

A Comparative Study Between Froth Flotation And Gravity Separation Method To Recover Fossil Resins From Bukan Kwato Coal, Lafia, Nassarawa State, Nigeria

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Abstract

Background: Fossil resins are sticky plants secretions that harden on atmospheric exposure overtime, and has been found within coal and other sedimentary rocks. They are formed tens of millions of years ago through the diagenetic or catagenic transformations of plant exudates induced by various geochemical processes. The recovery of fossil resins from Bukan Kwato using froth flotation method and gravity separation method is the focus of this research.

Results: The two methods were then compared in order to see which has the highest recovery. The obtained samples were analyzed using sieve analysis. It was also analyzed using XRF analysis which reveals that the samples have 2.09% of carbon and 2.72% of sulphur from froth flotation method. It also has 2.08% of carbon and 2.41% of sulphur from gravity separation method.

Conclusively: it was observed that froth flotation method has the highest recovery and is more efficient.

Key words: Coal, Froth Flotation, Gravity Separation, Fossil Resins, Sieve analysis

Background

Bituminous coals are known to contain appreciable quantities of natural fossil or sub-fossil resins. Fossil resins are sticky plants secretions that harden on atmospheric exposure over time, and has been found within coal and other sedimentary rocks. They were formed tens of millions of years ago through the diagenetic or catagenic transformations of plant exudates induced by various geochemical processes [1]. Fossils are remains of plants, animals, fungi, bacteria, and single celled living things that have been replaced by rock material or impression of organism preserved in rock – fossil literally is any preserved remains, impression, or trace of any living thing from a past geological age. Examples include bones, shells, exoskeletons, stone imprints of animals or microbes, objects preserved in amber, hair, petrified, wood, oil, coal and DNA remnants. The totality of fossil is known as the fossil record.

Coal is a sedimentary rock, it usually occurs in coal bed found in coal mines. It comprises of carbon, hydrogen and sulphur. When plants and animal decay, they convert into peat, which is in turn converted into lignite and sub-bituminous coal, and lastly bituminous coal to anthracite.

Froth flotation is a process that selectively separates materials based upon whether they are water repelling (hydrophobic) or have an affinity for water (hydrophilic) [2]. Many researchers have researched on the uses, characterization, features - chemical and physical analysis – of fossil resins, but no emphasis has been made on the recovery of fossil resins on Nigeria coal deposits. On this note, this research seeks to determine the most effective method of recovering fossil resins from the coal in Bukan Kwato, Lafia, Nassarawa state, Nigeria.

The objectives of the research is to determine the mineralogical composition of coal at Bukan kwato coal, Lafia, Nassarawa State, Nigeria using X-ray diffraction (XRD); carry out laboratory analysis; subject equal amount of coal to both gravity and froth flotation method; compare the data gotten from the analysis; and determine the best method for the recovery of fossil resin based on the data.

Literature Review

Pawel et.al., carried out research in an attempt to correlate the physical properties of fossil and sub-fossil resins with their age and geographic location [3]. Testing of the correlation between physi-

cal properties of natural resins such as micro-hardness, density and UV-excited fluorescence emission with their age, geological conditions, botanical and geographical origin and chemical structure was performed. These physical parameters especially micro-hardness, are the result of resins fossilization processes like cross-linking and polymerizations of compounds present in the fossils.

Resins from Herbal Origin and a focus on their application in an article written by Bhanuben et. al., reveals an important role and wide applications played by the resins [4]. Herbal system is considered as a complementary and alternative system of medicine which plays a significant role in India. Resins being the natural products are the secondary metabolites which have therapeutic actions in humans and which can be refined to produce the drugs [4].

Yu Q. et.al. carried out research on the characterization of resin types from the Hiwatha Seam of the Wasatch Plateau Coal field [5]. Fossil resin types occurring in the Hiawatha seam of the Wasatch Plateau coalfield, Utah, have been separated according to color and their physical and chemical properties evaluated. These resin types were separated into four color fractions—yellow, amber, light-brown, and dark-brown. Each resin type was then been subjected to comprehensive spectroscopic examination and other physical methods of characterization, measuring physical properties such as density etc. Magnetic properties were measured among the spectroscopic methods of characterization using FTIR and ¹³C NMR spectroscopy. These analyses reveal that the resins are distinct from other coal macerals, having low aromaticity with small amounts of oxygen functionalities and a low degree of unsaturation. The four resin types show distinct differences in certain physical and chemical properties such as density, hexane solubility, magnetic susceptibility, and lots more.

Fossil Resins Separation Techniques

Froth Flotation

Miller et.al. carried out a project work on the analysis of selective resinite flotation from Wasatch Plateau Coal by pH Control [6]. It has been found that selective resinite flotation from Wasatch Plateau coal can be achieved at high pH. For example, a high-grade resin concentrate (>70% resin) was obtained at an excellent recovery (>90%) by single-stage flotation above pH 12 from a feed containing only 7.2% resin. The rougher concentrate was then cleaned to produce a cleaner concentrate containing >90% resin. The flotation results thus obtained by pH control can be explained to be due to: (1) improved dispersion of the resinite/coal suspension as evidenced by microscopic observation, electro-kinetic behavior, and sedimentation phenomena: and (2) improved depression of coal particles; their hydrophilic state being stabilized by surface precipitation and/or adsorption of calcium species at higher pH values as determined by FTIR and XPS spectroscopic analysis.

Gravity Separation Technique

In Egypt, El-Maghara coal represents a big source of coal with a potential reserve of about 50million tons, which currently used for energy generation. Additionally, this coal contains a substantial amount of resin whose value is much more worthy than the coal as a fuel. The fossil resins, are the most valuable material associated with coal. They represent a large group of invaluable natural resins. In this study, attempts of fossil resin separation from El-Maghara coal by conventional gravity beneficiation methods were conducted on different size fractions of the crushed sample. In this respect sink-float tests, jigging, and shaking table were applied and statistically optimized to obtain different pre-concentrates. The result of separation showed that a noticed increase in resin content from 17% to 24%, and decrease in ash content with high recovery 92.2% could be achieved.

Uses of Resins

(a) Plant Resins

According to Kozowyk (2016), plant resins are valued for the production of varnishes, adhesives, and food glazing agents. They are also prized as raw materials for the synthesis of other organic compounds and provide constituents of incense and perfume. The oldest known use of plant resin comes from the late Middle Stone Age in Southern Africa where it was used as an adhesive for drafting stone tools.

(b) Synthetic Resins

Many materials are produced via the conversion of synthetic resins to solids. Important examples are bisphenol A diglycidyl ether, which is a resin converted to epoxy glue upon the addition of a hardener. Silicones are often prepared from silicone resins via room temperature vulcanization. Other possible uses of resins are as: adhesives, binder in rubber and toys making, slur in Painting, for coatings, gums production, used in the thermoplastic industries. Resins based on coal chemicals in windmill helps produce sustainable energy, it also allows the addition of pigment in bitumen, creating better visibility on cyclinglanes. Heavy-duty flooring lines are made possible due to advancement in resin, as they are also used for pharmaceutical purposes.

Methods

Field Work (Sample Collection)

The field work involved the collection of coal sample from a Lafia coal mine called Bukan Kwato, Nassarawa State, for analysis. The sample was carefully collected randomly from the coal mine, and neatly packed in well-labeled polyethylene bags, and then transported to the laboratory for analysis.

A designed map of the coal mine site was constructed as seen below in Figure 1.

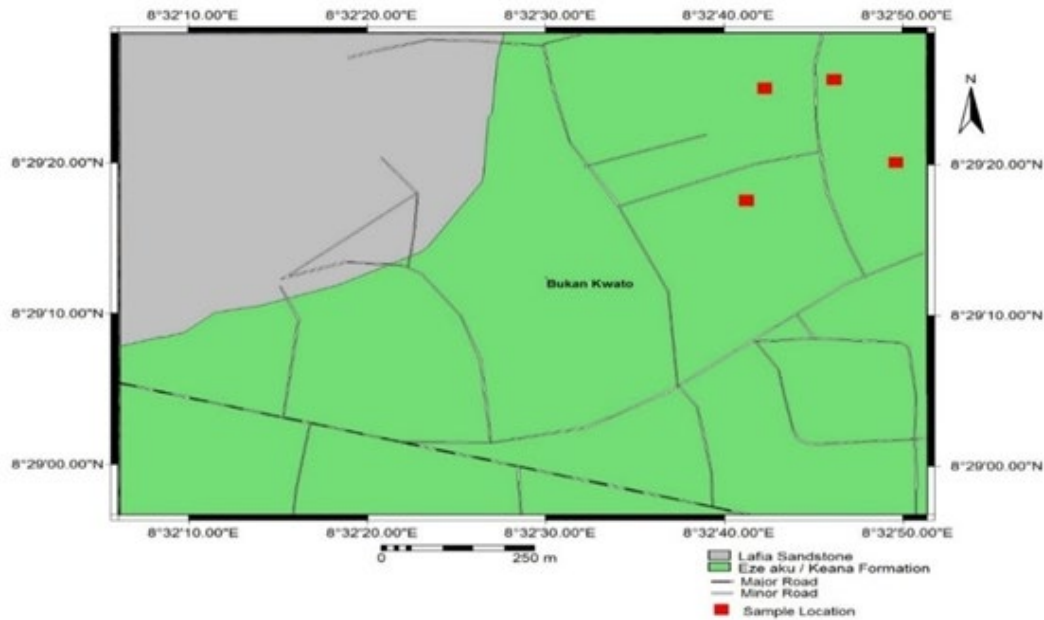


Figure 1: Sample location at Lafia coal mine (Source: NGAS Geological Map, 2019)

Materials/Equipment

The materials used during the research include the following:

- i. Global Positioning System (GPS) (ii) Coal (iii). Jaw Crusher (Laboratory size) (iv). Roll crusher (Laboratory size) (v). Sieve shaker with complete set of sieves (vi). Beam Electronic Weighing Balance (vii). Froth Flotation Machine (Laboratory size) (viii). Pulverizing machine (ix). Reagents (x). Gravity separation machine (Air-float).

Crushing

The coal sample sized 20mm was crushed to smaller sizes of 10mm using the jaw crushing machine i.e., primary crusher.

Sieve Analysis

3000g of the sample material was charged to the set of sieves that

were arranged horizontally, and agitated for about 15mins where the coal was later sieved. Sieve analysis or gradation test is a practice or procedure used to assess the particle size distribution of a granular material allowing the material to pass through a series of sieves of progressively smaller mesh size and weighing the amount of material that is stopped by each sieve as a fraction of the whole mass.

Pulverization and Weighing of coal samples

Part of the sample was pulverized using the Bico-pulverizing machine. The pulverized sample was weighed to 300g using the digital weighing machine. The weighed material as seen in figure 2 below is then used for the froth flotation process.



Figure 2: Digital weighing balance

Froth Flotation

As used in the paper recycling and waste-water treatment industries, this process is used for separating complex minerals. It involves selectively separating hydrophilic minerals from hydrophobic materials. Denver froth flotation machine was used and the steps followed were:

- i. The machine was thoroughly cleaned in order to get an adequate result.
- ii. The coal sample (300g) was divided into three different segments for ease of working and getting more accurate results.
- iii. Water of 1000ml was poured into the flotation cell used, where one segment was poured into the water in the cell and allowed to agitate for 5 minutes.
- iv. Aliphatic hydrocarbon (Kerosene which served as a collector) was added and allowed to agitate for 5 minutes, then the reagent, methyl isobutyl keton (MIBK), was added to serve as a frother which helped to stabilize the flotation for easy collection. Even though without the reagent froth can still be formed however little the quantity and quality, the reagent helps to increase the froth formation and also stabilize it for collection.
- v. The impeller was then lowered into the cell where the mixture of coal and water was poured, allowing it to agitate for 15 minutes.
- vi. Kerosene and methyl isobutyl keton (MIBK) were then added in drops and allowed to agitate again for another 5 minutes before the air was opened and bubbles (froth) were formed which raised

to the cell top. The bubbles (froth) formed was collected using a fetcher and a collector (basin).

vii. The resins overflow was collected in a flat tray.
viii. The process was repeated twice before the entire 300g was exhausted.

ix. After the process was completed, the collected bubbles (froth) were allowed to dry and the residue in the cell allowed to settle down and then decanted. It took two days to complete the drying.

x. The dried sample was then taken for XRF analysis.

The used froth flotation machine and the obtained resins are seen in Figure 3, 4 and 5 below.

Gravity separation method

For the Gravity (Air-float) method, the following procedures were carried out:

- (i) The coal sample was crushed and sieved to a size of 0.071mm.
- (ii) Sieved sample was poured uniformly into the Air-float machine.
- (iii) A hand brush was used to control the travelling speed of the sample, the heavier particles moved upward while the lighter ones moved towards the lowest end of the Air-float table.
- (iv) While this is going on air blower from below supplied a controlled volume of air in an upward flow.
- (v) The lighter materials in the last two outlet were collected and taken for XRF analysis.



Figure 3: Froth flotation machine



Figure 4: Collection of Resins using froth flotation

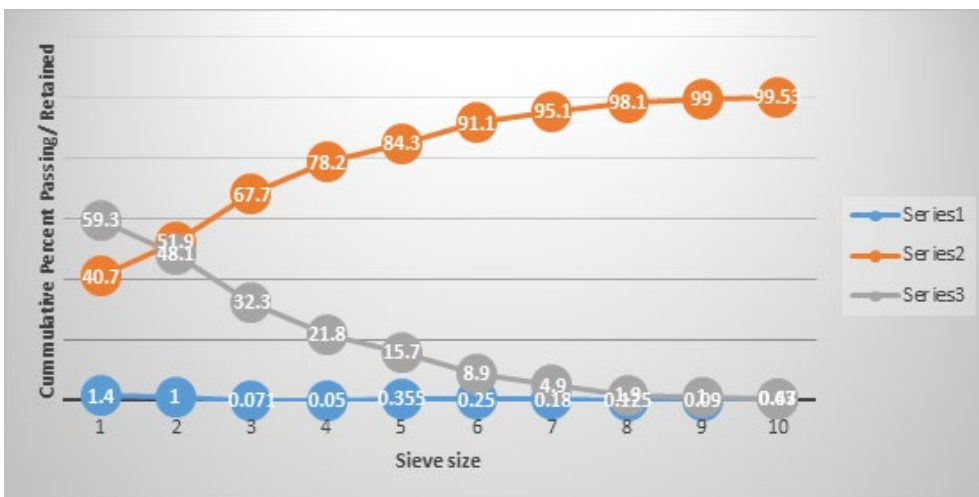


Figure 5: Graph of Cumulative percent passing/retained against Sieve Size Range

Results

The obtained results is shown in the tables 1, 2,3,4 and 5 below:

Table 1.0: Sieve Analysis Result

Sieve size	Weight (g)	Weight or Assay (%)	Cumulative weight retained	Cumulative weight passing
+1.4mm	40.1	40.7	40.7	59.3
1.0	11.0	11.2	51.9	48.1
0.071	15.5	15.8	67.7	32.3
0.05	10.3	10.5	78.2	21.8
0.355	6.0	6.1	84.3	15.7
0.25	6.9	6.8	91.1	8.9
0.180	3.9	4.0	95.1	4.9
0.125	2.9	3.0	98.1	1.9
0.090	0.9	0.92	99	1
0.63	0.5	0.51	99.53	0.47
Pan	0.5	0.51	100	98.3

Table 2.0: Chemical Analysis Result for Run-Off Mine

S/N	Sam- ple	Al ₂ O ₃	SO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	V ₂ O ₅	Cr ₂ O ₅	MnO	Fe ₂ O ₃	NiO
		1.60	5.9	0.36	1.10	0.12	1.20	1.60	0.06	0.17	1.10	35.48	0.04
1	Coal	CuO	ZnO	SrO	ZrO ₂	BaO	Y ₂ O ₃	WO ₃	Au	PbO			
		0.29	ND	0.59	0.14	0.40	0.22	0.08	0.003	ND			

Table 3.0: Results showing the Froth Flotation Process

Sample	Coal Overflow	Coal Underflow	Total
Weight(g)	97.5	140	237.5
% weight	32.5	46.67	79.17

Table 4.0: Results of the froth flotation process

S/N	SAMPLE	Al	Si	S	K	C	Ti	V	Cr
		3.31	15.87	2.72	1.19	2.09	8.53	0.35	0.19
1	Coal A	Mn	Fe	Ni	Cu	Zn	Pb	Mo	Re
		0.16	11.52	0.13	0.39	0.65	2.51	5.93	0.31

Table 5.0: Result from gravity separation process

S/N	SAMPLE	Al	Si	S	K	C	Ti	V	Cr
		4.09	15.30	2.41	1.23	2.08	8.03	0.31	0.21
1	Coal B	Mn	Fe	Ni	Cu	Zn	Pb	Mo	Re
		0.16	11.56	1.09	0.40	0.52	1.11	5.50	0.27

Discussion

The result from froth flotation process as seen in Table 1 below shows the weight of the coal underflow and overflow which was determined with the aid of the weighing balance. Table 2 shows the percentage of iron in the coal obtained from Bukan Kwato, Lafia. It has a retained iron percentage of 35.48%. Table 3 shows the percentage of coal overflow as 32.5% and coal underflow as 46.67%. Table 4 and 5 shows the various percentages of carbon

obtained which is 2.09% obtained after froth flotation process and 2.08% obtained from gravity separation process while Figure 6 clearly shows the cumulative weight percentage passing of particle size 1mm sieve size aperture and this agrees with Will's and Nuhu's theory. It can be said that the liberation size of this mineral is 1mm. Comparatively, all the constituent substances especially sulphur (2.79%), carbon (2.09%) separated from the coal using froth flotation are of higher quantity than that obtained from the

gravity separation process which are Sulphur (2.41%) and Carbon (2.08%). Thus, its safe to say that the froth flotation process would be better utilized in obtaining fuel resins from Bukan Kwato coal using the reagent, methyl isobutyl keton (MIBK) [7].

Conclusions

The recovery of Resins from Bukan Kwato coal in Lafia LGA of Nassarawa state was at a reasonable amount because the chemical analysis shows that an average amount of 15% to 16% of resin was recovered from the coal which is a high and therefore very economical. Also, it was observed that Carbon which is an important element in resin was analyzed at substantial amount of 2.09%. However, Oxygen, Nitrogen and Hydrogen were absent but Sulphur was found present with 2.72%..

List of abbreviations

- (i) MIBK – Methyl Isobutyl Keton
- (ii) XRF – X-ray Florescence Analysis
- (iii) XRD – X-ray Diffraction Analysis

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