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Successful separation and detection of gold sulphide in ore samples from the banded iron formation of Goa, India

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Gold (I) sulphide (Au₂S) is an inorganic compound and the principal sulphide of gold. This study aimed to separate gold sulphide from ore samples such as banded hematite quartzite mined from the banded iron formation of Goa, India. The samples were dried, powdered and then concentrated by the panning method. The coarse ferromagnetic material was separated and sieved into different fractions of varying sieve sizes, i.e. 250, 150, 106 and 53 µm. Next, they were subjected to serial washing, and after drying, were tested for the presence of gold by ICP-AES, optical microscopy, phase contrast microscopy, SEM-EDS and total content of the sulphur was analysed by CHNS/O elemental analyzer.

Keywords: Banded iron formation, detection, gold sulphides, ore samples, separation.

GOA forms a part of the northwestern Western Dharwar Craton (WDC), which serves Asia's major metallogenic province¹. The iron ores of Goa are associated with greenstone and occur as banded hematite quartzite (BHQ) and banded magnetite quartzite (BMQ)². Previous research findings show the presence of gold in the WDC and parts of the Konkan region, India. Reports are available on the occurrence of gold (0.06–0.16 ppm) in lateritic powdery ore of Keri and Kalne villages and <0.1 ppm gold in laterite and iron ores in Sindhudurg district, Maharashtra, India³. Studies have established that BHQ in Goa, India, contains 12–13 ppm of gold⁴. The occurrence of gold in sulphidic banded iron formations (BIFs) ranged from 0.7 to 3.2 ppm (ref. 5). Gold(I) sulphide having the formula Au₂S is an inorganic compound and principal sulphide of gold. Gold usually occurs in its native form in the lattice of sulphides (especially arsenopyrite, pyrite and chalcopyrite), carbonate, silicate and oxide minerals. For sustainable development of the gold industry, recovery of gold from refractory sulphide gold ores is important. However, there are many challenges in the treatment of sulphide refractory gold ores. The incorporation of gold into sulphide minerals has long been inferred by several investigators⁶. In this study, a novel method for gold sulphide recovery from iron ore samples in the BIF of Goa has been successfully used with prospects of bio-mining of gold as a long-term objective.

A significant part of the primary gold reserves of the world is contained in sulphide ores, many types of which

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Table 1. Distribution of gold sulphides in the world

Type	Occurrence	Country
Primary gold sulphide deposits	Carboniferous strata of the Arctic zone	Russia ¹⁰
Arsenical gold sulphide	–	Greece ¹¹
Refractory gold sulphide	–	Argentina ¹²
Primary gold sulphide deposits	Migori granite–greenstone complex – Archean rocks	Migori gold belt, Kenya ¹³
Gold, pyrite and arsenopyrite	Birimian greenstone belt of Boromo–Goren (lower Proterozoic)	Burkina Faso ¹⁴
Gold, pyrite and arsenopyrite	Granite–greenstone belts	Kaapvaal Craton, South Africa ¹⁵
Auriferous pyrite and arsenopyrite	Lower Carboniferous black-shale volcanic carbonate–terrigenous sequences	Kazakhstan ¹⁶
Gold-bearing quartz deposits and stratiform-type gold sulphide deposits	Archean South Kolar schist belt	India ¹⁷

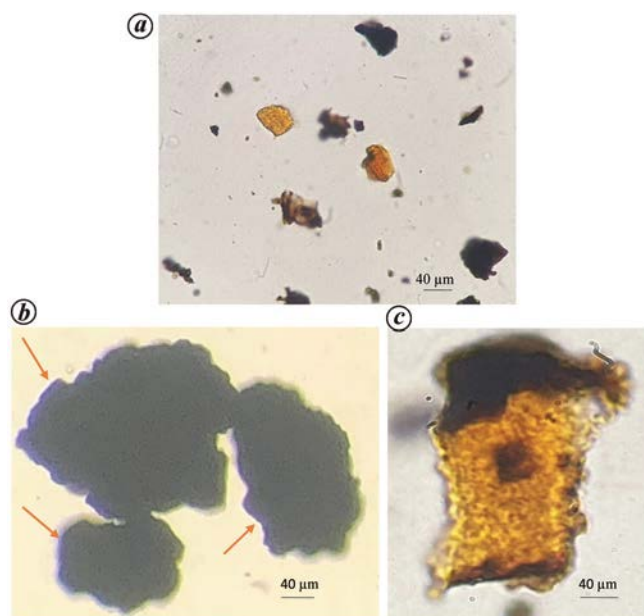


Figure 1. Bright-field microscopy of heavy fraction of banded hematite quartzite. *a*, Composite view of the heavy fraction components. *b*, Presumptive gold sulphide particles showing surface plasmon resonance. *c*, Gold sulphide particles attached to a gold particle.

are refractory in gold processing. Deposits of refractory sulphide ore will be the main source of gold production in the future. Various mechanisms have been proposed for the precipitation of gold in sulphides⁷. A study based on chargeability anomaly, field observations and mineralogy indicates the presence of gold-bearing sulphide mineralization⁸. Auriferous sulphide ores contain very finely disseminated gold and other precious metal particles inside the sulphide⁹. Table 1 shows the occurrence of gold sulphide and its reported global distribution^{10–17}.

Fifty grams of BHQ sample from Velguem, North Goa⁴ was dried and powdered using a mortar and pestle¹⁸. Panning method was used to concentrate the recovered sample¹⁹, which was subjected to magnetic separation using 2 T neodymium magnet²⁰ and sieved into different fractions with a sieve size of 250, 150, 106 and 53 μm . Only the 106 μm sieve size sample (10 g) was used for inductively coupled plasma atomic emission spectroscopy (ICP-AES).

ICP-AES is an analytical technique used for the detection of chemical elements. It is a plasma technique with flame temperature in the range 6000–10,000 K. Quantification of gold was carried out by ICP-AES method (Cera Laboratories, Mumbai) using Agilent 5110 ICP-AES. Ten grams of the completely dried sample at 105°C with 106 μm size sieved fraction was sent for analysis¹⁸.

BHQ samples with known gold concentration were used for sulphide detection. Ten grams of dry BHQ sample was powdered and sieved through a 106 μm sieve. The 106 μm fraction was subjected to magnetic separation using 2 T neodymium magnet²⁰. The coarse ferromagnetic materials were eliminated by multiple washing using water by removing the lighter and soluble fractions. Heavier insoluble fraction (I) was subjected to magnetic separation to remove fine ferromagnetic particles, which had escaped previous treatment. Later, the heavier fraction (II) was dried and tested for sulphur by Lassaigne's and lead acetate tests²¹. Samples were prepared for optical and phase contrast microscopic studies and used to detect gold sulphide by scanning electron microscopy (SEM-EDS) and total sulphur content by elemental analysis²².

The 106 μm sieve size, dried heavy fraction was used to prepare slides by mounting the material directly in the DPX mountant. The slides were examined to detect components of heavy fraction such as gold particles, auriferous quartz and presumptive black gold sulphidic material using optical microscopy (Nikon Eclipse E200 MVR, Tokyo, Japan) and phase contrast microscopy (Olympus BX41).

A small sample of heavy fraction with presumptive gold sulphide was mounted on a carbon tape and analyzed using SEM-EDX technique to examine the surface microstructure¹⁹ (Carl-Zeiss SEM USIC, Goa University). The images were obtained under operational conditions 15–35 kV (ref. 23).

Total elemental sulphur analysis was carried out on 1 g of heavy fraction using CHNS/O Elemental Analyzer Perkin Elmer PE 2400 Series II. This works on the frontal chromatography technique for separating and estimating elements such as C, H, N, S and O in a sample. The analysis was carried out at the Sophisticated Instrumentation Centre for Applied Research and Testing (SICART), Gujarat, India.

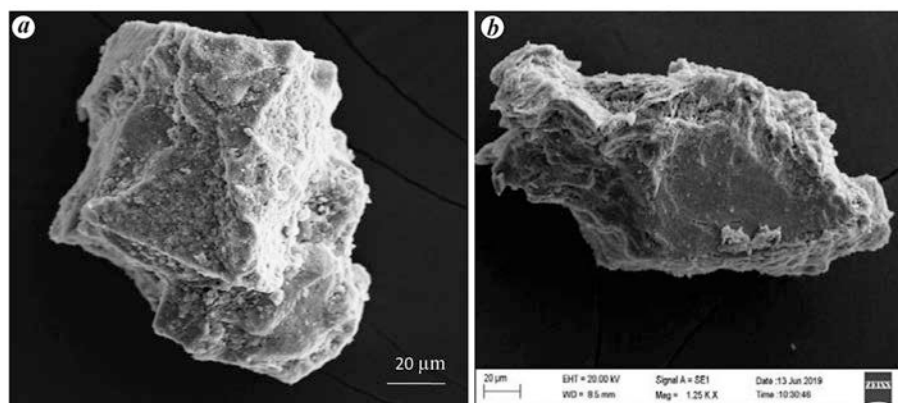


Figure 2 a, b. Secondary electron images of gold sulphide grains showing irregular and rough surface.

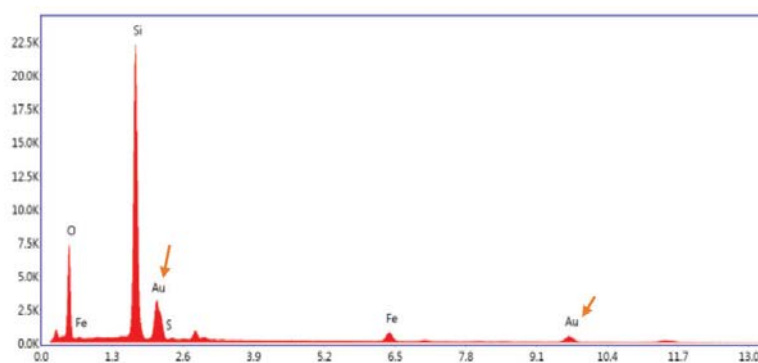


Figure 3. SEM-EDS spectrum of heavy fraction with presumptive gold sulphide grains. The peaks indicating presence of elemental gold and sulphur can be seen.

This study presents the detection and separation of gold sulphide in the heavy fraction of BHQ sourced from the BIF in Goa. Similar findings have been reported in Brazilian²⁰ and Australian²⁴ mines. In our previous studies, we have reported the detection and separation of gold sulphide in the heavy fraction of BMQ sourced from the BIF in Goa²⁵. Although the iron ore mining industry in Goa is 67 years old, there is no information on multi-elemental analysis (such as Ag, Au, Pt, REE). However, our previous studies have established that BHQ contains 12–13 ppm of gold⁴. Gold values ranging from 0.98 to 4.72 ppm have been reported from the Gadag greenstone belt, WDC²⁶. Microscopically, the 106 µm sieve fraction indicated the presence of particles of secondary gold, gold sulphide, auriferous quartz, pyrite and other impurities (Figure 1 a). Figure 1 b and c show the morphological classification of gold sulphide detected in this study. Figure 2 a and b shows the typological classification of gold sulphide. Most of the particles were uneven, with an irregular shape and rough surface. They ranged from 0.1 to 1 mm in size and were coarse, sub-angular to angular, with no mechanical damage. The presence of elemental gold and sulphur can be seen in the SEM-EDS spectra (Figure 3). Similar studies on the presence of gold in sulphides have been reported earlier from the Archean Hutti–Maski schist belt^{27,28}. The sodium fusion test or Las-

saigne's test and lead acetate test were found to be positive, as indicated by the violet colour and brownish-black precipitate confirming the presence of sulphur in the heavy fraction. Elemental analysis showed 0.669% (w/w) sulphur, corresponding to approximately 9.32% (w/w) gold sulphide in the heavy fraction of BHQ.

There is ample interest in the prospecting of ores with gold sulphide useful in biomining. However, there are constraints in detecting gold sulphide, especially iron ores. This study presents the successful detection and separation of gold sulphides from the BHQ sample in Goa. We obtained the gold sulphide-rich heavy fraction with particle sizes ranging from 0.1 to 1 mm. The gold content was earlier found to be 12–13 ppm (ref. 4), whereas sulphur analysis of the heavy fraction showed 0.669% of sulphur, indicating 9.32% (w/w) of gold sulphide in the BHQ. Since there are huge deposits of BHQ in Goa which are exported without rational multi-elemental analysis or detection or quantification of gold sulphide, the present study demonstrates the potential for more advanced research in this area for systematic geochemical prospecting of auriferous ore bodies for gold sulphides. We used separated crude gold sulphide from local auriferous ores⁴ as chemical feedstock for small-scale biomining experiments^{29,30}. Work is in progress with encouraging results. Sulphides can be used in the bio-oxidation

of gold by employing hyperacidophilic microbes^{31,32}. We recommend the simple technique used in the present study detect gold sulphide in any ore with large ferromagnetic overload. This would help in undertaking more comprehensive and advanced analytical studies on auriferous ores in India and the rest of the world. Deposits of refractory sulphide ores could be the main source of gold production in the future^{33,34}.

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