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Extraction of gold from Calabar gold dust

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Abstract
A statistically sampled ore from a recently discovered surface gold dust deposit in Calabar, Cross-River State, Nigeria was collected and subjected to analysis via precipitation method of hydrometallurgical route for the recovery of gold. Beneficiation process of gravity concentration and leaching (solvent extraction) using aqua regia with Hf of 1:3 for solubilization of gold was carried out followed by precipitation (reduction) of gold with sulphur dioxide. The analysis of sample using quantification showed that the percentage composition of impure Gold (Au) is 35% with Fe_3O_4 as the major impurity which was removed through specialized washing. A 30% recovery of Au from the ore was finally obtained.

1. Introduction

The metal gold, a yellow, dense and ductile, appearing in subgroup 1B of the periodic table is geologically widely distributed but sparsely concentrated in the earth's crust. Its geographical distribution is highly limited[1,2]. Gold exists in combined state with the major elements like lead, iron, magnesium, silver, copper and other trace elements that constitute the ore. Gold is highly used in jewelry (carat value) and coinage (bullion). The research work is aimed at recovering gold from a recently discovered ore called gold dust which in nature occurs at Calabar, Cross River State, Nigeria. There are three methods of extraction and refining of gold, viz – amalgamation, cyanidation and precipitation. Recovery of gold involves benefication process embracing physical separation, followed by separating the chemically bonded elements through decomposition of the ore mineral and also through chemical reactions.

Basically, hydrometallurgical process involving three unit processes of leaching, extraction and precipitation was used.

Gold is usually associated with silver in proportions which may vary between about 1:10,000 in some silver-lead ores and 10:1 in some Australian gold ores. Gold is also present in se water to the extent of about 0.004ppb. Estimates from gold found in meteorites shows that gold concentration in the earth as a whole is about 0.25ppm. Worthwhile ores contain 5-30ppm. Extraction can also come from the ores of lower concentration. Nevertheless, gold as a precious metal has two distinct characteristics-Constant quantity and relative scarcity[3,4,5]. Micro composition of the ore is important when selecting the most appropriate gold recovery technique[6,7]. In refining precious metal and some concentrates, the gold is converted to the chloride by treatment with aqua regia.

$$Au+3HNO_3 + 4HCl = HAuCl_4 + 3NO_3 + 3H_20.$$

After heating to remove nitrogen oxides, gold is precipitated from this solution by reduction with sulphur dioxide or ferrous sulphate. Any platinum metals can be recovered from this solution after the complete precipitation of the gold [8].

2. Experimental

2.1. Materials/equipment

Gold dust ore, aqua regia (1 part nitric acid and 3 parts hydrochloric acid for solvent extraction), sodium disulphide ($NaS_2S_2O_3$) – for generation of sulphur dioxide, sulphuric acid for producing gold sulphate, measuring cylinder, conical flask and beakers, beam balance weighing and high temperature crucibles, metal/wire guage, gas burner, heating furnances, watchman number one filter paper, sample container, AAS.

2.2. Methods

Table 1
The result of the A.S.S analysis

Element	Percentage (%)
Zn	2.1
Mn	6.3
Mg	1.8
Cu	0.04
Fe	12.06
Ca	21.07
Pb	Not detected
Ag	Not detected
Au	Lamp

A statistically sampled ore from the virgin deposit was critically analysed to determine the concentration of some elements like Ca, Mg, Zn, Fe, Mn, Cu and Pb and subsequently subjected to a series of communition, screening and separation operations. Lump ore was commissioned with screening operation [7] and solvent extraction using aqua regia after gravity concentration was carried out.

2.3. Precipitation method of gold extraction

Weigh a given sample of pulverized gold dust into 250ml conical flask and measure 20ml of aqua regia into the flask. Then apply heat till the flask content reach near dryness and bring the flask down and allow to cool. Repeat the process and on heating to near dryness introduce 20ml 1:3; HF aqua regia mixture and carry out the process again for complete solubulation.

2.4. Leaching equation

$$Au + 3HNO_3 + 4HCl \rightarrow HAuCl_4 + 3NO_2 + 3H_2O$$

After treatment of the ore with aqua regia, the gold is converted to chloride as in the leaching equation above. Make-up the mixture with about 20 to 20 ml of distilled water. Agitate the result mixture for complete desolution and filter into the beaker. It is worthy to note that the mixture should be heated until the evolution of brownish nitrogenous gas ceases. After the reheating in the presence of nitrogen oxides, gold is precipitated from the solution by reduction with sulfur dioxide.

3. Results and discussion

The result of the analysis using, AAS along with the standard solution at FIIRO is as shown in table 1.

The rest 56.23% to make up 100% including Au could not be determined due to unavailability of Gold lamp at the institute.

The concentration of impure gold in the ore was determined mathematically from the data obtained from the extraction experiment. This impure gold powder contained high concentration of Fe_2SO_4. As Fe^{3+} constitutes major contaminant, washing process was used to remove the Fe_35O_4 in the impure Au powder. Result after washing is shown in table 2. A 30.125% Au was detected using quantification method. From the results obtained, we observed some difficulties and most paramount was poor precipitation of the pure gold. Though, the precipitation method was efficient to some degree, since the suspected precipitated gold particles were noticed. The general reducing sulphur dioxide for gold precipitation was computed.

Table 2
Result after washing

No. of washing	Purer Au power after washing	%Concentration of the purer Au powder
1st washing	2.65g	33.125%
2nd washing	2.51g	31.375%
3rd washing	2.41g	30.125%

Finally the result of poor precipitation of suspected pure gold, volatilization process was used in the recovery of the gold complexes in the solution. A quantification method was used to establish the percentage of Au composition to about 30.125%.

4. Conclusion

This recovery path with great potential savings in energy and cost implication with relative environmentally-friendly operation is really attractive.

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