

Original Research Paper

# Leaching of a Nigerian Refractory Gold Ore Using Aqua Regia

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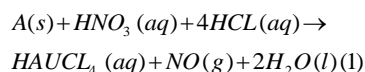
**Abstract:** In this research, refractory gold ore from Imogbara deposit in Iperindo area of Osun state Nigeria were collected systematically from various segments of the deposits at different depths to obtain a set of samples that truly represent the deposits. The 50 kg sample collected was subjected to panning in a flowing river to obtain about 1 kg-upgraded sample. The upgraded sample was air dried for few days, pulverized to pass through 300 µm sized sieve and concentrated using shaking table. About 50 g of the gold concentrate was roasted in a furnace operated at temperature of 850°C with a slightly opened door. Reflected light and thin section microscopic observation of the ore sample was carried out. Samples of As-received and roasted gold concentrate from 80% passing 75 µm were leached in a single stage leaching using 3<sup>2</sup> factorials at temperature of 90°C, with aqua regia prepared from HCl and HNO<sub>3</sub> concentration of 1M, 2M and 4M, leaching time 30, 60 and 90 min. The leaching was done using an oven operated at 90°C and a magnetic stirrer operated at 300 rpm at a temperature of 80°C. Leachates from combinations that gave the best-leached result were analyzed using Atomic Absorption Spectrophotometer. Roasted sample leached with high acid concentration of 4M and low time of leaching on a magnetic stirrer operated at a speed of 300 rpm at temperature of 90°C gave the highest percentage of weight loss of 6.58%. The AAS test carried out on the roasted sample leachate confirms gold concentration of 2.2 g/t.

**Keywords:** Concentrate, Leaching, Panning, Refractory Gold, Roasted

## Introduction

Gold is known to be chemically un-reactive and occurs naturally in the elemental form. When it is found in large nuggets or in river gravel, it can be separated by the simple process of washing off the lighter minerals as done in panning. However, the main gold production comes from large deposits finely and sparsely distributed in hard quartz rock (Cottrel, 1980). The recovery of gold from the lean gold-bearing ores depends largely on the nature of the deposit, the mineralogy of the ore and the distribution of gold in the ore. The recovery process involves gravity concentration, froth flotation and leaching. Gravity methods are used to pre-concentrate heavy gold ore by reducing the associated less dense minerals such as silica, while froth flotation can be used to remove sulphide impurities that may interfere with the leaching recovery of gold. The leaching of gold is commonly carried out with a combination of a complexing leaching reagent and an oxidizing agent (Wills and Napier-Munn, 2006; Cottrel, 1980). In cyanidation, sodium cyanide salt and oxygen are used as complexing leaching reagent and oxidizing agent, respectively. In

mineral acid leaching with aqua regia; a 3:1 Molar mixture of hydrochloric and nitric acid is employed. Though cyanide leaching is the most widely used method to recover gold from its ore, it is considered more harmful than the use of aqua regia as it can lead to emission of the toxic hydrogen cyanide gas and has caused series of environmental accidents. Aqua regia reaction with gold to give tetrachloroaurate (III) complex anion [AuCl<sub>4</sub>]<sup>-</sup> occurs as follows:



Several research works have been reported on the gold deposits of Nigeria. Baba *et al.* (2011) reported the dissolution kinetics of the Ijero-Ekiti gold ore by hydrochloric acid leaching followed by extraction with Tributylphosphate (TBP) in kerosene. The leaching investigation showed that the gold ore dissolution in HCl solution increases with increasing acid concentration, temperature and with decrease in particle size at a stirring

rate of 300 rpm. The kinetics data indicated that the leaching of the ore followed diffusion control mechanism with surface chemical reaction as the rate limiting step. The production and characterization of gold nanoparticles from Itaganmodi gold deposit was reported by O Gundare *et al.* (2014). The production of gold nanoparticles was carried out through mechanical attrition by ball milling and thin film deposition.

However, no successful research has been reported on the recovery of gold metal from the gold deposits of the Nigerian schist belt by the aqua regia leaching route. It has been reported that a typical high grade gold ore attracts a price of only about \$0.92 per ounce as against \$1,272.56 for the value added gold metal. The gold metal price thus translates to over 1383 times that of the high grade ore underscoring the importance of processing raw minerals to metal products to maximize returns (Ebay, 2017). The aim of this study is to establish the optimum parameter required for the effective leaching dissolution of gold concentrate of imogbara refractory gold ore.

## Materials and Methods

### Materials

#### Sample Collection

Gold samples used were collected systematically at various depths from an active goldmine in Imogbara village in Iperindo, Atakumosa local government area of Osun State. The 50 kg sample collected was subjected to panning in a flowing river to obtain about 1 kg-upgraded sample. The panned sample was air dried and there after dry crushed using Pascal Engineering crushing mill with machine number 18862 operated at 415 V, 2200 W, 4.9 A and 50 Hz.

### Methods

#### Sieve Analysis

Exactly 300 g of the crushed ore was measured using coning and quartering selection method and this was used to conduct size distribution test using sieves of sizes 600, 425, 300, 212, 150, 106 75  $\mu\text{m}$  and 53  $\mu\text{m}$  which were selected according to British Standard, BS 1796 (Wills and Napier-Munn, 2006). The sieves were set into column and placed in a mechanical shaker that was shaken for a period of 25 min. Thereafter, the materials on each sieve was weighed and the weight of the sample remained on each sieve was divided by the total weight to give a percentage retained on each sieve (Wills and Napier-Munn, 2006).

#### Gravity Concentration

For a pre-concentration of gold ore, gravity separation by shaking table was carried out in two (2) stages; (a) The first stage of comminuted samples and (b) On a sample of middling resulting from the first stage of tabling.

The dimensions of the shaking table was 75 cm long and 40 cm wide. About 30 g of sieved gold ore (-300  $\mu\text{m}$ ) from different batches was prepared into slurry of about 25% solid by weight (90 mL of water used) and fed into the shaking table. The middling from tabling was re-run by tabling. The parameters used for shaking tabling are; tilt slope angle: 2°; water flow rate: 120 L/hr; stroke length: 1.25 cm; stroke frequency: 26 time/min and Sample feed rate: 250 g/min.

#### Determination of Particle Shape and Size

The particle size and shape of the ore concentrate was determined by optical microscopy using LEICA Galen III Research Microscope with an integrated camera (Celestron digital microscope imager, model 44421). The ore particles was observed with the microscope at X100 magnification. The photomicrographs taken was analyzed using Rasband Image-J software.

#### Thin Section Photomicrography

The CETI light transmission microscope was used to view concentrate samples of the gold ore. A mixture of araldite (resin) and araldite (hardener) were thoroughly mixed together in equal proportion in a square container. Thereafter, 0.5 g of the screened ore was poured into the mixture that has been prepared and mixed together. The whole mixture was then placed on a glass slide having a rectangular shape and then left on a table for about an hour to get hardened. After hardening, a grinding wheel machine was used to thin the sample on the glass slide, while the finishing thinning to the appropriate diameter was done on a lapping/thinning plate which was sprayed with silicon carbide. The sample was heated on a hotplate for about 5 min for drying. After the drying, Canada balsam paste was applied on the sample surface and a cover slip was used to cover the surface for preservation. Thereafter, the prepared slide was viewed under the microscope in the light transmission mode at the magnification of  $\times 100$ . The microscope was adjusted for proper viewing to get the best possible view and the view obtained was taken using a digital camera Vaughan and Craig (1994).

#### Fire Assaying Test

Fire assaying test was carried out using the method described by. A convenient flux was prepared to contain 30 g of  $\text{Na}_2\text{CO}_3$ , 35 g of  $\text{PbO}$ , 4 g of  $\text{SiO}_2$ , 35 g of  $\text{Na}_2\text{B}_4\text{O}_7$ , 1 g of  $\text{CaF}_2$  3.2 g of flour for a 15 g gold ore sample. After all the flux components has been weighed and transferred to the crucibles, silver wire was added. Then, the entire concentrate sample was spread out on a piece of paper and 'leveled off' with a long spatula to a thickness of about 1 cm. to ensure a representative sample, small portions were taken with a spatula from various points along the edges and middle and transferred into the weighing pan until 15 g

were obtained. The 15 g sample was transferred to the crucible and the content was mixed thoroughly with a wire type mixer until a uniform colored mixture was obtained. The fusion was carried out by first pre-heating the furnace to 1000°C after which the crucibles with their contents were placed in the furnace. Thereafter, the furnace door was closed and the temperature was turned down to 900°C and the timer set for 17 min. At the end of 17 min the temperature was raised to 1000°C and the timer set for an additional 15 min. At the end of fusion, the crucibles were removed singly using crucible-scorifier tongs and the melt was swirled and poured into prepared iron mold. After cooling, the slag of the cooled melts was broken with a steel rod and hammer. The button was then transferred to a 50 mL beaker and 10% HCl was added to about 0.5 cm over the button. The beaker was covered with a watch glass, placed on the edge of a hot plate and content was allowed to digest for about 1-2 h to remove the last traces of slag. The button was rinsed and brushed under running water and shaped into a cube with a hammer and anvil. The lead button obtained from the crucible fusion was treated by a process called cupellation to separate the noble metals from the lead. This consists of an oxidizing fusion in a porous vessel made of compressed bone ash called cupel. A cupel of 3.8 cm diameter was used. The cupels was placed in a cold furnace and temperature set at 800°C. At this temperature, a shielding scorifier loaded with graphite electrodes was placed in front of the cupels. This assisted in reducing the otherwise highly oxidizing atmosphere so that oxidation proceeded at a desirable rate. Following this, the furnace door was closed and the temperature raised to 1000°C. When this temperature was attained, the buttons were transferred to the sample cupels using button tongs. The door was closed for a few minutes then opened to see that the buttons were ‘open’ (lead buttons become molten and red throughout). At this point, the furnace door was kept opened and temperature allowed to decrease rapidly to 820°C. This temperature was maintained for 10 min and then raised to 840°C holding for another 10 min, this was repeated at temperatures 860°C and 880°C respectively. Following this, the sample cupels with beads was removed from the furnace and placed on the steel table. The bead was thereafter dissolved with 2 cm<sup>3</sup> aqua regia. The gold content was determined using atomic absorption spectrophotometer with a graphite furnace, auto sampler and printer.

### Leaching Dissolution of Gold Metal

In the single stage leaching, Ore samples was leached using 3<sup>2</sup> factorial at temperature of 90°C, HCl and HNO<sub>3</sub> concentration of 1M, 2M and 4M, leaching time 30 min, 60 min and 90 min. using an oven, a magnetic stirrer and a stirred batch reactor. The aqua regia for each concentration level was prepared by mixing 3:1 of HCl and HNO<sub>3</sub>. The oven leaching was carried out using 5 g of gold ore sample in each case. The sample was measured into a conical flask of 100 mL capacity while 25 mL of the prepared aqua-regia was added. The conical flask was carefully covered with an aluminum foil and the mixture was thoroughly shaken for 3 min to ensure homogeneity. The flask was placed in an oven preset to the leaching temperature of 90°C. The leaching was done for a period of 30, 60 and 90 min for different samples of each level of aqua regia concentration. On completion of leaching time, each reaction flask was brought out of the oven and content was allowed to cool down before filtering using Whatman filter paper. This procedure will be repeated for each combination level. Most effective condition for digestion will be identified based on weight loss and AAS measurement of total gold in solution (Baba *et al.*, 2011). Another set of leaching was carried out using a magnetic stirrer. The procedure was designed using 3<sup>2</sup> factorial. Aqua-regia concentration and time of leaching was maintained as in the case of oven leaching described earlier. Also leaching temperature was 90°C and stirring rate of 300 rpm was utilized for these set of leaching. In each level, 5 g of the ore sample was measured into a 100 mL conical flask and 5 mL of prepared aqua regia was added to the flask. The flask was covered with aluminum foil and shaken for 3 min. The stirrer was dropped into the mixture and flask was placed on the magnetic stirrer plate and temperature set to 90°C. Once the leaching time was completed, the mixture was allowed to cool down and filtered out using Whatmann filter paper. The residue was air dried and latter oven dried to a constant temperature. Weight of residue was taken using an electric weighing balance and weight difference was calculated. Each leaching level was done in duplicate and average weight difference was recorded. This procedure was repeated for all the various levels. AAS analysis of the best leached filtrate was carried out to estimate the total gold extracted. The kinetic of the reaction was monitored by conductivity and pH probes. The test was further carried out in multistage according to Adeleke *et al.* (2011; 2014). The procedure was repeated with the gold concentrate as-roasted.

**Table 1:** High Gravity Atomization Conditions for the Determination of Gold

Step number	Furnace temperature/°C	Time ramp/s	Hold time/s	Internal gas flow cm <sup>3</sup> /min
1	120	10	10	300
2	1000	10	25	300
3	2200	0	3	0
4	2650	1	3	300

### Gold Determination Using Atomic Absorption Spectrophotometer

Total gold was determined using Perkin Elmer 1100B AAS with a graphite furnace, HGA (high graphite atomization) 700. An autosampler AS-70 was used to charged gold solution sample into the tube. A calibration curve was plotted using standard gold solutions 200, 400, 600, 800 and 1000 ng Au/cm<sup>3</sup> prepared using a nickel matrix modifier in an argon current. An instrumental conditions of wavelength 242.8 nm, slit 0.7 nm; lamp current 10 mA; integration time 5 s was selected and used for the analysis. Gold was then determined in each standard solution under the condition presented in Table 1 and a calibration curve was obtained.

## Results and Discussion

### Size Distribution

The size distribution of the crushed ore is presented in Table 2. The cumulative distribution of the particles is as shown in Fig. 1. About 86% of the pulverized ore has particle size above 75 µm. This indicate that much energy will be required in bringing down the ore sample to 80% passing 75 µm required for this studies.

### Gravity Concentration Using Shaking Table

The tabling result is presented in the Table 3 and 4; More than 30% of the of the concentrated ore falls into the concentrate division during the shaking tabling separation test.

**Table 2:** Particle size distribution of as-received sample

S/N	Sieve size (µm)	Retained weight (g)	Cumulative oversized (g)	Percentage retained (%)	Cumulative percentage oversized (%)
1	X>600	5.30	5.30	1.77	1.77
2	600>X>425	24.50	29.80	8.17	9.94
3	425>X>300	23.90	53.70	7.97	17.91
4	300>X>212	62.40	116.10	20.80	38.71
5	212>X>150	52.60	168.70	17.53	56.24
6	150>X>106	45.30	214.00	15.10	71.34
7	106>X>75	46.70	260.70	15.57	86.91
8	75>X>53	34.20	294.90	11.40	98.31
9	X<53	5.10	300.00	1.70	100.00

**Table 3:** Stage 1 Tabling Products for –300 MM Fraction.

Sample	Weight %
Concentrate 1	32.7
Mildling 1	45.3
Tailings 1	22.0

**Table 4:** Stage 2 tabling products for middling obtained from stage 1

Sample	Weight %
Concentrate 2.1	3.5
Concentrate 2.2	13.2
Mildling 2.1	60.8
Tailings 2.1	22.5

### Reflected Light Microscopy

Micrographs of as-received and roasted concentrate samples viewed under reflected light microscope, are presented in Fig. 2 and 3 respectively. The gold particle is indicated by arrow 1. In the as received sample, the gold particles are locked up by Pyrite indicated by arrow 3. The silica gangue present in the ore is indicated by arrow 4 while arrow 2 indicate porosity. In the roasted sample, the gold particles are relatively unlocked and its indicated by arrow 1. The silica gangue and the porous sections are indicated by arrows 3 and 2 respectively.

### Thin Section Photomicrographs

The light transmission microscopy view of thin section slides prepared for both the as-received and calcined sample of the ore are presented in Fig. 4 to 6. Gold particles could be seen on both plates but more prominent on the calcined sample.

### Fire Assay Analysis

The sample structure was effectively destroyed through the fire assay that was carried out on the sample and this brought about a complete separation of the silver and gold from the gangue associated with the ore. The time required for the whole process from smelting to dissolution of silver gold bead was less than 3 h which is relatively shorter compared to extensive hours that will be required for acidic dissolution of equivalent sample Leaching with Aqua Regia Table 5 to 10.

**Table 5:** Oven leaching of as-received concentrates with aqua-regia

Combination	Aqua regia concentration	Temp.	Time	Initial mass (g)	Final mass (g)	Weight loss	Percentage weight loss
t <sub>H</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	90 min	5	4.764	0.236	4.72
t <sub>H</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	90 min	5	4.893	0.107	2.14
t <sub>H</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	90 min	5	4.753	0.247	4.94
t <sub>m</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	60 min	5	4.761	0.239	4.78
t <sub>m</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	60 min	5	4.802	0.198	3.96
t <sub>m</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	60 min	5	4.843	0.157	3.14
t <sub>l</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	30 min	5	4.746	0.254	5.08
t <sub>l</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	30 min	5	4.794	0.206	4.12
t <sub>l</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	30 min	5	4.770	0.23	4.60

**Table 6:** Oven leaching of roasted gold concentrates with aqua-regia

Combination	Aqua regia concentration	Temp.	Time	Initial mass (g)	Final mass	Weight loss	Percentage weightloss
t <sub>H</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	90 min	5	4.777	0.223	4.46
t <sub>H</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	90 min	5	4.894	0.106	2.12
t <sub>H</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	90 min	5	4.725	0.275	5.50
t <sub>m</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	60 min	5	4.861	0.139	2.78
t <sub>m</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	60 min	5	4.874	0.126	2.52
t <sub>m</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	60 min	5	4.858	0.142	2.84
t <sub>l</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	30 min	5	4.695	0.305	6.10
t <sub>l</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	30 min	5	4.872	0.128	2.56
t <sub>l</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	30 min	5	4.851	0.149	2.98

**Table 7:** Magnetic stirrer leaching of as received samples with aqua-regia

Combination	Aqua regia concentration	Temp.	Time	Initial mass (g)	Final mass (g)	Weight loss	Percentage weight loss
t <sub>H</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	90 min	5	4.695	0.305	6.1
t <sub>H</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	90 min	5	4.832	0.168	3.36
t <sub>H</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	90 min	5	4.765	0.235	4.7
t <sub>m</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	60 min	5	4.715	0.285	5.7
t <sub>m</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	60 min	5	4.755	0.245	4.9
t <sub>m</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	60 min	5	4.793	0.207	4.14
t <sub>l</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	30 min	5	4.685	0.315	6.3
t <sub>l</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	30 min	5	4.707	0.293	5.86
t <sub>l</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	30 min	5	4.787	0.213	4.26

**Table 8:** Magnetic stirrer leaching of roasted samples with aqua-regia

Combination	Aqua Regia Concentration	Temp.	Time	Initial mass (g)	Final mass (g)	Weight loss	Percentage weight loss
t <sub>H</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	90 min	5	4.715	0.285	5.7
t <sub>H</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	90 min	5	4.811	0.189	3.78
t <sub>H</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	90 min	5	4.735	0.265	5.3
t <sub>m</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	60 min	5	4.693	0.307	6.14
t <sub>m</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	60 min	5	4.724	0.276	5.52
t <sub>m</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	60 min	5	4.781	0.219	4.38
t <sub>l</sub> C <sub>H</sub>	4M HCl + 4M HNO <sub>3</sub>	90°C	30 min	5	4.671	0.329	6.58
t <sub>l</sub> C <sub>m</sub>	2M HCl + 2M HNO <sub>3</sub>	90°C	30 min	5	4.727	0.273	5.46
t <sub>l</sub> C <sub>l</sub>	1M HCl + 1M HNO <sub>3</sub>	90°C	30 min	5	4.756	0.244	4.88

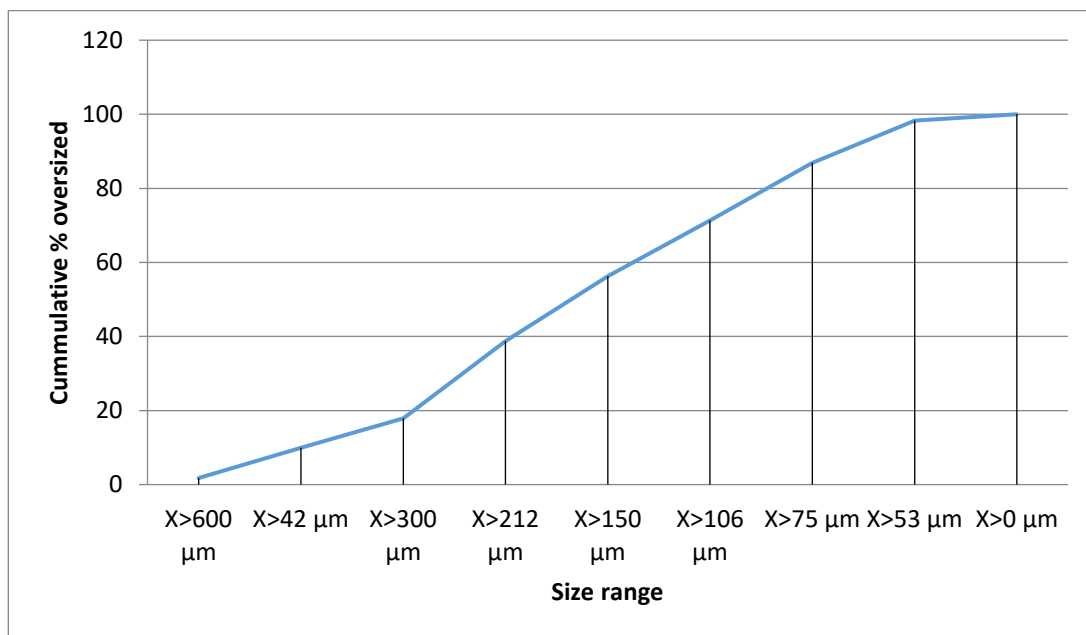
Note: t<sub>H</sub>, t<sub>m</sub> and t<sub>l</sub> means high, medium and low time respectively while C<sub>H</sub>, C<sub>m</sub> and C<sub>l</sub> means high, medium and low concentrations respectively

**Table 9:** Total gold content in as received and roasted concentrate fire assay buttons

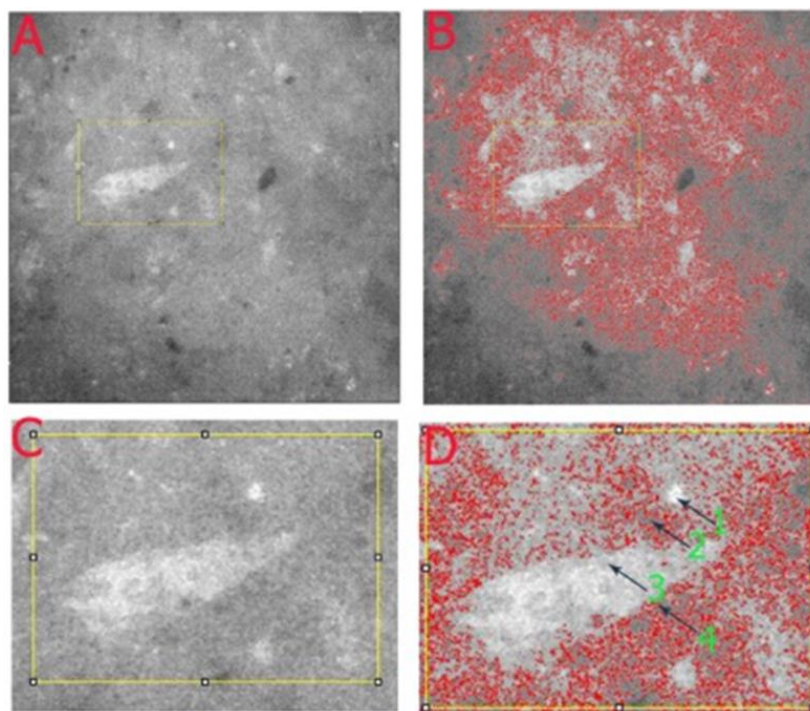
Sample	Sample weight (g)	Absorbance A	Gold g/t
Concentrate	15	14.4	2.4
Roasted concentrate	15	17.4	2.9

**Table 10: Gold Contents in Leachates of Best Leached**

Sample	Sample weight (g)	Absorbance A	Gold g/t
Roasted concentrate leached on magnetic stirrer	5	13.2	2.20
Received concentrate leached on magnetic stirrer	5	11.4	1.90
Roasted concentrate leached in an oven	5	11.1	1.85
Received concentrate leached in an oven	5	9.9	1.65

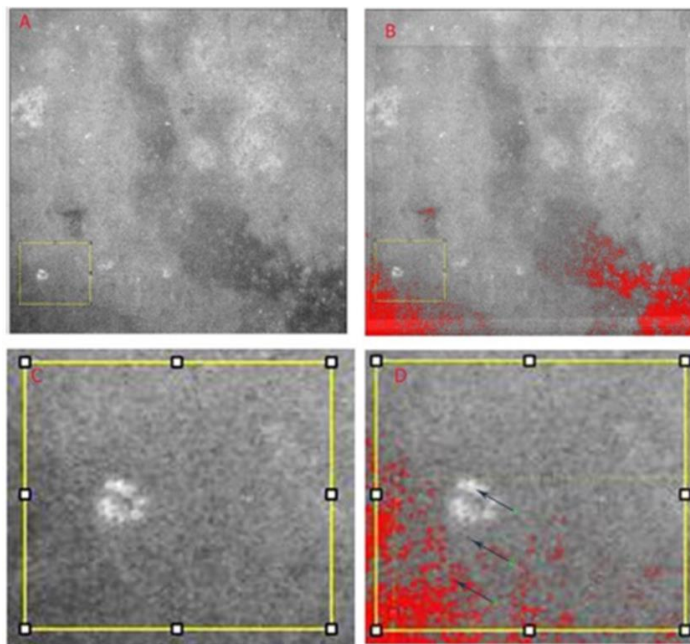


**Fig.1:** Cumulative size distribution curve

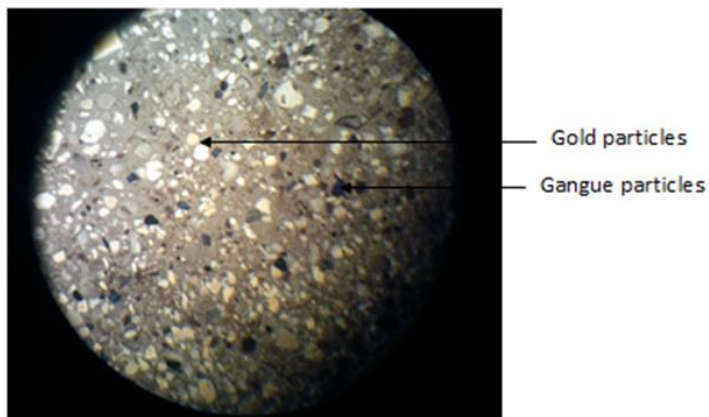


**Fig.2:** Reflected light micrographs of concentrate 80% passing 75 μm

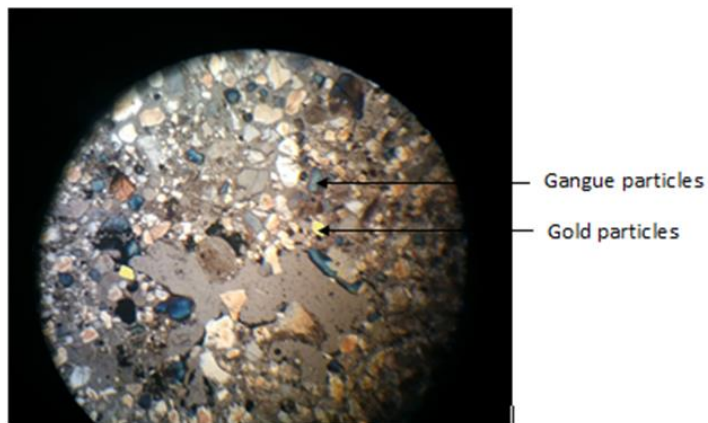




**Fig. 3:** Reflected light micrograph of roasted concentrate sample 80% passing <math><75 \mu\text{m}</math>



**Fig. 4:** Thin section micrograph of as Received sample



**Fig. 5:** Thin section micrograph of roasted sample

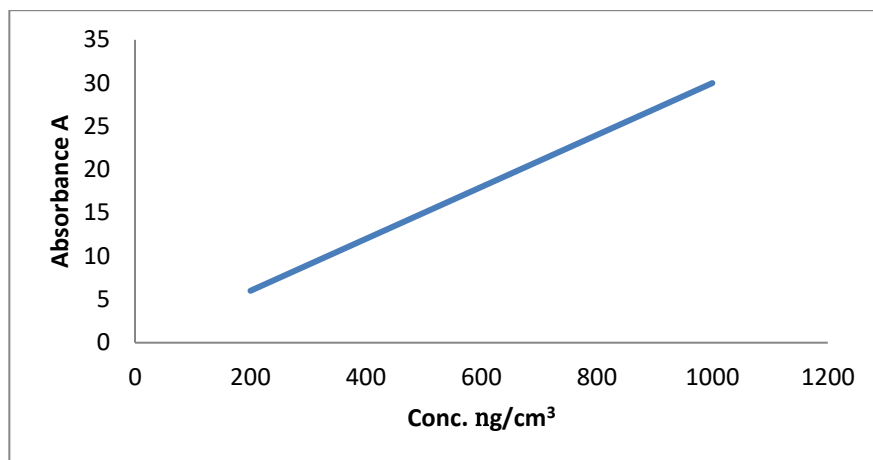


Fig. 6: Calibration curve for the total gold using a graphite furnace

## Conclusion

The mineralogical examination of concentrate of this Nigeria refractory gold ore using reflected light microscopy reveals the encapsulation of gold particles by pyrite. However, roasting procedure was able to liberate the gold particle to a significant level. Aqua regia leaching of roasted samples of the ore using a magnetic stirrer with moderately high acid concentration gave a high gold dissolution.

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## Author's Contributions

**Oluwasanmi Samuel Teniola:** Carried out the laboratory experiments and contributed to the writing of the manuscript

**Abraham A. Adeleke:** Designed the research plan and organized the study

**Simeon Ademola Ibitoye:** Involved in the reviewing of the manuscript

**Moshood Deinde Shitu:** Sourced for the gold ore and contributed to the writing of the manuscript.

## Ethics

The author of this manuscript conceived the idea and were involved in all the laboratory work

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