Characterization and identification of Au pathfinder minerals from an artisanal mine site using X-ray diffraction

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ABSTRACT

Gold-associated pathfinder minerals have been investigated by identifying host minerals of Au for samples collected from an artisanal mining site near a potential gold mine (Kubi Gold Project) in Dunkwa-On-Offin in the central region of Ghana. We find that for each composition of Au powder (impure) and the residual black hematite/magnetite sand that remains after gold panning, there is a unique set of associated diverse indicator minerals. These indicator minerals are identified as SiO₂ (quartz), Fe₃O₄ (magnetite) and Fe₂O₃ (hematite), while contributions from pyrite, arsenopyrites, iridosmine, scheelite, tetradymite, garnet, gypsum and other sulfate materials are insignificant. This constitutes a confirmative identification of Au pathfinding minerals in this particular mineralogical area. The findings suggest that X-ray diffraction could also be applied in other mineralogical sites to aid in identifying indicator minerals of Au and the location of ore bodies at reduced environmental and exploration costs.

Introduction

At mineralogical mining sites, the fast location of ore bodies is paramount in order to reduce exploration costs. For this purpose, pathfinding minerals are important. These minerals act as an aid in the original ore body discovery. Au that can be traced from the presence of pathfinding minerals mostly originates as anhedral crystal assemblies (i.e., without well-defined crystal facets) that naturally exist as single or

polycrystalline mineral aggregates that are usually found in situ in hydrothermal quartz veins and other kinds of key deposits in metamorphic and igneous rocks [1, 2].

The most common mineral at most Au mining sites is pyrite (FeS₂) that can also be found in oil shales and coal [3]. Other common minerals at Au mining sites are arsenopyrite, different forms of silicate minerals (garnet) and magnetite (Fe₃O₄). Both mineralogical and geochemical information are indispensable to

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provide an initial valuation of the potential ore zone of an exploration area.

Quantitative interpretation of X-ray diffraction (XRD) data [4] has long been applied to distinguish between mineral assemblages, and to define chemical and mineralogical compositions [5]. Previous XRD studies of Au and associated minerals have mostly been performed to determine the grain size morphology and crystallinity [6]. XRD has been used to conclude that highly hydrated and water-saturated environments contribute to the migration of Au within alluvial regimes and on hydrothermal mineral assemblages [7–9]. Multivariate statistical analysis and geostatistical methods have been applied to identify pathfinding elements [10, 11]. Bayari et al. [4] found that mineralized regolith profiles and mobility of elements (minerals) in the soil at the Bole–Nangoli gold belt in the north-eastern Ghana could mainly be attributed to amorphous mineral phases. Furthermore, Zhao and Pring (2019) [12] studied the mineral transformation in Au and silver (Ag) in fluids using the telluride group of minerals associated with Au and focused on the texture, reaction mechanism and the kinetics of the oxidation leaching of the tellurides.

Cairns et al. [13] identified topsoil minerals and pathfinders of Au by considering the fine grain size and amorphous nature of the minerals. Furthermore, XRD studies on the influence of thermal stability of magnetite investigate the effect of temperature on the phase transitions [14–21]. This information is of importance for the investigation of magnetite as a pathfinder mineral of Au. As follows from this background, there is still a need for the characterization of pathfinding Au-associated minerals by XRD on residual samples to establish their relationship and to preserve information about the physiochemical situations of their origin.

In this work, we investigate the crystal structure of Au in relation to the corresponding pathfinding minerals, such as quartz (SiO_2), Fe_3O_4 , Fe_2O_3 , FeS_2 and Fe_{1-x}S , collected from an *artisanal* mining site, i.e., a small-scale hand-mining site, in the central region of Ghana. XRD was used for phase identification and to obtain structural information including Rietveld refinement. In addition to the known minerals, we also identified hematite (Fe_2O_3) as an important pathfinding mineral. The present study can be used to enable future identification of pathfinding minerals for Au exploration.

Experimental details

Description of the field site

The sample collection site is located close to the Kubi Gold (Adansi Gold) on the outskirts of Dunkwa-On-Offin, ($5^\circ 58'11.32''$ N, $1^\circ 46' 59.15''$ W) as shown in Fig. 1. Dunkwa is the capital of the Upper Denkyira East Municipal District located in the central region of Ghana and is drained by several rivers and streams with the Offin river serving as the main river source. The location follows the geology of Ghana which is associated with the antiquity of crystalline basement rock, volcanic belts and sedimentary basins. Most Au is found in steeply dipping quartz veins in shear zones within the Birimian basins with sulfur-rich minerals, such as arsenopyrites and FeS_2 . Other sources of Au found are alluvial placer Au in the Offin river deposits in gravels as well as some mineralized placer Au reconstituted with minerals, such as Fe_3O_4 and Fe_2O_3 in quartz-pebble conglomerates of the Tarkwaian deposits [22]. Extremely oxidized, weathered or putrefied rock commonly located at the upper and exposed part of the ore deposit or mineralized vein known as “gossan” or iron cap serves as a guide to trace buried Au ore deposits in this area [23]. The surface oxides of the minerals at this site are usually red, orange to yellowish-brown color serving as an alteration to the parent rock or soil.

Sample preparation

Sediment samples that contain Au were extracted from a depth of 10 m of an artisanal mining site in Dunkwa-On-Offin. Figure 2 shows the depth profile at the mining site. Each sample was divided into two parts where one portion was refined into pure solid Au, while the other part of the powder sample was subjected to Au panning, that is, washing and magnetic extraction of Fe-based minerals as shown in Fig. 2. The final three samples (Fig. 3) containing a solid Au nugget, untreated (impure) Au powder and the separated black sand-like minerals were examined by X-ray diffraction. The size of the two powder crystal samples ranges from 0.05 cm to about 0.2 cm in the maximum dimension of which most were hopped single crystals with an octahedral crystal structure with a few being of non-octahedral forms.

Figure 1 a Geographical and geological maps of the mining areas in and around Dunkwa-On-Offin, in the Ashanti Gold Belt of central Ghana, CC-BY license [24]. The right map indicates

different rock types in four pronounced mining zones. b Photograph of sampling collection area of the artisanal mining site. On-site photographs by G. Nzulu in Nov. 2019.

Figure 2 **a** Wet residual sample from the Dunkwa-On-Offin artisanal mine site. **b** Refined part of a sample into a Au nugget of 22 carats as measured with a digital electronic Au purity Analyzer DH 300 K from VTSYIQI. **c** Dried sample after coarse rinsing before fine panning. Note that the sample contains white quartz as well as black magnetite and hematite. **d** Final impure Au after fine panning. Photographs by G. Nzulu.

Magnetizing process

During the panning process, the black sand that consists of Fe_3O_4 (magnetite) and Fe_2O_3 (hematite) sink to the bottom of the pan. While the black sand

remained in the pan, a strong permanent magnet was swept over (to and fro) in a circular motion, a couple of centimeters above the material to maximize the magnetic susceptibility (induced ferromagnetics in the Fe_2O_3) for easy capture of the magnetite and

Figure 3 **a** Final residual sample containing Au, sand, and other magnetic materials to undergo magnetic separation. **b** Impure $\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4$ minerals. Photographs by G. Nzulu.

Figure 4 X-ray diffractogram of the bulk solid Au sample showing distinct peaks.

Table 1 Structural refinement parameters of solid bulk Au from XRD

Symmetry: cubic			Space group = Fm-3 m			
Wavelength Cu K α = 1.5406 Å			COD ID: 9008463		Ref. cell volume = 67.83 Å ³	
Wavelength Cu K β = 1.5444 Å					Refined cell volume = 67.82 Å ³	
Observed			Calculated		Difference	
2 theta	d	h k l	2 theta	d	2 theta	d
38.211	2.35344	1 1 1	38.185	2.35500	0.026	0.00156
44.394	2.03895	2 0 0	44.393	2.03900	0.001	− 0.00005
64.615	1.40276	2 2 0	64.578	1.44200	0.037	− 0.03924
77.616	1.22911	3 1 1	77.549	1.23000	0.067	− 0.00089
81.761	1.17696	2 2 2	81.724	1.17740	0.037	− 0.00044
98.238	1.01882	4 0 0	98.137	1.01960	0.101	− 0.00078
Cell						
Parameters	Refinement	Reference		Error		
a (Å)	4.07803 ± 5.7603E-5	4.07825		− 0.00022		
b (Å)	4.07803 ± 5.7603E-5	4.07825		− 0.00022		
c (Å)	4.07803 ± 5.7603E-5	4.07825		− 0.00022		
Alpha (°)	90.0000	90.0000				
Beta (°)	90.0000	90.0000				
Gamma (°)	90.0000	90.0000				

hematite. The process was repeated until there was no more added material on the surface of the permanent magnet. The magnetically captured material was dominated by magnetite that is a pathfinding mineral (Fig. 3b) in addition to minority minerals that can be identified using XRD.

X-ray diffraction measurement

The samples (both solid and powder forms) were irradiated using a PANAnalytical X'pert [25] powder diffractometer with a theta–2 theta configuration. The

operating conditions and equipment settings were Cu-K α radiation wavelength of 1.5406 Å ($\approx$ 8.04 keV); Cu long fine focus tube set to 45 kV and 40 mA; scan step size of 0.033; counting time of 10.16 s per step and scan range between 30 and 100° in 2 theta scans. The size of the solid bulk Au nugget was $2 \times 1.6 \times 0.5$ cm. The powder samples of impure Au, Fe₂O₃ and Fe₃O₄ had varying grain sizes (0.05–0.2 cm) and were put on a sample holder mounted on the diffractometer's sample mounting stage such that the crystal face was properly oriented and closely aligned with the diffractometer circle of

Figure 5 X-ray diffractogram of the impure powder Au with other pathfinding Au minerals.

the goniometer. The XRD data were quantitatively analyzed by Rietveld refinement using the MAUD software [26, 27].

Results and discussion

Figure 4 shows an X-ray diffractogram of the Au nugget sample in Fig. 2b with the result of Rietveld refinement assuming pure Au together with the residual of the fit [28]. The six pronounced peaks in the diffractogram are indexed as a cubic fcc Au structure (Fm-3 m space group) with lattice parameter of $a = 4.079 \text{ \AA}$. Table 1 lists the full assigned observed peak list as well as the resulting crystallographic parameters from the refinement. These are in agreement with literature assignments for Au [28, 29].

Figure 5 shows an X-ray diffractogram of the impure powder Au sample shown in Fig. 3a. Table 2 lists the refined crystallographic parameters of Au and the pathfinder minerals identified from powder Au samples. These data are in agreement with reference data [27–32]. The diffractogram from the unrefined powder sample shows the presence of other minerals, that is, pathfinder minerals for Au. These are dominated by SiO_2 (quartz) with some Fe_3O_4 (magnetite).

The lattice parameter of the SiO_2 in the impure Au were found to be $a = 4.91 \text{ \AA}$ and $c = 5.43 \text{ \AA}$ (space group P3221), consistent with reference data [30]. This sample also contains Fe_3O_4 (cubic, space group

Fd-3 m) with a lattice parameter of $8.36 \text{ \AA}$, consistent with literature data [27].

Figure 6 shows an X-ray diffractogram from the residual black sand after Au panning. The diffraction peaks of this sample were identified as the crystalline structure of Fe_2O_3 (hematite). Table 3 lists the diffraction peaks and crystallographic parameters determined from the Rietveld refinement of Fe_2O_3 . This is in accordance with literature and reference data [32, 33]. The crystal structure of Fe_2O_3 is rhombohedral with a space group R-3c and lattice constant of $5.0991 \text{ \AA}$ [34, 35].

Comparing Figs. 4 and 5, it can be seen that the latter sample contains Au together with pathfinder minerals in the form of magnetite and quartz. The most abundant mineral observed in the diffraction pattern of the impure powder Au sample is SiO_2 (quartz) having three distinct peaks at $2\theta = 40.284^\circ$, 67.957° and 90.818° corresponding to {111}, {212} and {312} crystalline planes of the SiO_2 phase, respectively. The refined pattern of SiO_2 shown in Fig. 5 is in agreement with the literature data in refs [36–39], which also holds true for the moderate amount of magnetite present [27]. This shows that impure Au or final concentrate (non-pure Au) have a high quantity (percentage) of pathfinder minerals as impurities. Note that Au atoms easily substitute with Ag atoms forming an alloy with the same fcc crystal structure and that it is impossible to distinguish pure Au from an Au–Ag alloy with XRD.

The diffractogram in Fig. 6 contains major peaks at $2\theta = 32.609^\circ$, 34.915° , 38.658° , 40.196° , 48.618° ,

Table 2 Structural refinement parameters of impure Au powder sample containing other pathfinder minerals

(a) Impure Au parameters [52.23%] Initial symmetry: cubic				Space group = Fm-3 m			COD ID: 9008463 Ref. cell volume = 67.830 Å ³ Refined cell volume = 67.832 Å ³	
Observed			Calculated		Difference			
2 theta	d	h k l	2 theta	d	2 theta	d		
38.178	2.35539	1 1 1	38.185	2.35500	— 0.007	0.00039		
44.641	2.02824	2 0 0	44.393	2.03900	0.248	— 0.01076		
64.615	1.44126	2 2 0	64.578	1.44200	0.037	— 0.00074		
77.617	1.22910	3 1 1	77.549	1.23000	0.068	— 0.00009		
81.728	1.17735	2 2 2	81.724	1.17740	0.004	— 0.00005		
98.171	1.01934	4 0 0	98.137	1.01960	0.038	— 0.00026		
Cell								
Parameters	Refinement			Reference	Error			
a (Å)	4.07830 ± 4.7828E-4			4.07825	5.0 E-5			
b (Å)	4.07830 ± 4.7828E-4			4.07825	5.0 E-5			
c (Å)	4.07830 ± 4.7828E-4			4.07825	5.0 E-5			
Alpha (°)	90.0000			90.0000				
Beta (°)	90.0000			90.0000				
Gamma (°)	90.0000			90.0000				
(b) SiO ₂ structural parameters [33.89%] Crystal system: hexagonal				Space group = P3221			COD ID: 1538064 Ref. cell volume = 112.979 Å ³ Refined cell volume = 113.36 Å ³	
Observed			Calculated		Difference			
2 theta	d	h k l	2 theta	d	2 theta	d		
40.284	2.23698	1 1 1	40.300	2.23613	0.026	0.00085		
67.957	1.37829	2 1 2	67.744	1.38210	0.001	— 0.00381		
90.818	1.08167	3 1 2	90.831	1.08155	0.067	0.00012		
Cell								
Parameters	Refinement			Reference	Error			
a (Å)	4.90970 ± 1.9E-3			4.91304	— 0.00334			
b (Å)	4.90970 ± 1.9E-3			4.91304	— 0.00334			
c (Å)	5.43023 ± 3.7E-3			5.40463	0.02557			
Alpha (°)	90.00 ± 0.00			90.0000	0.0000			
Beta (°)	90.00 ± 0.00			90.0000	0.0000			
Gamma (°)	120.00 ± 0.00			90.0000	0.0000			
(c) Fe ₃ O ₄ structural parameters. [13.88%] Crystal system: cubic				Space group = Fd-3 m			COD ID: 9005813 Ref. cell volume = 583.816 Å ³ Refined cell volume = 584.277 Å ³	
Observed			Calculated		Difference			
2 theta	d	h k l	2 theta	d	2 theta	d		
33.800	2.64979	1 0 4	33.153	2.70000	0.647	— 0.05020		
35.571	2.52190	1 1 0	35.612	2.51900	— 0.041	0.00290		
38.813	2.31830	0 0 6	39.277	2.29200	— 0.464	0.02630		
40.317	2.23523	1 1 3	40.855	2.20700	— 0.538	0.02820		
44.002	2.05620	2 0 2	43.519	2.07790	0.483	— 0.02170		
49.843	1.82806	0 2 4	49.480	1.84060	0.363	— 0.01250		
58.059	1.58740	0 1 8	57.590	1.59920	0.469	— 0.01180		
Cell								
Parameters	Refinement			Reference	Error			
a (Å)	8.36 ± 0.00			8.3578	0.0022			
b (Å)	8.36 ± 0.00			8.3578	0.0022			
c (Å)	8.36 ± 0.00			8.3958	0.0022			
Alpha (°)	90.00 ± 0.00			90.0000	0.0000			
Beta (°)	90.00 ± 0.00			90.0000	0.0000			
Gamma (°)	90.00 ± 0.00			90.0000	0.0000			

53.966°, 62.990°, 69.975°, 80.837°, 83.475° and 91.533° identified as {101}, {110}, {006}, {113}, {024}, {116}, {214}, {208}, {128}, {134} and {042} crystalline planes of Fe₂O₃ (hematite), respectively. These refined peaks are in good agreement with the rhombohedral structure of Fe₂O₃ [32, 33].

Generally, including possible microstrain in the Rietveld refinement has a negligible effect on the convergence of the fit (residual), indicating that the samples are essentially strain-free. The results from the impure Au powder sample indicate that SiO₂ (quartz) is the dominant impurity mineral serving as the host rock containing all the pathfinder minerals at

Figure 6 X-ray diffractogram of the Fe_2O_3 mineral.

Table 3 Structural refinement parameters of Fe_2O_3 powder sample

Crystal system: rhombohedral				Space group = R-3c		
COD ID: 900139		Ref. cell volume = 302.722 Å3		Refined cell volume = 313.870 Å3		
Observed			Calculated		Difference	
2 theta	d	h k l	2 theta	d	2 theta	d
32.609	2.74380	1 0 1	33.158	2.70000	− 0.549	0.04380
34.915	2.56768	1 1 0	35.612	2.51900	− 0.697	− 0.04868
38.658	2.32725	0 0 6	39.277	2.29200	− 0.619	0.03525
40.196	2.24167	1 1 3	40.855	2.20700	− 0.659	0.03467
48.618	1.87122	0 2 4	49.480	1.84060	− 0.862	0.03062
53.966	1.69772	1 1 6	54.091	1.69410	− 0.125	0.00362
62.990	1.47447	2 1 4	62.451	1.48590	0.539	− 0.01143
69.975	1.34340	2 0 8	69.601	1.34970	0.374	− 0.00630
80.837	1.18806	1 2 8	80.711	1.18960	0.126	− 0.00154
83.475	1.15710	1 3 4	84.916	1.14110	− 1.441	0.01600
91.533	1.07508	0 4 2	91.345	1.07680	0.188	− 0.00172
Cell						
Parameters		Refinement		Reference		Error
a (Å)		5.0991 ± 1.4E-3		5.0380		0.0611
b (Å)		5.0991 ± 1.4E-3		5.0380		0.0611
c (Å)		14.0767 ± 5.2E-3		13.7720		0.3047
Alpha (°)		90.00 ± 0.00		90.0000		0.0000
Beta (°)		90.00 ± 0.00		90.0000		0.0000
Gamma (°)		90.00 ± 0.00		120.0000		0.0000

the mining site. It is known that SiO_2 is a so-called *gangue mineral* (i.e., a commercially nonvaluable mineral that surrounds or is mixed with a valuable mineral) in hydrothermal ore veins [40], to preserve information about the physiochemical situations of the origin of the veins and to understand the formation of mineral deposits. These dominant SiO_2 species contain structural defects that favor mineral infusion

due to underlying conditions and geological processes, such as crystallization, metamorphism, alterations, changes in crystallization temperatures and precipitation [41–43].

Au associated with Fe_3O_4 is mostly formed in skarns of granular magnetite usually found in contact with metamorphosed areas with magma intrusion into carbonate or silico-carbonate rocks that also

consist of garnet and silicate minerals, among others. The residual black sand together with other dense minerals is considered to be ore that is left over during Au refinement and washing at riverbanks when recovering its Au content [44]. This shows that two of the three most common iron ore minerals; Fe_3O_4 and Fe_2O_3 are widely spread within the mining site and contribute to the Au host minerals alongside SiO_2 . In a near-surface environment (oxide area) Fe_2O_3 act as the gangue mineral and can be transformed to Fe_3O_4 depending on the environmental conditions such as high temperature, oxidation, and pH [45]. The same color of Fe_2O_3 in comparison with black Fe_3O_4 makes it difficult to distinguish between the two in branded iron formations and standing water [44, 46]. It is likely that during the formation of Fe-oxides in the alluvial regime at the Dunkwa–Kubi geological site, Au is internally captured within structures associated with Fe_2O_3 (hematite) that acts as crusts in saprolite and laterite environments. These minerals reveal information about the physiochemical conditions of the origin of structures (structural defects) useful for the understanding of mineral deposit formations.

Conclusions

This study has revealed that sediments and black sands containing Au are associated with pathfinding minerals in impure compositions. This is indicative that Au and pathfinding minerals are all deposited in nature during hydrothermal activation. The XRD analysis identified Au, SiO_2 (quartz), Fe_3O_4 (magnetite) and Fe_2O_3 (hematite). From the XRD patterns, the impure Au and Fe_2O_3 samples can be attributed to the decomposition and transformation of these indicator minerals. Also, the surface (oxide zones) mineralization is altered by Fe_2O_3 as one of the indicator minerals apart from the garnet and the gangue mineral SiO_2 to host Au with other pathfinder minerals beneath the surface.

These results are of importance for the mining industry to underscore the usefulness of XRD in studying soil and sand sediments from mining sites by identifying pathfinder minerals of Au in potential geological sites.

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Compliance with ethical standards

Conflict of interest P. E. and M. M. declare no competing financial interest. G. K. N.'s industry PhD studies are funded by Asante Gold Corporation. Asante Gold Corporation or G.K.N. have no potential financial benefit from this study. The samples in this study are from an artisanal mining site open to the indigenous public.

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