

CHARACTERIZATION STUDY OF BABAN TSAUNI (NIGERIA) LEAD-GOLD ORE

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Abstract:

This study characterized the Baban Tsauni lead-gold ore. The study included the use of XRD, XRF, AAS and both reflected and transmitted-light microscopy. The results showed that the ore consists mainly of galena (PbS), gold (Au) and quartz (basement rock complex). Cadmium can be extracted as a byproduct from this ore while sphalerite (ZnS) and covellite (CuS) occur as trace minerals. The mineralogical studies revealed that galena has coarse grains while the gangue mineral (quartz) has medium to massive grains. The textural relationship between galena and quartz showed a simple locking pattern while the gold is finely disseminated in the quartz. Chemical analysis revealed a mean lead grade of 27.79% and a mean gold grade of 0.02938%. The results indicated that galena can be separated by conventional gravity separation or froth flotation methods.

Keywords: Minerals; Galena; Particle size; Liberation size; Mineralogy.

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1.0 Introduction

Global mobility would have been impossible without lead battery technology. The combined properties of low melting temperature (327°C) high density and ductility makes lead a favourite metal for casting small arms ammunitions and shotgun pellets [13].

Lead remains an important metal and our country needs to properly develop all lead ore deposits in order to achieve their profitable exploitation. The profitability of a mineral deposit is a function of both quantity and quality of the ore. Similarly the mining method and mineral processing route applicable to a deposit depends on the two parameters. The quality of a mineral deposit is determined by subjecting representative samples to characterization study.

Ore characterization is the study of the nature of an ore as it relates to chemical and physical properties in terms of types of minerals (chemical composition), the grades of minerals (concentrations of metals in percentage or parts per million), the grain sizes of minerals, the texture of minerals (grain size distribution, shape, and locking pattern) and the type of associated gangues. Every mineral deposit has its peculiar characteristics in terms of these physical, mineralogical and chemical properties. The response of a mineral to beneficiation depends on these characteristics [4]. Baban Tsauni lead ore was discovered during a geological/physiological mapping of the Federal Capital Territory (FCT) that was carried out in 2004 and 2007 by the Nigerian Geological Survey Agency [6].

2.0 Experimental procedures

2.1 Determination of phases

Four samples of the ore were taken by rifling. Each sample weighing about 3grams was ground to pass $125\mu\text{m}$ and fed into Empyrean X-ray diffractometer (Model-DY674 {2011model}). The XRD characterizes the minerals by comparing the X-ray diffraction of each

mineral with inbuilt standard diffraction patterns of known mineral crystals. The computer software (X pert High score plus) cross-matched the peaks and arrived at the mineral constituents and their chemical characteristics.

2.2 Mineralogical analysis of polished sections

Three representative lumps were selected, by grab sampling from the samples which were collected from each sampling location and cuboids of sections, 10mm by 10mm by 10mm, were cut from each lump. The sections were mounted in cups with epoxy resin. The mounted sections were ground with 120 grit silicon carbide abrasive papers followed by 320, 400, 600 and 800 abrasive papers [14]. Thereafter the samples were polished with diamond polishing paste grade 6, followed by grade 3, 1 and finally 0.25 μ m.

The polished sections were viewed under a research microscope (Metallux 11) and the grain sizes were measured with a calibrated stage micrometer. Five hundred measurements were taken. Grain size distributions were calculated and the liberation size determined. The cleavage patterns of minerals were examined. Texture characterization was undertaken by observing the locking pattern, the grain size distribution in the ore, shape of grains, parting lines and the effect of polishing if any. A frequency curve of grain size distribution was plotted.

2.3 Mineralogical analysis of thin sections

Four representative lumps were selected out of the samples and cuboids of sections, 40mm by 15mm by 10mm, were cut from each lump. Each section was mounted on a glass slide using Canada balsam and was ground to a thickness of about 0.03mm [12]. The thin sections were then covered with another glass and examined under a transmitted-light microscope to determine the shapes and colours of the different minerals in the ore. Standard graticules were also used to assess the grain sizes of the different minerals and gangue.

2.4 Particle size analysis

A sledgehammer was used to break the massive ore particles into sizes that could be crushed with a laboratory jaw crusher. The closed side of the laboratory jaw crusher was set at 10mm to minimize the level of fine particles. The products were screened and the materials that passed through 1.4mm standard screen were further screened into eight close size fractions, in readiness for chemical analyses. The materials retained were passed through a roll crusher and a ball mill in that order. To avoid over-grinding in the roll mill the materials were screened into +5mm and +3mm. The roll mill products which passed through 3mm standard sieve were used as feed to ball milling operations. The particle size analysis was carried out by placing the ore samples in the uppermost sieve. The nest of BBSS (1796) standard sieves was placed on the laboratory sieve shaker and bolted down. The amplitude of vibration was adjusted to 8mm and the assembly was allowed to vibrate for 6 minutes. Thereafter the nest of sieves was taken apart and the mass of materials retained on each sieve was measured [10].

2.5 Determination of chemical compositions

The concentrations of metals (assays or mineral grades) in the ore were determined, for each of eight particle sizes of products from crushing operation, by energy dispersive X-ray fluorescence analysis (EDXRF). Similarly the close size fractions obtained after fifteen minutes of ball milling operation were subjected to chemical analysis to determine the grades of all the minerals in the ore. One gram of each sample was ground manually with an agate mortar and pestle to a grain size of less than 125 μ m. About 0.3 – 0.5g of the ground sample was weighed and mixed with one drop of a binder (PVC dissolved in toluene). The mixture was compressed into a 19mm diameter pellet in a steel die using a 10 tonnes hydraulic press. The pellet was loaded in the chamber of a PW 4030 X-ray spectrometer (a Mini pal 4 version of a compact energy dispersive X-ray spectrometer). A voltage (30kV maximum) and a current (1mA maximum) were applied to produce the X-rays which excited the sample for 10minutes. The spectrum from the sample was then analysed by the dedicated Mini pal analytical software to determine the concentrations of the elements in the sample.

3.0 Results and discussion

The XRD Pattern is shown in Figure 1 and the peak patterns of some minerals in the ore

are presented in Figures 2 to 5. The results (Table 1) reveal that the lead mineral occur as galena – lead sulphide (PbS). The results also revealed the presence of several trace minerals that have reacted with galena to form well defined crystals (Galenobismutite, Chlorine bearing galenobismutite, Heyrovskyite, Marrucciite, Tsumebite, Soucekite, Boulangerite, Albite and Jordanite).

Pulses

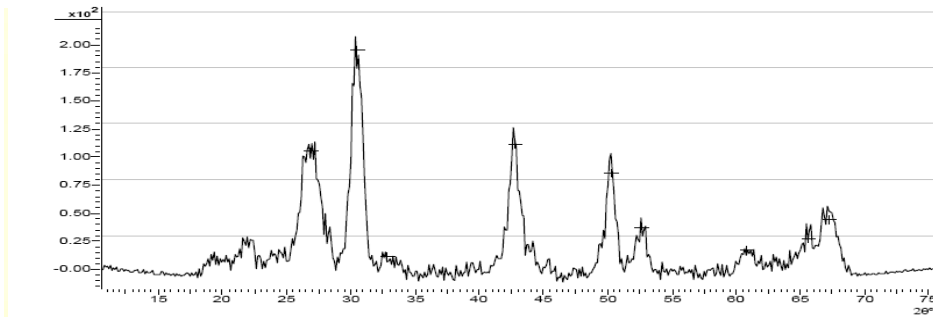
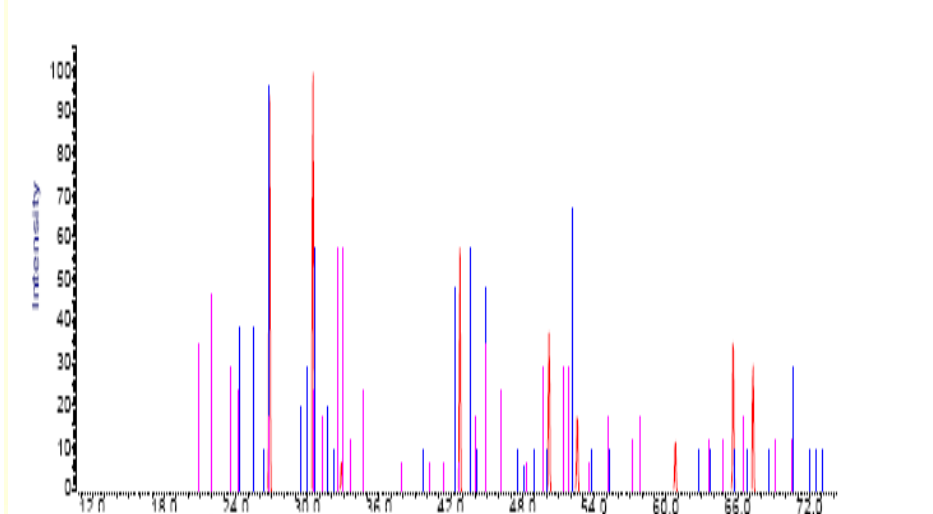


Figure 1: The XRD Pattern of Baban Tsauni Lead ore



Position [o2 Theta] (Copper (Cu))

Blue: Lead bismuth sulphide; Heyrovskyite, Bismuthian [(Pb,Bi)₆Bi₂S₉]

Red: Bismuth copper lead sulphide selenide; Soucekite, [PbCuBi(S,Se)₃]

Figure 2: XRD Peak Patterns for Heyrovskyite and Soucekite.

Heyrovskyite (Figure 2), bismuthian has the formula given by (Pb,Bi)₆Bi₂S₉ with six bismuth atoms occurring as substitution elements in lead sulphide while two atoms occur as sulphide in their right [4]. Heyrovskyite is known to contain a trace amount of cadmium and selenium [15]. Though the XRD result did not reveal these trace elements the chemical analysis

by energy dispersive x-ray fluorescence (EDXRF) results (Tables 5 and 6) revealed the presence of cadmium and selenium.

Soucekite (Figure 2) is bismuth copper lead sulphide selenide and its chemical formula is $\text{PbCuBi}(\text{S},\text{Se})_3$. Selenium occurs as substitution element for sulphur while lead, copper and bismuth occur as primary metals in the sulphide. Its crystal system is orthorhombic and the cell parameters are; $a=8.153(\text{\AA})$, $b=8.498(\text{\AA})$, $c=8.080(\text{\AA})$ and $z=4$.

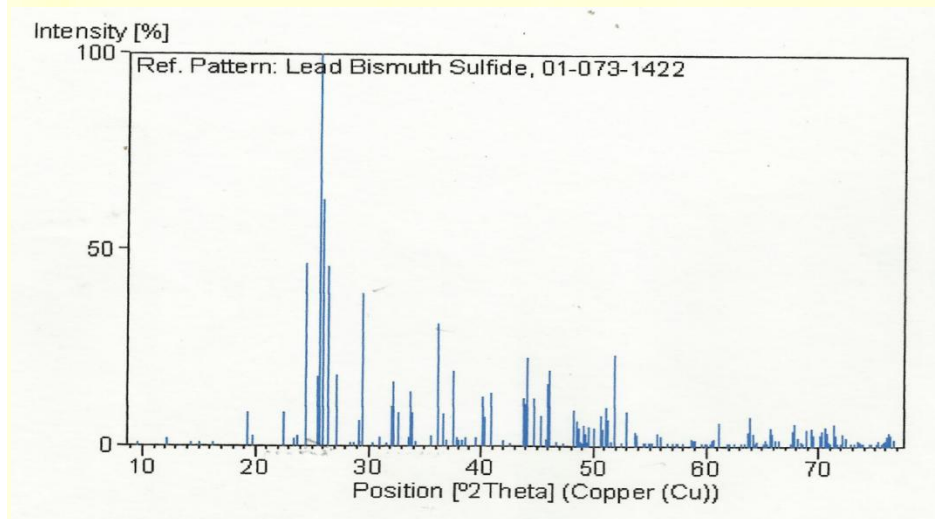


Figure 3: XRD Peak Patterns for Galenobismutite

Galenobismutite (Figure 3) is Lead Bismuth Sulphide (PbBi_2S_4) with orthorhombic crystal system. It is whiter than galena in polished sections and strongly anisotropic. Its polarization colour range from yellow to dark brown. Its cell parameters are: $a=11.669(\text{\AA})$, $b=14.533(\text{\AA})$, $c=4.090(\text{\AA})$, and $z=4$.

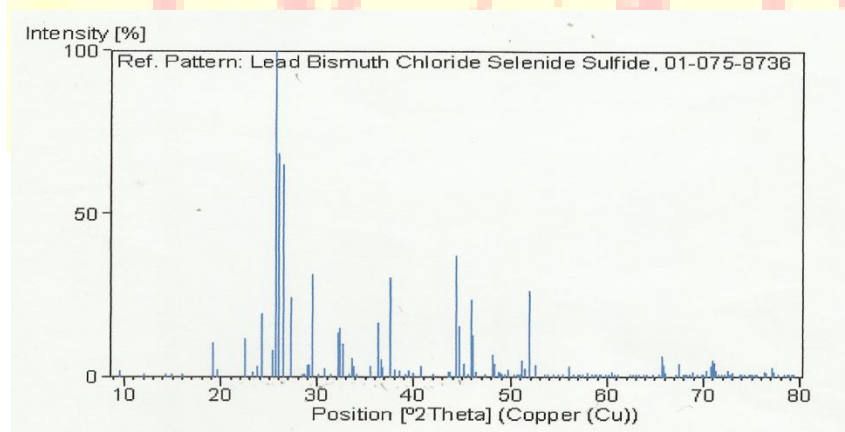


Figure 4 XRD Peak Pattern for Chloride Bearing Galenobismutite

Chloride bearing galenobismutite (Figure 4) is lead bismuth chloride selenide sulphide and its chemical formula is given by; $(Pb_{1.15}Bi_{1.82})(S_{3.584}Se_{0.262}Cl_{0.156})$. Its crystal system is orthorhombic and the cell parameters are: $a=11.8460(\text{\AA})$, $b= 14.6670 (\text{\AA})$, $c=4.0850 (\text{\AA})$, and $z=4.00$.

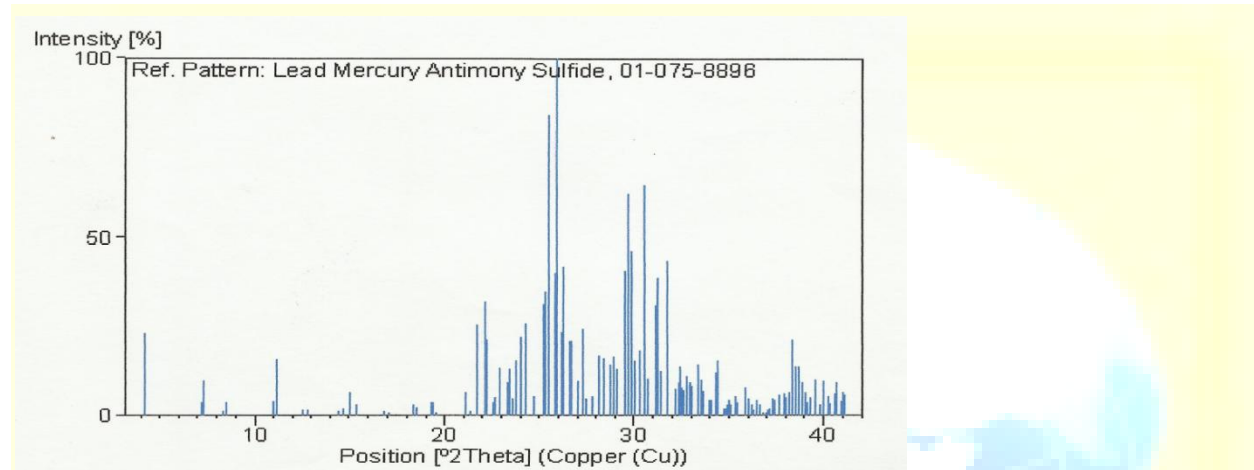


Figure 5: XRD Peak Patterns for Marrucciite

Marrucciite (Figure 5) is lead mercury antimony sulphide and has a chemical formula given by; $Hg_3Pb_{16.07}Sb_{17.93}S_{46}$ (Table 1). It has monoclinic crystal system and its cell parameters are: $a=48.3200(\text{\AA})$, $b= 4.1170 (\text{\AA})$, $c=24.0560 (\text{\AA})$, and $z=2.00$. The results on chemical composition of the ore (Table 5) confirmed the presence of mercury.

Table 1 Description of mineral phases from XRD results

Major Valuable Mineral	Gangue Minerals
Galena (PbS)	Soucekite-Bismuth Copper Lead Sulphide Selenide ($CuPbBi\{S,Se\}_3$)
	Heyroskvylte-Lead Bismuth Sulphide ($(Pb,Bi)_6Bi_2S_9$)
	Galenobismutite- Lead Bismuth Chloride Selenide Sulfide { $(Pb_{1.15}Bi_{1.82})(S_{3.584}Se_{0.262}Cl_{0.156})$ }
	Galenobismutite- Lead Bismuth Sulfide ($PbBi_2S_4$)
	Marrucciite- Lead Mercury Antimony Sulfide ($Hg_3Pb_{16.07}Sb_{17.93}S_{46}$)
	Boulangerite- Lead Antimony Sulfide ($Pb_5Sb_4S_{11}$)
	Albite, calcian – Sodium calcium Aluminium Silicate

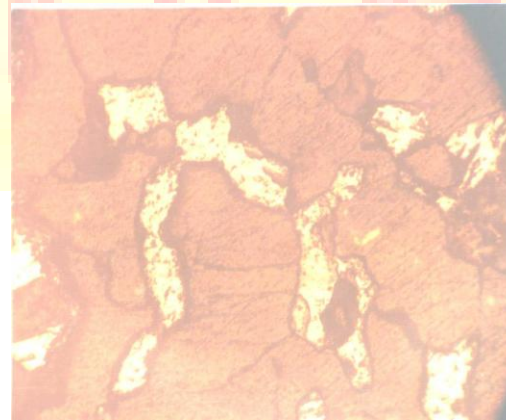
($[\text{Na}_{0.75}\text{Ca}_{0.25}][\text{Al}_{1.26}\text{Si}_{2.74}\text{O}_8]$)
Jordanite- Lead Arsenic Sulfide ($\text{Pb}_3\text{As}_2\text{S}_6$)
Tsumebite- Copper lead Phosphate Sulphate Hydroxide
($\text{CuPb}_2[\text{PO}_4]\text{SO}_4[\text{OH}]$)
Quartz- Silicon Oxide (SiO_2)

Other gangue minerals in the ore include tsumebite, boulangerite, albite, jordanite, and quartz. Tsumebite is a hydrous basic phosphate and sulphate of lead and copper ($\text{CuPb}_2[\text{PO}_4]\text{SO}_4[\text{OH}]$). Its physical colour is emerald green [8]. The presence of tsumebite accounted for green colour observed on few of the site samples. It is transparent and green under transmitted light (Figure 6{d}). These results indicate that copper and lead are present as primary metals in a phosphate/sulphate mixture while albite (sodium calcium aluminium silicate) is present as minor gangue mineral.

The study on the polished sections under reflected light microscope (metallux 11) also revealed that the ore consists of galena (white: Figures 6a to 6c), quartz (grey: Figures 6b and 6c). Triangular pits resulting from polishing (Figures 6a and 6c) were readily seen and this characteristic feature confirms that lead mineral is present as lead sulphide (PbS), that is galena ([7]; [1]; [2]).



(a)



(b)

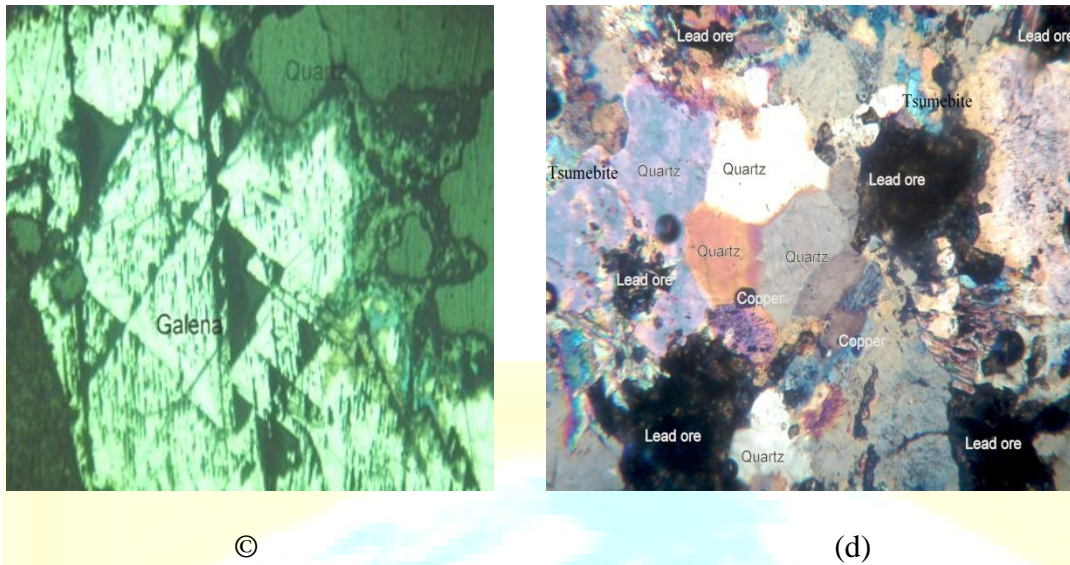


Figure 6 (a) Triangular pits and perfect cleavage of galena, (b) Quartz grains, interlocked with galena and galenobismutite, (c) Galena in simple locking with quartz grains and mutual grain boundaries and (d) Micrograph of thin section showing the colours, shapes and sizes of different minerals in the ore.

The perfect cubic cleavage in all directions and the cubic habit that were observed (Figure 6c) are known characteristics of galena ([9]; [7]). The quartz (Figure 6b) showed mutual grain boundaries that give indication that breakage along grain boundaries will occur when the sample is ground. Breakage along boundary lines implies that comminution energy is less than required for ore with complex locking patterns. In addition some of the quartz grains are free at coarse particle size, thus making it possible to easily remove some of the gangue mineral by gravity separation methods at coarse size. The mineralogical study on polished sections revealed that quartz is the principal gangue, accounting for about 60% of the ore, galena about 37%, copper ore 1% and others about 2%.

Results of mineralogical studies on thin sections under transmitted-light microscope are shown in Figure 6d. The study revealed that quartz (white, blue, pink or brown) is the main gangue mineral with medium to massive grains. Galena was identified as the major valuable

mineral while minor presence of copper mineral (tsumebite – green) was observed in some of the samples. The transmitted-light micrographs of all the thin sections show that the colour of the gangue mineral vary from white, blue, grey, brown to pink due to the presence of trace elements in the quartz [7]. Microscopic size analysis (with the aid of standard graticules) revealed that the textural feature of the gangue mineral is medium grains with sizes ranging from 1mm to 5mm.

This result implies that the liberation of the gangue mineral of Baban Tsauni ore can be achieved at coarse particle size and pre-concentration removal of gangue carried out by a cheap gravity separation method. Some of the quartz grains revealed fine dissemination of valuable mineral (gold) in them.

Results of grain size analysis on polished sections are shown in Table 1 and Figure 7. The grain size of the lead ore varied between 50.5 μ m and 950.5 μ m. About 78.8% of the grains have sizes within the range of 150.5 μ m to 550.5 μ m. This result indicates that the lead ore is fairly coarse and fine grinding of this ore is not required in order to achieve reasonable liberation of the lead mineral from the quartz.

The stage micrometer measurement of the diameters of five hundred grains of galena resulted in a mean value of 349.9microns. This result indicates that the ore must be ground below 349.9 say 350microns in order to achieve liberation of the valuable mineral from quartz. This result implies that the ore is coarse and that the minerals will be practically free from each other at a size of 350microns (the liberation size). This follows that the Baban Tsauni ore will respond well to gravity separation at a fairly coarse size. This implies that the cost of beneficiation of the ore will be relatively low when compared to ores with complex textures.

Table 2: Grain size distribution in polished sections.

Grain size (μm)	Frequency (%)	Cumulative % frequency (and below)	Cumulative % frequency (and above)
50.5	11.2	11.2	100
150.5	14.2	25.4	88.8
250.5	16	41.4	74.6
350.5	22.4	63.8	58.6
450.5	17.4	81.2	36.2
550.5	8.8	90	18.8
650.5	3.6	93.6	10
750.5	2.2	95.8	6.4
850.5	2.4	98.2	4.2
950.5	1.8	100	1.8

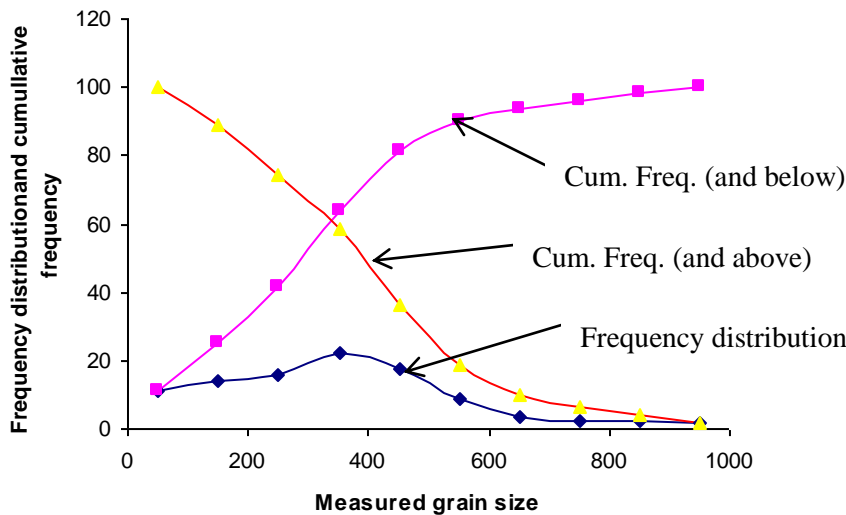


Figure 7: Grain size distribution in polished sections

The results of particle size analysis after three minutes of grinding operation are presented in Table 3 while Figure 8 show the plots of cumulative per cent mass passing or retained versus particle size. The results show that the relationship between cumulative per cent mass passing or retained for grinding (Figure 8) of the ore follow the characteristic curve which

most minerals exhibit ([11]; [4]). The intercept of the two cumulative characteristic curves occurs at 50% as expected [11] and the corresponding particle size (D_{50}) is 421.3microns. This implies that the mean size fraction of comminution products falls within less than 500 μ m but greater than 350 μ m. This indicates that the mean particle size falls within the vicinity of the liberation size which was found to be 350 μ m.

Table 3: Particle size analyses of products after 3minutes of grinding operation.

Sieve size (μ m)	Weight retained (g)	Weight (%) retained	Cumulative weight (%) retained	Cumulative weight (%) passing
1400+1000	24.3	24.40	24.398	75.602
1000+710	11	11.04	35.442	64.558
710 +500	10.2	10.24	45.683	54.317
500+355	8.8	8.84	54.518	45.482
355+250	7.6	7.63	62.149	37.851
250+180	7.4	7.43	69.578	30.422
180+125	6.1	6.12	75.70	24.297
125+90	5.4	5.42	81.12	18.876
90+63	7.7	7.73	88.86	11.145
63	11.1	11.14	100.00	

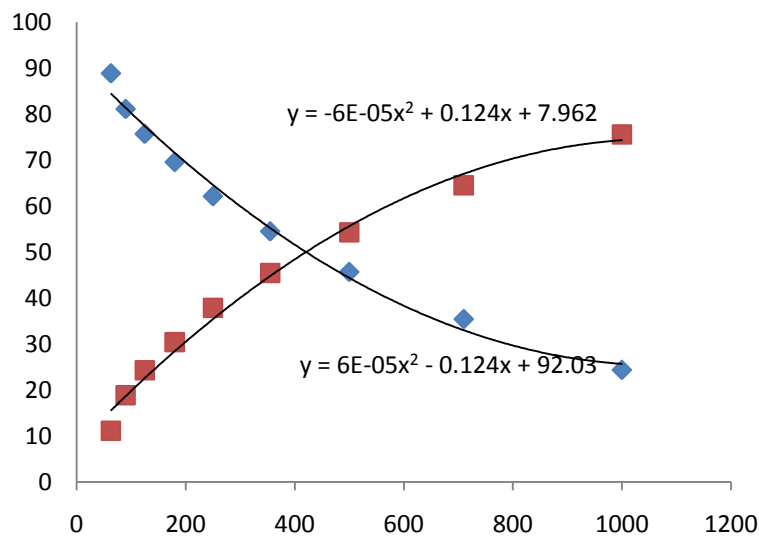


Figure 8: Cumulative Per Cent Mass Passing (Pink) and Retained (Blue) Versus Particle Size.

The size of particles from grinding operation was characterized by applying Gates-Gaudin-Schuhmann size distribution model (Equation 1) to the data in Table 3.

$$\log y = \log 100 - m \log k + m \log x \tag{1}$$

where y = cumulative per cent mass passing sieve size x , m = distribution modulus and k = the size modulus.

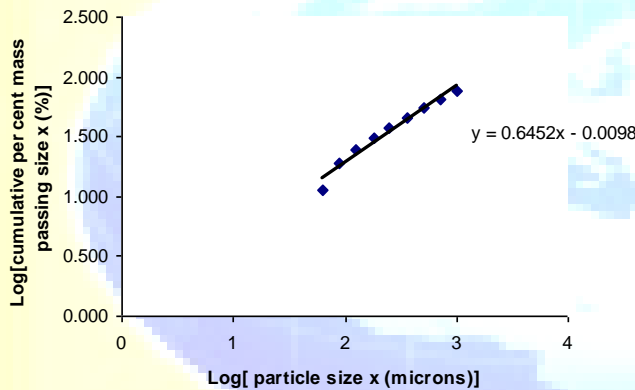


Figure 9: Log-log plot of cumulative % mass passing versus particle size for products from grinding operation after three minutes.

Since the plot is on Log-Log basis, the actual equation of the line is,

$$\text{Log}y = 0.6452 \log x - 0.0098 \tag{2}$$

where y = the cumulative per cent mass passing the sieve size and x = sieve size in micrometer.

The distribution modulus (m) is the slope of the line and it has a value of 0.6452. This value is small and it implies that the spread of particle sizes in the distribution is large [3].

The size modulus k is, by definition, the particle size at which the cumulative per cent mass is 100. Hence putting into Equation 2 the values $y = 100$ and $x = k$ yields,

$$\text{Log}100 = 0.6452 \log k - 0.0098 \tag{3}$$

$$\text{Hence } \log k = \frac{\log 100 + 0.0098}{0.6452} = 3.1150031.$$

Therefore, $k = 1303.2\mu\text{m}$.

The size modulus is a measure of the top size and has the value of $1303.2\mu\text{m}$ after 3 minutes of grinding operation. The implication is that 100% passing feed size of $1400\mu\text{m}$ dropped to 100% passing $1303.2\mu\text{m}$ after three minutes of grinding operation. This is 93.086% of the initial top size. This size reduction characteristic applies to particles in the size range $63\mu\text{m}$ to $1400\mu\text{m}$ since grindability increases with decrease in particle size ([3]; [13]).

Table 4 presents a summary of distribution of the valuable minerals (lead and gold) in the fractions. Though the presence of gold was not captured in the XRD results, assay analysis by EDXRF revealed high grade of gold. Table 6 is a typical assay result showing gold concentration of 0.08%. The 7th column of Table 4 show gold assay in various fractions and a mean gold assay of 0.02938%. According to [15], gold can be exploited profitably at an assay of 0.0001%. The foregoing implies that Baban Tsauni ore is 2,938 times richer than the base minimum required for exploitation of gold. The results showed that lead grade increased with decrease in particle size and the increase was more significant for particle size below $500\mu\text{m}$. The gangue mineral (quartz) has a higher grindability (13.57kWh/tonne [15]) than free galena (11.4kWh/tonne [3]). Thus the remarkable increase in lead grade resulted from increase in liberated galena particles which were easier to grind than free quartz particles.

Table 4: The distribution of lead and gold in size fractions during grinding operation.

Sieve Size(μm)	Particle size (μm)	Mass retained (g)	% mass retained	Lead assay (%)	Lead distribution (%)	Gold assay (%)	Total Silica (SiO_2)
<2000+1400	1673.32	10.9	2.1923	11.873	0.937	0.04	78
<1400+1000	1183.22	45.2	9.0909	24.77	8.103	0	45
<1000+850	921.95	34.9	7.0193	16.51	4.17	0.096	73.6

<850+710	776.85	62.1	12.4899	19.2	8.629	0	69.6
<710+500	595.82	102	20.5149	22.45	16.573	0.08	67.8
<500+355	421.31	50.4	10.1368	29.13	10.625	0.03	58
<355+250	297.91	77.6	15.6074	32.19	18.078	0	53.3
<250+180	212.13	27	5.4304	43.69	8.537	0.01	54.6
<180+125	150	20.1	4.0426	31.54	4.588	0	56
<125+90	106.07	18	3.6203	37.104	4.834	0	43.2
<90+50	67.08	22	4.4248	34.321	5.465	0.04	52.7
<50		27	5.4304	48.421	9.462	0	37.3
	Average			27.7904		0.02938	58.9175

The average assay of lead in the fractions was 27.79047% while the fraction below 50microns was found to be 48.421% and a lead distribution of 9.462%. Table 5 show a detailed EDXRF result of particle size fraction bellow 50microns. The metallic minerals of aluminium, copper, zinc, potassium, calcium, titanium, chromium, manganese, iron, nickel, cadmium, barium, tungsten, rhodium, osmium and mercury were present in this sample as minor gangue minerals and all the size fractions revealed their presence at varying concentrations (Table 6).

Table 5: Assay result of raw samples after grinding (-50microns)

Oxides	Conc.(%)	Element	Conc.(%)
PbO	52.2	Pb	48.421
CuO	0.144	Cu	0.115
ZnO	0.01	Zn	0.008
Al ₂ O ₃	3.97	Al	2.101
SiO ₂	37.3	Si	20.888
SO ₃	0.85	S	0.340
K ₂ O	0.49	K	0.407
CaO	0.579	Ca	0.414
TiO ₂	0.083	Ti	0.050
Cr ₂ O ₃	0.095	Cr	0.065
MnO	0.002	Mn	0.002
Fe ₂ O ₃	1.32	Fe	0.922
NiO	0.061	Ni	0.048
CdO	0.95	Cd	0.831

BaO	1.3	Ba	1.165
WO ₃	0.25	W	0.198
Re ₂ O ₇	0.05	Re	0.038
OsO ₄	0.14	Os	0.105
HgO	0.19	Hg	0.176

Table 6: Assay result of raw samples after grinding (-710 +500microns)

Oxides	Conc.(%)	Element	Conc.(%)
PbO	24.2	Pb	22.45
CuO	0.058	Cu	0.05
Au	0.08	Au	0.08
Al ₂ O ₃	4.88	Al	2.58
SiO ₂	67.9	Si	38.02
SO ₃	0.2	S	0.08
K ₂ O	0.314	K	0.26
CaO	0.511	Ca	0.37
TiO ₂	0.089	Ti	0.05
Cr ₂ O ₃	0.035	Cr	0.02
MnO	0.021	Mn	0.02
Fe ₂ O ₃	0.528	Fe	0.37
NiO	0.0695	Ni	0.05
BaO	0.486	Ba	0.44
WO ₃	0.11	W	0.09
OsO ₄	0.082	Os	0.06
P ₂ O ₅	0.21	P	0.09
SeO ₂	0.061	Se	0.043

The EDXRF analyses puts average total silica as 58.9175% and this agrees reasonably well with the results (obtained earlier) from mineralogical study of polished sections. Zinc was not detected in most of the raw samples, (0 to 0.031%) while copper was found to be a minor element (0.044% to 0.17%). This implies that copper and zinc cannot be profitably processed from Baban Tsauni ore deposit since these values are below the cut-off grades of 4% and 0.4% for zinc and copper respectively [4].

Gold can be profitably processed when the grade is 0.0001% [4]. The Baban Tsauni ore deposit however has an average assay of 0.029381% as presented in Table 4. This value is

2938.1 times higher than the cut-off grade required for profitable exploitation of gold. Thus Baban Tsauni ore is a high grade gold ore.

4.0 Conclusion

The results of this work have shown that Baban Tsauni ore is a rich lead –gold ore in basement quartz host rock. The lead mineral in the ore is galena. Galena exhibited coarse grains sizes and simple locking pattern with the gangue mineral. The mineralogical characteristics of this ore suggest that it will respond well to beneficiation by gravity separation methods.

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