 1998) and residence time (Rubio *et al.,* 2002) influence the performance of a flotation unit. The types and quantity of reagents are the most important part of flotation process. In commercial plants, control of reagent additions is the most important aspect of the flotation strategy (Bulatovic, 2007). Overall separation efficiency in flotation is dependent on surface chemistry factors such as particle bubble attachment, mineral reagent interactions, reagent chemistry etc. as well as hydrodynamic factors which contribute to kinetics of flotation such as agitation, air flow rate, dispersion and cell design (Crossley, 2000). Graphite is naturally hydrophobic, and the most used technique for graphite beneficiation is froth flotation (Arbiter, 1985; Crossley, 2000). Kerosene as a collector, MIBC as a frother, and sodium silicate as a depressant are common reagents used for graphite flotation (Chehreh *et al.*, 2016).

Table 1: Specification of graphite adopted for various industrial purposes

End product	Percentage of graphite used	Fixed Carbon	Size (Microns)
Mag – Carb Refractories	12	87 – 90%	150 – 710
Alumino – carb (graphitized) alumina refractories	8-10	85 min	150 – 500
Clay – bounded crucibles	60 – 65	+ 80%	-200 + 100
Silicon carbide crucible	35	80 – 89%	+ 180
Graphite foils and products based them or (e.g. sealing gashed in refineries, fuel pump automobiles	100	90% min preferably 99%	250 – 1800
Pencil	50 – 60	+ 95 – 98%	50 max
Brake-Lining	1 - 15	98%	75 max
Foundry	–	40 – 70%	53 – 75
Batteries			
(a) Dry cell	-	88% mm	75 max
(b) Alkaline	-	98% mm	5 – 75
Brushes	-	Usually 99%	Usually less than 53
Lubricants	-	98 – 99%	53 – 106
Paint	Up to 75	50 – 55%	Amorphous powder flake
Braid used for sealing (e.g. in ship)	40 – 50	95% mm	-
Graphitized grease (use in seamless steel tube manufacturing)	-	+ 99%	38 max
Recarburisation of steel	100	99%	Micronized
Colloidal graphite	100	99.9%	Colloidal

The expected grade of graphite for pencil production is 95-98% fixed carbon while, size in microns is specified as a maximum of 50 microns (Indian Mineral Review, 2015).

With wide areas of application of graphite and significant opening in the international market (SRG Graphite., 2017), Nigeria will gain much if her graphite resources are properly processed. However, the greatest problem is that Nigeria still imports pencil even though the raw materials (graphite) required is available in the country thereby, necessitating the optimization of concentration parameters for the flotation of Alawa graphite to pencil grade.

2. MATERIALS AND METHODS

2.1. Sample Collection, Equipment and Materials

Representative sample (50 kg) obtained from the study area; Alawa, Rafi local government area of Niger State, Nigeria were used for the test work. The following are the equipment and materials used; Laboratory

Jaw crusher, pulverizer, set of sieves, laboratory sieve shaking machine, weighing balance, furnace, kerosene, sodium silicate, methyl isobutyl carbinol (MIBC), lime

2.2. Methods

Alawa graphite lumps were crushed, pulverized, coned and quartered to yield a representative sample. Proximate analysis was carried out to reveal the percentage fixed carbon by determining the percentage of moisture, volatile matter and ash content using the standard procedures of ASTM C56, 3175, C561 respectively. Elemental composition was determined by the use of X-ray Fluorescence (XRF) machine; Twenty (20) grams of the prepared sample weighed into a sample cup and carefully placed in measuring position on the machine. The machine was switched on, set at 14 and 20kV for major and trace elements respectively. The concentration process adopted was froth flotation, on a 12D Denver laboratory flotation machine. The speed of the machine's impeller was kept constant at 1500 rpm for both conditioning and flotation. The pulps were mixed for three minutes prior to addition of collector (Kerosene) and after an additional mixing of three minutes, the frother (Methyl isobutyl carbinol) was added. After three-minute mixing period, air was introduced into the cell and the froth products were collected for five minutes. Three factors such as; particle size, reagent dosage (frother, depressant and collector), and pH value were properly considered in the research at three different levels.

i. Size fractions (63μm, 50μm, and 45μm)
ii. pH ranges (8, 8.5 and 9). Na_2CO_3 was used to adjust the pH
iii. Dosages of sodium silicate (2, 3 and 4 ml/200 g) as depressant
iv. Dosages of MIBC (2, 3 and 4 ml/200 g) as frother
v. Dosages kerosene (2, 3 and 4 ml/200 g) as collector

3. RESULTS AND DISCUSSION

Results of various laboratory experiments and analysis carried out are presented and discussed in the following sections.

3.1. Characterisation Results

The head sample was obtained by proper mixing of the entire sample from different pits together, and a representative sample taken. It was analyzed and the average Fixed Carbon content of the deposit was found to be 36.21% by proximate analysis (Table 2). Elemental composition was revealed to be 67.14 % Si, 7.036% Fe, Calcium of 4.09%, Aluminum of 4.35% and 3.70% Potassium by XRF (Table 3). The presence of these constituents can be attributed to the bio and geochemical mineralization of the graphite ore. The values of 36.21% FC and 67.14% Si of the mineral indicate that the graphite is of low grade. However, the presence of the aforementioned mineral constituents cannot prevent the utilization of the mineral since there are mineral processing techniques available to beneficiate the material.

Table 2: Result of proximate analysis

	Content %			
Head sample	MC	VM	AS	FC
	14.11	18.26	31.42	36.21

MC: moisture content. VM: volatile matter. AS: ash content. FC: fixed carbon

Table 3: Result of XRF analysis

Element	Al	Si	K	Ca	Ti	V	Cr	Mn	Fe	Mg	Pb	Zn	Ba
%	4.35	67.14	3.70	4.09	0.40	0.38	0.06	0.86	7.03	0.15	0.19	0.42	0.26

3.2. Flotation Experiments

Table 4, shows flotation test results conducted on three sieve sizes of 63 µm, 50 µm and 45 µm. The flotation variables of reagent dosage and pH value were kept constant in each case at 3 ml, and 8.5 respectively. Kerosene (which is the standard reagent for graphite) was used as collector, MIBC as frother and sodium silicate as depressant. Experimental studies focused on investigating the effect of particle size on separation performance. The decrease in particle sizes resulted in increase in percentage grade; this explained the phenomenon that as size reduces materials is liberated. It was observed that the highest fixed carbon (FC) on the concentrates was obtained at 50 µm sieve size as well the highest recovery of 94.13%. The value of 2.17 and 2.04 were obtained as ratio of concentration and enrichment ratio respectively.

Table 5 shows flotation test result obtained by varying collector dosage at 2 ml, 3 ml, and 4 ml. The flotation variables of sieve size, MIBC, sodium silicate and pH value were kept constant through-out at 50 µm, 3 ml, 3 ml and 8.5 respectively. The experimental result showed that the % grade decreased as the amount of dosage was increased. Meanwhile, the optimum dosage of collector was 2 ml, which gave 79.52 g concentrate with % grade of 63.22% FC and a recovery of 74.81%. The value of 2.52 and 1.88 were obtained as ratio of concentration and enrichment ratio respectively.

Table 4: Flotation test results for different sieve sizes

Sieve size (Nominal) (µm)	Feed		Products				Recovery (%)	Ratio of concentration (F/C)	Enrichment ratio (c/f)
			Concentrates		Tailings				
	Wt (g)	FC (%)	Wt (g)	FC (%)	Wt (g)	FC (%)			
63	200	33.60	82.13	65.40	117.87	9.42	79.93	2.41	1.95
50	200	33.60	92.13	68.60	107.87	6.31	94.05	2.17	2.04
45	200	33.60	90.62	67.60	109.38	6.32	91.15	2.21	2.01

Table 5: Flotation test results obtained by varying kerosene as collector

Reagent Dosage (ml)	Feed		Products				Recovery (%)	Ratio of concentration (F/C)	Enrichment ratio (c/f)
			Concentrates		Tailings				
	Wt (g)	FC (%)	Wt (g)	FC (%)	Wt (g)	FC (%)			
2	200	33.60	79.52	63.22	120.48	6.79	74.81	2.52	1.88
3	200	33.60	117.49	55.82	92.51	7.27	97.59	1.70	1.66
4	200	33.60	134.86	38.26	79.14	9.66	76.78	1.48	1.14

Table 6: Flotation test results obtained by varying MIBC as frother

Reagent Dosage (ml)	Feed		Products				Recovery (%)	Ratio of concentration (F/C)	Enrichment ratio (c/f)
			Concentrates		Tailings				
	Wt (g)	FC (%)	Wt (g)	FC (%)	Wt (g)	FC (%)			
2	200	33.60	73.47	79.02	126.53	10.06	86.39	2.72	2.35
3	200	33.60	94.59	61.86	105.41	8.56	87.07	2.11	1.84
4	200	33.60	118.55	55.33	81.45	10.81	97.61	1.69	1.65

Table 6, shows flotation test result obtained by varying the frother as 2 ml, 3 ml, and 4 ml. The flotation variables of sieve size, collector, sodium silicate and pH value were kept constant through-out as 50 µm, 2 ml, 3 ml and 8.5 respectively. Furthermore, the results showed that the highest % grade of 79.02% was obtained at 2 ml, with recovery of 86.39%. The value of 2.72 and 2.35 were obtained as ratio of concentration and enrichment ratio respectively.

Table 7, shows flotation test result obtained by varying the sodium silicate at 2 ml, 3 ml, and 4 ml. The flotation variables of sieve size, collector, MIBC and pH value were kept constant through-out as 50 µm, 2 ml, 2 ml and 8.5 respectively. Following the experimental details, the result showed highest % grade at 3 ml, of 88.86% FC and recovery of 79.48%. The value of 3.33 and 2.65 were obtained as ratio of concentration and enrichment ratio respectively.

Table 8 shows flotation test results obtained by varying the pH value at 8, 8.5, and 9. The flotation variables of sieve size, collector, MIBC and sodium silicate were kept constant through-out as 50 µm, 2 ml, 2 ml and 3 ml respectively. The result at pH of 8.5 gave 62.24 g concentrate with 86.16% FC and a recovery of 79.80%, as the optimum pH value for beneficiating graphite by flotation processes. The value of 3.21 and 1.85 were obtained as ratio of concentration and enrichment ratio respectively.

Table 9 shows flotation test result conducted on combined optimum parameters; sieve size of 50 µm, 2 ml of collector, 2 ml of frother, 3 ml of depressant and pH value of 8.5 determined from experimental work (Table 4-9), these were obtained as a responses of percentage grade and recovery. Therefore, another flotation test conducted gave concentrate of 95.4% FC with recovery of 75.2%.

The two products were further characterized by X-ray diffraction (XRD) and the results are shown in Figures 1 and 2.

Table 7: Flotation test results by varying sodium silicate as depressant

Reagent Dosage (ml)	Feed		Products				Recovery (%)	Ratio of concentration (F/C)	Enrichment ratio (c/f)
			Concentrates		Tailings				
	Wt (g)	FC (%)	Wt (g)	FC %	Wt (g)	FC%			
2	200	33.60	64.67	85.35	135.33	7.55	82.32	3.09	2.54
3	200	33.60	60.11	88.86	139.89	9.39	79.48	3.33	2.65
4	200	33.60	76.92	70.55	123.08	10.51	80.75	2.60	2.09

Table 8: Flotation test results obtained by varying pH value

pH Value	Feed		Products				Recovery (%)	Ratio of concentration (F/C)	Enrichment ratio (c/f)
			Concentrates		Tailings				
	Wt (g)	FC (%)	Wt (g)	FC (%)	Wt (g)	FC (%)			
8	200	33.60	69.12	79.20	130.88	9.47	81.46	2.89	2.06
8.5	200	33.60	62.24	86.16	137.76	9.38	79.80	3.21	1.85
9	200	33.60	70.43	77.85	129.57	8.45	81.59	2.84	2.10

Table 9: Flotation test results obtained from optimum concentration parameters

Optimum parameters					Feed		Concentrate		Tailings		Rec. (%)	RC	ER
Particle size (µm)	Kerosene (ml)	MIBC (ml)	Sodium silicate (ml)	pH	Wt (g)	FC (%)	Wt (g)	FC (%)	Wt (g)	FC (%)			
50	2	2	3	8.5	200	36.21	57.11	95.4	142.9	2.40	75.2	3.5	2.6

Figures 1 and 2 show the XRD results of the products indicating 98% of graphite on the concentrate, which is in agreement with that of proximate analysis result of 95.4%, while, the tailings has 78% of silicate mineral (Quartz). Both results signified the effective flotation process of Alawa graphite with optimum concentration parameters. Therefore, result of the study shown that the recommended grade of graphite for pencil making has been achieved (Indian Mineral Review, 2015).

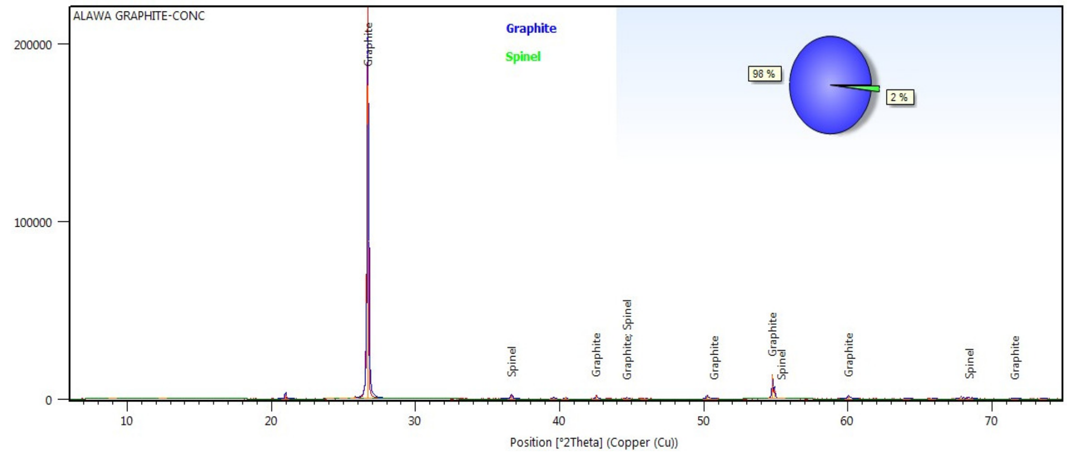

Figure 1: X-Ray diffraction pattern of Alawa Graphite Concentrate

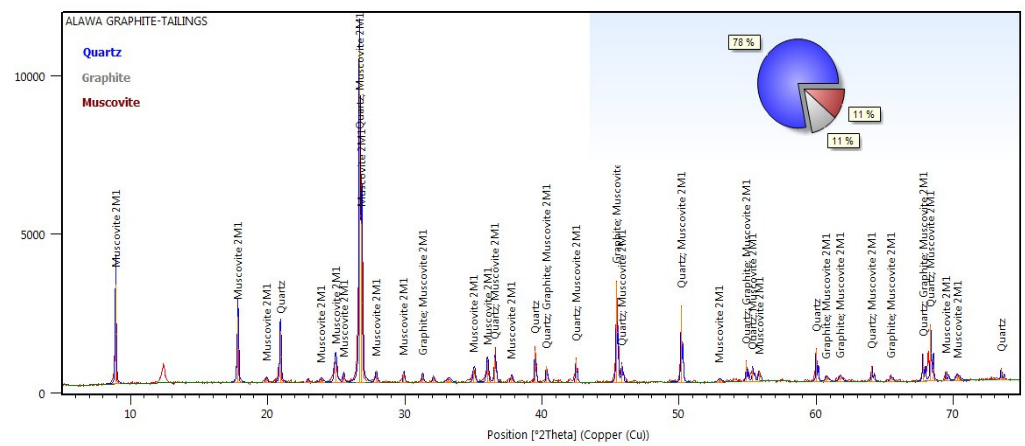

Figure 2: X-Ray diffraction pattern of Alawa Graphite Tailings

4. CONCLUSION

The study evaluated the amenability of the graphite to froth flotation as a concentration method considering three factors (particle size, reagent dosage (frother, depressant and collector) and pH value) at three different levels. The optimum particle size, collector, frother, depressant and pH value were determined as 50 µm, 2 ml, 2 ml, 3 ml and 8.5 respectively. Flotation test, using optimum concentration parameters, this yielded clean graphite concentrate of 95.4% fixed carbon from 36.2% with recovery of 75.2%. Therefore, the result of the study has shown that the recommended grade of graphite for pencil making has been achieved, reported by Indian Mineral Review (2015) to be 95-98% fixed carbon needed and size of 50 µm maximum.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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