Characterisation of Oyster Shell for Neutralisation of Bio-Leached Effluent*

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Abstract

Characterisation studies of Oyster Shell (Mercenera mercenera) collected from coastal towns of Ghana and its neutralising effect on bio-leached effluent has been studied using XRF, XRD, Zeta Meter, BET and SEM/EDX. The study confirmed that OS contains high calcium equivalent to about 54% CaO. The OS consists mainly of aragonite (96.1%) and calcite (2.6%) which are carbonates hence OS can be used to neutralise any acid solution. OS is very hard to mill as it has high Bond work index of 48.54 kWh/t. The Zeta Potential analysis indicates that OS will not be stable below pH of 3 and above pH of 10. Therefore OS powder dissolved and raised the pH of bio-leached effluent from pH 1.85 to 6.0 in 30 minutes. The arsenic removal increased with increasing OS concentration. The morphological study revealed that the surfaces of the reacting particles were coated with precipitates like $FeAsO_4$ at pH of 4.5. Consequently, surface area of reacting powder increased from 4.15 m^2/g to 75.46 m^2/g. In a similar manner, the D_{50} decreased from 16.69 μm to 7.77 μm for the reacting particles at pH 4.5. Particle size distribution at pH 7.0 showed that the D_{50} of the OS material increased to 9.23 μm which can be due to coating of precipitates like $CaSO_4$ on the reacting particles during acid neutralisation. Mobile arsenic extracted from the precipitate averaged 6.42 mg/L as against the EPA maximum allowable concentration of 5.0 mg/L indicating that the precipitate formed is fairly stable.

Keywords: Effluent, Neutralisation, Oyster Shells, Characterisation, Work Index

1 Introduction

Extraction of gold from refractory ores is normally low (less than 50%) using conventional fine grinding and cyanidation (La Brooy *et al*., 1994). This is because the gold is in submicroscopic state in the sulphides and carbonaceous materials as such the gold cannot be sufficiently liberated by mechanical grinding. Thus, the sulphides are concentrated by froth flotation after milling (Goodall *et al*., 2005; O'Connor and Dunne, 1994; Kydros et al., 1993; Gharaei *et al*., 2014; Peng and Grano, 2010) and the concentrate obtained is pretreated to liberate the submicroscopic gold particles which are locked in the sulphide minerals by pyrometallurgical or hydrometallurgical methods. The pyrometallurgical route (oxidative roasting) oxidises the sulphidic sulphur and arsenic into gaseous materials while the gold is concentrated in the iron oxide (hemaetite) residue for gold cyanidation.

Unfortunately, pyrometallurgical treatment produces environmentally unfriendly compounds such as sulphur dioxide (SO_2) and arsenic trioxide (AS_2O_3) particulates (Robinson, 1988; Arriagada and Osseo-Asare, 1984). Consequently, the flue gases are unfriendly environmentally and therefore the process has been replaced in recent times by hydrometallurgical methods.

The preferred hydrometallurgical pretreatment method of flotation concentrate nowadays is biooxidation (Tributsch, 1999) which is found to be cheaper to operate than other method like pressure leaching (Berezowsky *et al*., 1991). Biooxidation employs microorganisms, such as, *Acidithiobacilli* species in the oxidation of the sulphide minerals in the concentrate at pH between 1.2 and 1.8 at temperature between 42 and 45 °C. Upon completion of biotransformation of the sulphide concentrate, liquid and solid separation is carried out in a counter current decantation (CCD) circuit. The effluent generated is, therefore, laden with arsenic, sulphate and other base metals ions and these ions must be removed from the effluent before impoundment in any resipotory (Du Plessis and Maree, 1994). The removal of arsenic in a form that is stable is important because arsenic is carcinogenic and can be mobilised into the geo-environment. Furthermore, the effluent is also acidic and hence must be neutralised.

Lime has been used traditionally in processing bio-leached effluent to convert arsenic to stable ferric arsenate and also neutralise the excess acid. Though lime is cheap, the freight charges are high, therefore, the cost of neutralisation is high. Furthermore, the neutalisation reaction is very fast, a situation that results in formation of unstable precipitate such as calcium arsenate (Pantuzzo and Ciminelli, 2010; Zhu *et al*., 2006; Swash and Monhemius, 1994). To reduce the reaction rate, calcium carbonate has been used in addition to lime in a-two stage neutralisation process of the bio-

leached effluent. (Hamester *et al.*, 2012, Bologo *et al.*, 2009; Maree and du Plessis, 1994).

There are currently about eleven biooxidation plants in the world and Ghana hosts two of the largest biooxidation plants operated by AngloGold Ashanti and Golden Star Resources. For these companies, oyster shells (Mecenera Mecenera) are used as a substitute for calcium carbonate. There is, however, little information on the elemental and mineral compositions, as well as, surface characteristics of the oyster shell. This study therefore seeks to characterise oyster shells and investigate its effect on neutralisation of bio-leached effluent.

2 Resources and Methods Used

2.1 Materials

Representative samples of Oyster Shells (*Mercenera mercenera*) were collected from coastal towns of Ghana. The oyster shells were washed with distilled water and then dried in an oven at 105 ^{o}C. A bio-leached effluent was obtained from a Biox Plant in Ghana.

2.2 Bond Work Index Determination

About 10 kg dried OS sample was used for Bond Ball Mill Work Index (BBMWI) determination; the standard procedure was utilised (Napier-Munn, 1999). The procedure used for the test was repeated over a number of cycles till 250% circulating load was obtained. Equation 1 was used for the BBMWI calculation.

$$W_i = \left(\frac{4.45}{P_{106}^{0233} \times (G_{pb})^{0.87} \times \left(\frac{1}{\sqrt{P_{80}}} - \frac{1}{\sqrt{F_{80}}} \right)} \right) \quad (1)$$

where:

W_i is work index
P_{106} is the size of test sieve used;
G_{pb} is the net grams undersize per revolutions;
P_{80} is the product 80% passing size in microns; and
F_{80} is the feed 80% passing size in microns.

2.3 Pulverisation of Oyster Shell

The pulveriser used was Labtenics LM 2 Laboratory Pulverising Mill mounted on tough spiral springs. The material was milled in batches using C-2000 bowl with a disc (steel) as grinding medium and each sample was milled for 15 min. The mill was operated at a pressure of about 482.63

KNm^{-2}. The pulverized sample was used for the following:

2.4 Particle Size Distribution

The particle size of the powdered OS sample was measured using Mastersizer 2000 (Malvern Instruments Ltd, UK). The instrument functions on the principle of static light scattering as monochromatic light interacts with particles suspended in water at room temperature. In the process, the particles diffract light proportional to their size. Thus, the diffraction patterns are then cumulatively processed to achieve the volume and mass size distribution of the particles.

2.5 Surface Area Determination

The specific surface area of OS was measured by a 5-point N_2 adsorption BET method (Anon, 2015; Isa *et al.*, 2014; Ticknor and Saluja, 1990; Brunauer *et al.*, 1938) using Germini 2390 BET analyser. About 0.5 g representative sample was dried at 80 ^{o}C for 24 hr in order to expel moisture and gases before measurements. The average value of the adsorption and desorption specific areas of the particles were considered as the real values.

2.6 Zeta Potential Determination

Interfacial chemistry of the OS particles in KNO_3 (electrolyte) solution was determined by measuring the zeta potential (ζ) of the particles as a function of pH using Acoustosizer II Colloidal Dynamics equipment. The zeta potentials displayed good reproducibility within ±2.5 mV standard deviation at a confidence limit of about 0.1. The first zeta potential was calculated by fitting the theory to electroacoustics spectra with ESA size (or attenuation size if there is no ESA size). The second zeta potential was the Smolluchowski zeta which was also calculated from low frequency only using thin double layer static Smoluchowski formula which does not account for particle inertia or effects of concentration.

2.7 Elemental and Mineralogical Investigations

The elemental compositions of the OS were determined using X-ray Fluorescence (SECTROX-LAB-2000 XRF machine). In the process, about 4 g of OS sample was weighed and about 0.9 g of non-reactive adhesive gum was then added and mixed thoroughly for about 3 min. The mixture was thereafter pressed into pellet using a 68.95 KNm^{-2} laboratory hydraulic pressure device. The pellet was then placed in the XRF analyser where the elements in the sample were measured.
The mineralogical composition was determined using Scintag ARL X'tradiffractometer CuKα

radiation produced at 10 kV and 10 mA. Prior to measurement, approximately 10 wt% ZnO was added to the sample to ensure calculation of the amorphous content of the sample. The X-ray diffraction traces were collected between 5° and 90° 2θ at 0.02^{o} intervals at a rate of 0.05^{o} per min. The mineral identification and quantification were conducted using XPowder and SIROQUANT V3 softwares. SEM analysis was conducted using the Zeiss Evo Scanning Electron Microscope equipped with a Backscattered Electron (BSE) image detector.

2.8 Neutralisation of Bio-Effluent with Oyster Shell Powder

About 10 g/L, 20 g/L, 30 g/L, 40 g/L, 50 g/L and 60 g/L solid concentrations of OS were added to the effluent for neutralisation at near room temperature (about 27 ^{o}C) and stirred at 300 rpm. The pH and redox potentials of the slurries were monitored and measured at 30 min intervals for 300 min. The samples were filtered at the end of the period using 0.45 µm filter membrane. The filtrates were stored in clean labelled sample bottles for arsenic analysis.

2.9 TCLP Extraction Process

About 50 g (M_0) of dried OS precipitate was weighed into a 2 L volumetric flask and the mass was recorded. About 1L of the extraction fluid was added to the flask containing the sample. The flask was then closed with a stopper and clamped firmly on a lateral agitator and then allowed to agitate vigorously for 20 hours. At the end of shaking, solid/liquid separation was carried out with a 2 µm glass fibre filter. The filtrate was collected into a known weight 1 L beaker (M_1). The weight of the beaker and TCLP extract were weighed and recorded as M_2. The pH of the TCLP extract was measured and recorded.

In the case where the extract pH $\geq$ 2.0, concentrated nitric acid was added slowly to bring down the pH to below 2.0. The acid was added to preserve the TCLP extract.

The mass of the beaker containing the preserved extract was measured as M_3. The extract was thereafter bottled and submitted for arsenic analysis.

Concentration of arsenic in TCLP extract,

$$[As]_{TCLP} = \frac{AV*(M_3 - M_1)}{M_2 - M_1} \qquad (2)$$

where:

Assay Value (AV)

Mass of preserved extract= $M_3 - M_1$ and

Mass of TCLP extract= $M_2 - M_1$

$$[As]_{Mobile} = \frac{AV*(M_3 - M_1)}{M_0} \qquad (3)$$

# 3 Results and Discussion

3.1 Grindability Tests

The work index measured for the OS sample was 48.54 kWh/t which indicates that the OS sample was about four times harder than pure calcium carbonate sample (13.52 kWh/t) as given in the literature (Napier-Munn, 1996) and therefore requires much higher energy to efficiently mill the material.

3.2 Particle Size Distribution (PSD) of Powdered OS Sample

Particle size distribution (PSD) performed on the OS sample showed that, after 15 mins of pulverisation, the laser diffraction (Malvern Mastersizer X, Malvern UK) size analysis gave 10th, 50th and 90th percentiles denoted as D_{10}, D_{50} and D_{90} respectively of the OS sample as 0.925 µm for D_{10}, 16.689 µm for D_{50} and 103.538 µm for D_{90}. Furthermore, the specific surface area measured using BET Analyser was 4.15 m^2/g. It can be inferred from BET analysis that the oyster shell is not porous hence the surface area is not very high. This also explained the high Bond work index of the oyster shell.

3.3 Zeta Potential of Powdered OS Materials

The particle Zeta Potential of OS powder was negative at pH of about 11 (Fig 1) and increased to zero at pH of 10.5 which is the material's isoelectric point (iep). This confirms work done by Tsunekawa and Takamori (1987) on $CaCO_3$ to be between pH 10.5 to 11. It can therefore be inferred that OS has similar surface chemical properties to pure $CaCO_3$. From pH 10.50, zeta potential increased in the positive potential region and increased in magnitude from the alkaline region until ~ pH 7. The zeta potential then conscripted until pH 6 when it increased until pH 3. On the reverse sweep, the zeta potential increased in the opposite direction thus making the sweep incongruent. The zeta potential rose in magnitude as the pH sweep went from pH 3 to 5 from where it dropped at pH 6 and remained unchanged until pH 9 after which the magnitude inched up slightly at pH 10 (Tsunekawa and Takamori, 1987). It can be inferred that OS will not be stable below pH of 3 and above pH of 10.
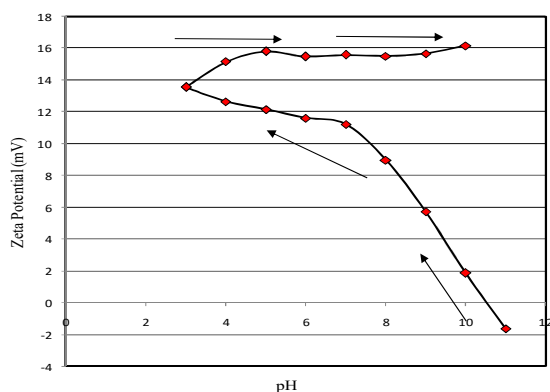
Fig. 1 Zeta Potential of 8 wt. % Solid OS in 10^{-3} M KNO_3 as a Function of Solution pH. (The arrows indicate the direction of pH sweep)

3.4 Chemical and Mineralogical Composition of Oyster Shell

3.4.1 Elemental Analysis of Oyster Shell

The elemental constituents of the OS are shown in Table 1. The major element identified was Ca (38.92%) with others such as Na, Si, Al, K, Mg, Mn, Fe and S each below 0.7%. Other minor elements measured in ppm include Strontium (904.6 ppm), Ba (315.4 ppm), V (154.6 ppm), Ti (53.2 ppm) and Cr (46.5 ppm). The relatively high level of strontium can be attributed to the high presence of calcium. It is well known that strontium is strongly associated with calcium (Tabouret *et al*., 2010). Other minor elements detected were below 50 ppm.

The major oxides identified in the sample as shown in Table 2 indicate 54.50% CaO present in Oyster Shell and about 15% SO_3 as trace element (Medvediev *et al*., 2014). Other oxides measured were below 2%.

Table 1 Elemental Compositions of OS

Elements Identified	Units %	Elements Identified	Units Ppm
Na	0.542	V	154.6
Mg	0.046	Cr	46.5
Al	0.054	Co	5.8
Si	0.264	Ni	12.2
P	0.031	Cu	11.9
S	0.151	Zn	1.9
Cl	0.0001	Ga	2.9
K	0.050	As	4.4
Ca	38.929	Rb	1.8
Ti	0.005	Sr	904.6
Mn	0.044	Mo	1.0
Fe	0.16	Sb	<0.0009
		Ba	315.4

Table 2 Major Oxides

Elements	Oyster Shell (%)	Elements	Oyster Shell (%)
Na_2O	1.46	Cl	0.001
MgO	0.076	K_2O	0.121
Al_2O_3	0.205	CaO	54.50
SiO_2	0.565	TiO_2	0.009
P_2O_5	0.141	MnO	0.057
SO_3	15.06	Fe_2O_3	0.046

3.4.2 Mineralogical Composition of Oyster Shell

The major minerals identified in the Oyster Shell sample were aragonite (96.1%) and calcite (2.6%) (Table 3). Aragonite is known to be a polymorph of calcite (calcium carbonate) (Klein and Dutraw, 2008). An amorphous content of 1.3% was observed in the Oyster Shell sample.

Table 3 Mineral Composition of OS

Weight %	OS
Aragonite (CaCO)	96.1
Calcite (CaCO$_3$)	2.6
Amorphous	1.3

Scanning Electron Microscopy result shows that, the particles of the OS sample were mostly fine and were interspersed with few large plate-like surfaces of the particles. The morphology of the sample also possessed broader dark spots which can be attributed to the presence of non-metals in the sample. EDS spectra of OS collected are represented in Fig 3, Fig. 4 and Fig. 5 of the spots in Fig. 2.

X-ray spectra collected on OS sample at spot A showed the presence of Ca, C and O (Fig. 3) as the main elements present which confirms the phenomenal dominance of aragonite and calcite in the OS as is shown in the XRD analysis. Analysis of spot B gave the spectrograph in Fig. 4 in which elements such as Ca, Al, Si, K, Na, V and O were identified. Apart from V, all the other elements combine to form muscovite. The spectrum showed that vanadium muscovite (Houzar and Šrein, 2000), if present may be a rare mineral in the Oyster Shell material and below detection of XRD or amorphous in nature. From Fig. 5, elements such as Ca, Si, Al, Na, C, and O were detected at spot C during the spectral observation. The elements present are an indication of the possible presence of a neutralising agent ($CaCO_3$) which confirms the reason why Oyster Shell can be used as an acid neutralising material.
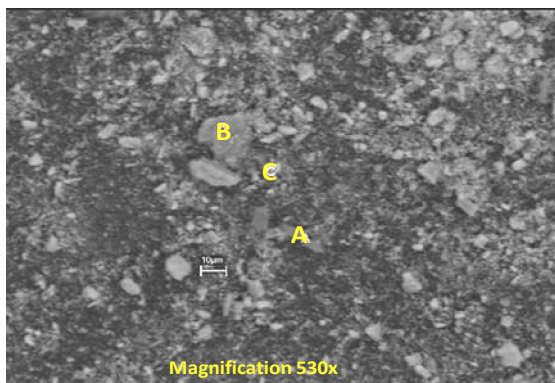
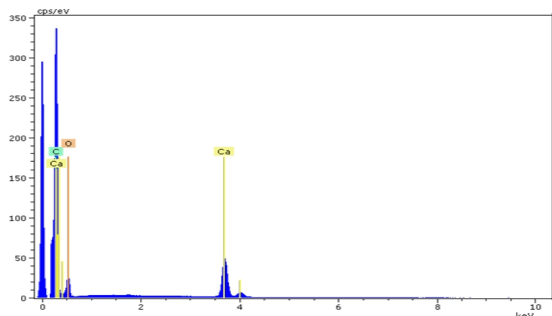**Fig. 2 BSE Image of Oyster Shell Sample**
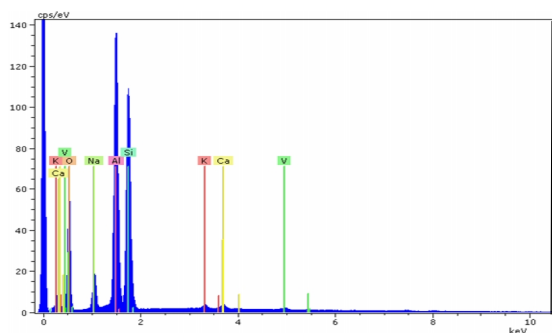

Fig. 3 Spectrograph of Spot A Fig. 2

Fig. 4 Spectrograph of Spot B in Fig. 2
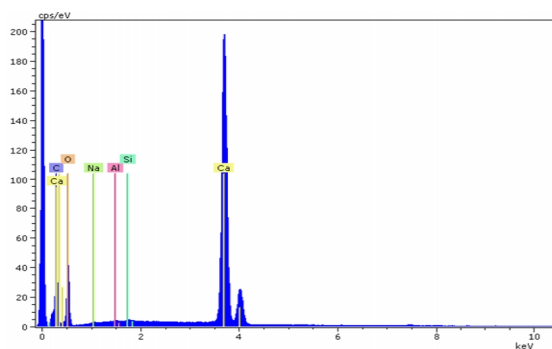
Fig. 5 Spectrograph of Spot C located in Fig. 2

3.5 Composition of Bio-Leached Effluent

Speciation of the bio-leached effluent was conducted to identify and quantify ions present. The elemental composition is presented in Table 4. The total iron (which consists of Fe^{3+} and Fe^{2+} ions) measured was 4541 mg/L and the pH of the effluent was 1.85 with redox potential (Eh) above 500 mV in the presence of *Acidithiobaccili* species. Magnesium measured in the effluent was 1560 mg/L which may have been solubilised from dolomite present in the flotation concentration biooxidised. Arsenic (As^{5+} and As^{3+}) measured was 1276 mg/L because of biooxidation of arsenopyrite. The sulphate concentration was 8400 mg/L which was primarily H_2SO_4. The high acidity results in low pH of 1.85 can be explained according to the Eh-pH diagram of S-O-H of sulphur system (Brookins, 1987) can be attributed to pyrite and arsenopyrite minerals treated. The high sulphur solubilisation of 16760 mg/L was due to microbial transformation of sulphur during biooxidation process (Rohwerder and Sand, 2003).

Aluminium concentration in the bio-leached effluent was 150 ppm which may exist in ionic forms as Al^{3+} or $AlOH^{2+}$ according to Eh-pH diagram of Al-O-H system (Brookins, 1987). The calcium content measured was 600 mg/L as Ca^{2+} (Brookins, 1987). The presence of phosphorus (3.5 mg/L) may have resulted from addition of phosphoric acid during the biooxidation process as a nutrient requirement of the microorganisms. Silicon measured was 110 mg/L which may have been stabilised in the effluent as H_2SiO_4 as can be shown on Eh-pH diagram of Si-O-H system (Brookins, 1987). The Total Organic Carbon (TOC) measured in the effluent was 21 mg/L which can be due to the presence of organic matter of the material treated and biomass of the microorganisms (Rawlings *et al*., 2003). Trace elements measured in the effluent were vanadium and chromium and many others as shown in Table 5. It can be inferred that, the high levels of ions particularly arsenic in the effluent will pollute the environment; hence the effluent must be treated to remove the toxic ions.

Table 4 Major Components of Bio-Leached Effluent

Elements	Al	As	Ca	Fe	Mg	Mn	P	S	Si
Concentration (mg/L)	150	1276	600	4541	1560	200	3.5	16760	110

Table 5 Trace Elements measured in Bio-Leached Effluent

Elements	Concentration (mg/L)	Elements	Concentration (mg/L)
V	24	Cd	3.4
Cr	54	Sn	0.9
Co	1.3	Cs	6.5
Ti	0.09	Ba	8.9
Ni	2.9	La	13
Cu	2.0	Ce	18
Zn	1.7	Hf	1.6
Ga	0.6	Ta	1.6
Rb	0.2	Pb	1.1
Y	1.7	Bi	0.7
Zr	0.5	Th	0.7
Nb	1.3	U	2.2

3.6 Influence of OS Solid Loading on Effluent Neutralisation

The OS sample neutralisation reactions were highly exothermic and it was observed that the OS material reacted very vigorously resulting in a very fast rise of pH from 1.85 to about 6.0 within 30 min for all solid concentrations (Fig. 6). It was observed that, increasing concentration of OS material in neutralisation increases effervescence and carbon dioxide liberated. There was however minimal change in pH after the reactions reached pH 6.0 even with increased solid loading. The marginal change in pH after pH 6.0 can be attributed to formation of precipitates ($CaSO_4$) which passivated the reacting surface of the particles.
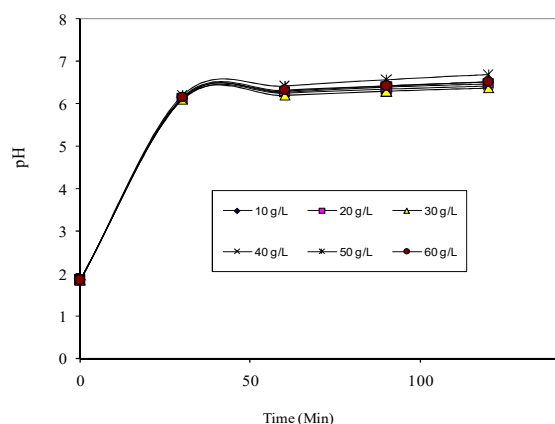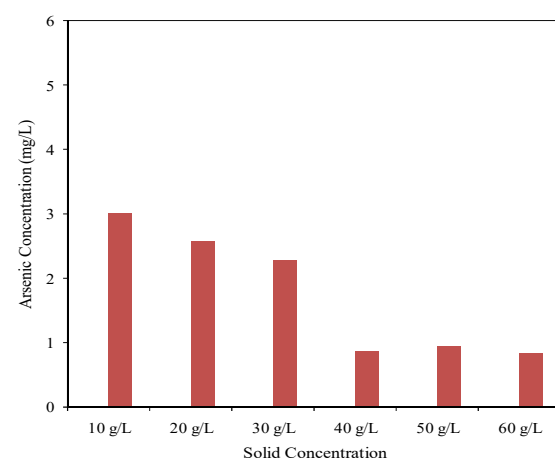
Fig. 6 Influence of Oyster Shell Solid Loading on pH during Neutralisation Time of Bioleached Effluent

3.7 Influence of OS Solid Loading on Arsenic Precipitation

The various solid loadings of OS used in the neutralisation of the effluent within the initial two hours is represented in Fig. 7. During the investigation, lime was added to raise the pH to 7.0. It can be observed that arsenic removal from solution increased with increasing solid concentration of OS between 10 g/L and 30 g/L. Thereafter, arsenic removal almost became steady from 40 to 60 g/L solids concentrations. Lime addition to complete acid neutralisation did raise the pH to 7.

Fig. 7 Influence of OS Solid Loading on Arsenic Precipitation from Bioleached Effluent

3.8 Stability of Precipitates

The stability of precipitates formed from neutralisation of bio-leached effluent with OS material were investigated using Toxicity Characteristic Leaching Procedure (TCLP). The result showed mobile arsenic concentration of 6.42 mg/L indicating that the precipitates that are formed can be re-leached in tailings impoundment.
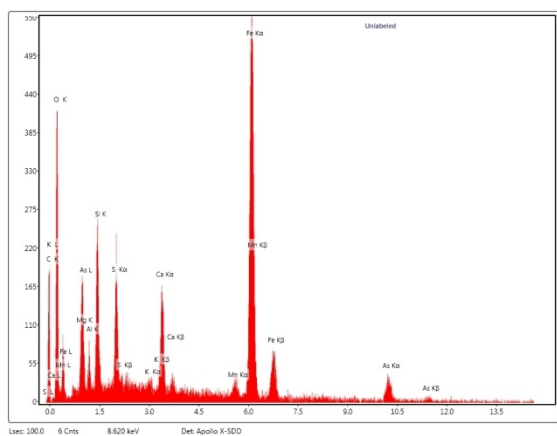
3.9 Morphology of Precipitates formed at pH 4.5

The morphology of the reacting OS at pH 4.5 is presented in Fig. 8. The surfaces of the particles were rough which depict formation of precipitates, on the particles surfaces. The precipitates thus passivated the reacting sites of the OS material thereby curtailing the reaction at pH of about 6.0. The EDS spot analysis (Fig. 9) shows that, the elements identified on the precipitate include 20.18% Fe, 2.46% Ca, 3.88% Si, 2.41% S, 4.37% As and 38.05% O, 25.77% C, 1.03% Mg, 0.62% Al, 0.73% Mn and 0.23% K. The molar values

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indicate that $FeAsO_4$ is predominantly present in the precipitates.

The high concentration of Fe, Ca, S, As and O in the precipitate confirmed removal of arsenic by Oyster Shell from the bio-leached effluent.
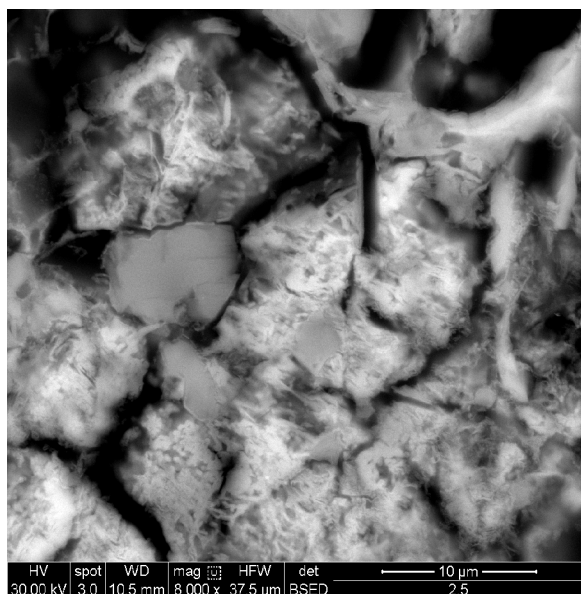

Fig. 8 Backscattered Electron Image of Precipitates at pH 4.5 at 8000x Magnification


Fig. 9 Spectrum of OS Precipitate at pH 4.5

3.10 Analysis of Precipitates Formed at pH 7.0

The shapes of the particles at magnification of 530X as shown in Fig. 10, were cylindrical and others were plate-like with rough surfaces and intermittent dark spots which can be attributed to the presence of non-metallic compounds. The EDS analysis obtained at spots A and B (Fig. 10) showed dominant elements as Fe, Mn, Ca, Si, As, S and O as is depicted in Fig. 11 which may be arsenate and sulphate compounds.
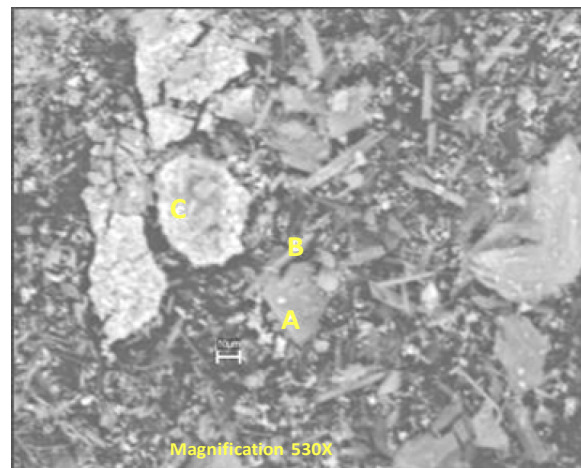
Fig.10 BSE Image of Precipitate formed at pH 7.0
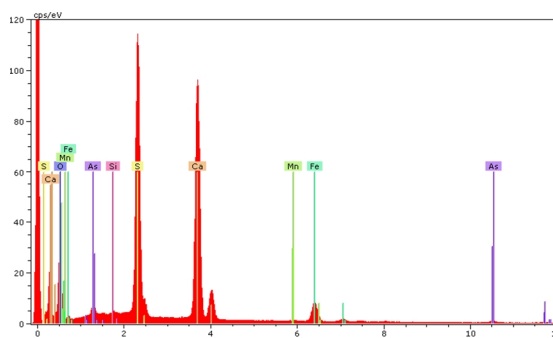
Fig. 11 Spectrum at Points A and B of Precipitate at pH 7.0

3.11 Specific Surface Area of Precipitates

The specific surface area measured for OS neutralised precipitate was 75.46 m^2/g after neutralisation at pH 7.0. The high specific surface area exhibited by the OS sample showed that, the material dissolved significantly and fast leading to a reduction in size and that the precipitates were also fine and this corroborate the work by Allen (1992) that, the higher the surface area of precipitates after a chemical reaction, the faster the material reacted.

3.12 Particle Size of Reacting Particles

The particle size distribution of reacting particles at neutralisation pH of 4.5 showed that, the D_{50} reduced from16.69 µm to 7.77 µm which can be attributed to dissolution of the OS. However, particle size distribution at pH 7.0 showed that the D_{50} of the OS material increased to 9.23 µm which can be due to coating of precipitates on the reacting particles during acid neutralisation.

4 Conclusions

The study confirmed that OS contains high calcium equivalent to about 54% CaO. The OS consists

mainly of aragonite (96.1%) and calcite (2.6%) which are carbonates hence OS can be used to neutralise any acid solution. It can be concluded that, OS is very hard to mill as it has high Bond work index of 48.54 kWh/t. The Zeta Potential analysis indicates that OS will not be stable below pH of 3 and above pH of 10. Therefore OS powder dissolved and raised the pH of bio-leached effluent from pH 1.85 to 6.0 in 30 minutes. The arsenic removal increased with increasing OS concentration. The morphological study revealed that the surfaces of the reacting particles were coated with precipitates at pH of 4.5.

Consequently, surface area of reacting powder increased from 4.15 m^2/g to 75.46 m^2/g. In a similar manner, the D_{50} decreased from 16.69 µm to 7.77 µm for the reacting particles at pH 4.5. Particle size distribution at pH 7.0 showed that the D_{50} of the OS material increased to 9.23 µm which can be due to coating of precipitates like $FeAsO_4$ and $CaSO_4$ on the reacting particles during acid neutralisation. Mobile arsenic extracted from the precipitate averaged 6.42 mg/L as against the EPA maximum allowable concentration of 5.0 mg/L indicating that the precipitate formed is fairly stable. It is found that OS can be used as base to neutralise bio-leached effluent.

Acknowledgements

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