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Extraction of copper from copper slag: Mineralogical insights, physical beneficiation and bioleaching studies

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Abstract–Copper slag was subjected to in-depth mineralogical characterization by integrated instrumental techniques and evaluated for the efficacy of physical beneficiation and mixed meso-acidophilic bioleaching tests towards recovery of copper. Point-to-point mineral chemistry of the copper slag is discussed in detail to give better insight into the association of copper in slag. Characterization studies of the representative sample revealed the presence of fayalite and magnetite along with metallic copper disseminated within the iron and silicate phases. Physical beneficiation of the feed slag (~0.6% Cu) in a 2 L working volume flotation cell using sodium isopropyl xanthate resulted in Cu beneficiation up to 2-4% and final recovery within 42-46%. On the other hand, a mixed meso-acidophilic bacterial consortium comprised of a group of iron and/or sulfur oxidizing bacteria resulted in enhanced recovery of Cu (~92-96%) from the slag sample. SEM characterization of the bioleached slag residue also showed massive coagulated texture with severe weathered structures. FE-SEM elemental mapping with EDS analysis indicated that the bioleached residues were devoid of copper.

Keywords: Copper Slag, Characterization, Microscope, Physical Beneficiation, Bioleaching

INTRODUCTION

Metallurgical and mineral processing industries generate a huge amount of wastes in the form of fines, slimes, slag, sludge etc., thereby creating environmental problems with ecological imbalances. Over decades, the primary aim has been directed towards the development of a zero waste technology by the utilization of such by-products or other industrial wastes. Simultaneous extraction of valuable metals by means of an ecofriendly technology is highly emphasized to bring in additional revenues to the producing industries [1,2]. Industrial wastes such as blast furnace slag from steel plants and fly ash from thermal power plant have earned huge applicability as an additive in cement making [3]. Still, several other wastes from the steel industries like LD (Linz - Donawitz) slag, LD dust, flue dust, sludge, iron ore slimes and red mud from aluminum industries have gained a reputation for their numerous applications as a value-added product [4]. Transformation of such solid wastes from one form to another in view of its valorization either by the same production unit or by a different industrial installation has thus become very essential not only for conserving metal and mineral resources but also for protecting the environment.

Copper ore globally at an average contains ~1% copper and the rest being silica, alumina, calcium, iron and magnesium. The cost of production of copper is very high due to the involvement of complicated steps right from ore processing to metal production. Although, the entire crush-grind-float treatment process to recover metal value is cost effective, recovery of any additional metal values from the generated wastes by a cost effective technique is highly desirable to earn additional revenue for the copper industry. One such material to be considered is the copper slag which is being produced during smelting and converting steps of copper matte production. It has been roughly estimated that for every ton of copper metal produced, about 2.2 tons of slag is generated causing several environmental and space/land (dumping) problems. The common management options for copper slag are recycling, recovering of metal, production of value added products such as abrasive tools, roofing granules, cutting tools, abrasive, tiles, glass, road-base construction, railroad ballast, asphalt pavements [5] etc. One of the greatest potential applications for reusing copper slag is in cement and concrete production [3].

Some investigation on slag has indicated that an appreciable amount of Cu, Co, Ni can be recovered by hydrometallurgical and flotation techniques [6-9]. However, hydrometallurgical applications for recovery of metal values from slags are not encouraging. For example, high concentration acid leaching has shown problems of the formation of silica gel, which induces an increase of leach liquor viscosity, difficult pulp filtration and crud formation during solvent extraction [7]. Therefore, from an environmental and economical

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point of view, the use of hazardous chemicals, high temperature/ pressure leaching and difficult downstream processing [7,10,11] makes the process unattractive. Owing to the problems of metal recovery from conventional methodologies such as pyro- and/or hydrometallurgy, the use of some selected microorganisms is gaining momentum in diverse areas to recover metal values from various industrial wastes over the past few years in an ecofriendly and economic way. The use of several acidophilic chemolithoautotrophic microorganisms has shown promising results to recover metal values from low grade ores of several mines [12,13], electronic wastes [14,15], fly ash [16,17], red mud [18], waste battery material [19], in coal desulfurization [20] and also in remediation of heavy metal contaminated soils [21] etc. As of now, bio-hydrometallurgical applications have been the most preferred route of metal extraction using such microorganisms [22,23].

In India, there are some copper producing plants, but hardly any effort has been made to recover any valuable material from copper slag. Very recently, Das et al. [24] reported the use of flotation technique to recover copper from a slag material produced from one of the copper producing plants situated at Dehaj, under Birla Copper, India. A detailed mineralogical characterization can put better insights into the association of copper in slag. Therefore, in the present study, a copper slag from one of the working plants in India was subjected to a comprehensive mineralogical characterization, followed by studying the copper recovery using flotation technique and preliminary shake flask bioleaching method. The original and bioleached residues of the slag sample were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), QEMSCAN (quantitative evaluation of minerals by scanning electron microscopy) and field emission scanning electron microscope (FE-SEM) coupled with energy dispersive X-ray spectroscopy (EDS) to determine the association of copper which can be recovered by the most convenient as well as economic way.

MATERIALS AND METHODS

1. Slag Sample

The original copper slag, i.e., the "as received" sample, was coarse with some fine grained, rounded to sub-rounded particles varying from 2000 to 75 microns in size with a black colored appearance. The material was subjected to sampling by Coning and Quartering methods for mineralogical characterization and chemical analysis. A representative sample of the slag after fine grinding was subjected to treatment with aqua regia, and the liquid obtained after digestion was analyzed by atomic absorption spectrometry (AAS model - Perkin Elmer AA-400) for chemical analysis.

2. Mineralogical Characterization of Copper Slag

A good representative of the copper slag sample after coning and quartering methods was taken up for detailed characterization studies. Characterization through XRD and electron microscope was done independently. X-ray diffraction studies were carried out by Philips X-ray diffractometer using Cu-Kα radiation (PANalytical, X'pert) equipped and operated at 40 kV and 30 mA to investigate the different mineral phases present in the sample. The mineralogical studies were carried out with the help of optical microscope (Leitz German make). Polished sections were prepared as per standard section preparation technique. The polished sections were studied using QEMSCAN (M/s. Intellection Pty. Ltd., Australia) instrument. QEMSCAN is considered as an extremely versatile SEM-based automated mineralogical analysis system that provides a rapid, digital, quantitative morphological and mineralogical analysis of the sample. The system works by locating particles that are scanned by the electron beam at a pre-determined resolution (from 0.2 μm upwards).

The method of scanning of the polished sample blocks in QEMSCAN was through the traced mineral search mode. Mineral chemistry was determined by spot analysis (point to point) on minerals by energy dispersive spectrometer (EDS) attached to the SEM. The resulting X-ray and backscattered electron signals were compared with a comprehensive "look-up" table of known minerals and the subsequent chemical phases formed the mineralogical identification data.

3. Physical Beneficiation Studies

Flotation studies were carried out using <100 micron sample grounded in a laboratory. A Denver D-12 sub-aeration flotation cell of 2 L capacity was used in all the flotation studies. The most widely used reagents for copper, i.e., Sodium silicate, isopropyl xanthate, and methyl isobutyl carbinol (MIBC), were used as dispersant, collector and frother, respectively. The ground sample was conditioned initially for a specified interval with 40% solids, collector and frother. The feed solid concentration (25%), impeller speed (1,200 rpm), pulp level, pH and froth collection time were maintained constant for all the experiments. The flotation optimization studies were carried out by varying sodium silicate 200-500 g/ton, frother 50-200 g/ton and collector 200-900 g/ton of reagent quantity. The generated flotation concentrate and tailings from the experiment were filtered, dried, weighed and analyzed for copper values to assess product quality and recovery values.

4. Microbial Leaching Studies

4-1. Microorganisms and Culture Conditions

A mixed consortium of acidophilic bacteria is considered more competent for bioleaching than a pure culture [25]. In the present study, we used a laboratory stock culture of mixed meso-acidophilic microbial consortium comprising predominantly of the *At. ferrooxidans* strains along with strains of *L. ferrooxidans* and *At. thiooxidans* were use as the inoculum for bioleaching studies [13]. The standard medium ($9K^+$) of Silverman and Lundgren containing $(NH_4)_2SO_4$ - 3 g/L, KH_2PO_4 - 0.5 g/L, $MgSO_4{\cdot}7H_2O$ - 0.5 g/L, KCl - 0.1 g/L and $FeSO_4$ - 44.2 g/L was used as the growth medium of the microbes [26]. Prior to bioleaching studies, repeated sub-culturing in $9K^+$ medium was done to ensure activation of the strains. The strains were adapted to Cu (1 g/L) [13] and after attaining a steady iron oxidation rate (IOR) of 600 kg/m^3/h were used for bioleaching studies.

4-2. Meso-acidophilic Bioleaching Studies: Optimization of Parameters for Copper Recovery

Bioleaching studies for the recovery of copper from the ground slag sample were carried out in 250 mL Erlenmeyer flasks using the adapted acidophilic microbial consortia at 35 °C. 10 mL each of the fully active microbial consortia was used as inoculums, which were suspended in 90 mL of $9K^+$ medium in each flask. The flasks were kept static to limit excess oxygen content in the medium. However, periodical shaking of the flasks manually for nearly 5-10 mins on an average of every 24 hours was carried out to avoid clumping of the material on one hand and ensure proper mixing of the sample with the acidified bacterial lixiviant on the other. The pH of the medium was adjusted to the desired values following dispersment or disturbance of any formed clumps and also to maintain the required medium acidity. However, the total incubation time varied with varying test conditions. The effect of various bioleaching parameters such pH, pulp density (w/v), inoculum percentage (v/v) and concentration of Fe (II) in the growth media were optimized under lab scale conditions to put an preliminary insight into the Cu dissolution process. The control set of experiments was carried out with $9K^+$ medium without any inoculum addition (Conditions: 10% pulp density, pH 2.0). Further, $HgCl_2$ was added to the control set to avoid any contamination [27]. The samples were periodically withdrawn for the analysis of copper by AAS (atomic absorption spectrophotometer) [13]. Following sampling, any extra loss by evaporation was readjusted by the addition of distilled water.

4-3. Analysis of Copper in the Bioleach Liquor

The concentration of copper in the bioleach liquor was analyzed by AAS (Perkin Elmer Model AA-400). The pH of the bioleaching medium under different experimental conditions was constantly monitored by a pH meter (Digital Systronics µ pH meter model 361 provided with a combined glass electrode) and was periodically adjusted with dilute H_2SO_4. Fe (II) and Fe (total) content in the media during bioleaching was analyzed by volumetric titration method using BDAS (Barium diphenylamine sulfonate) as an indicator [13].

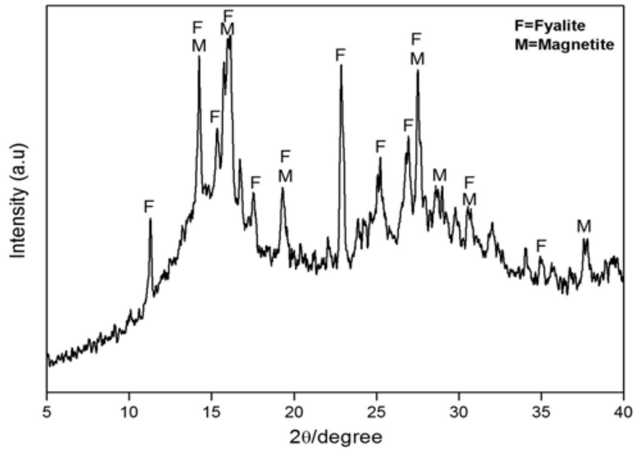

Fig. 1. XRD pattern of copper slag sample.

RESULTS AND DISCUSSION

1. Mineralogical Characterization of the Slag Sample

A qualitative mineralogical analysis using XRD is shown in Fig. 1. As can be seen, fayalite (F), an iron orthosilicate of the olivine family along with magnetite (M), are the major mineral phases for this sample. The association of any copper mineral could not be identified by this technique. Chemical analysis of the representative slag sample indicated 0.6% Cu content with 42.8% Fe, 28.4% SiO_2

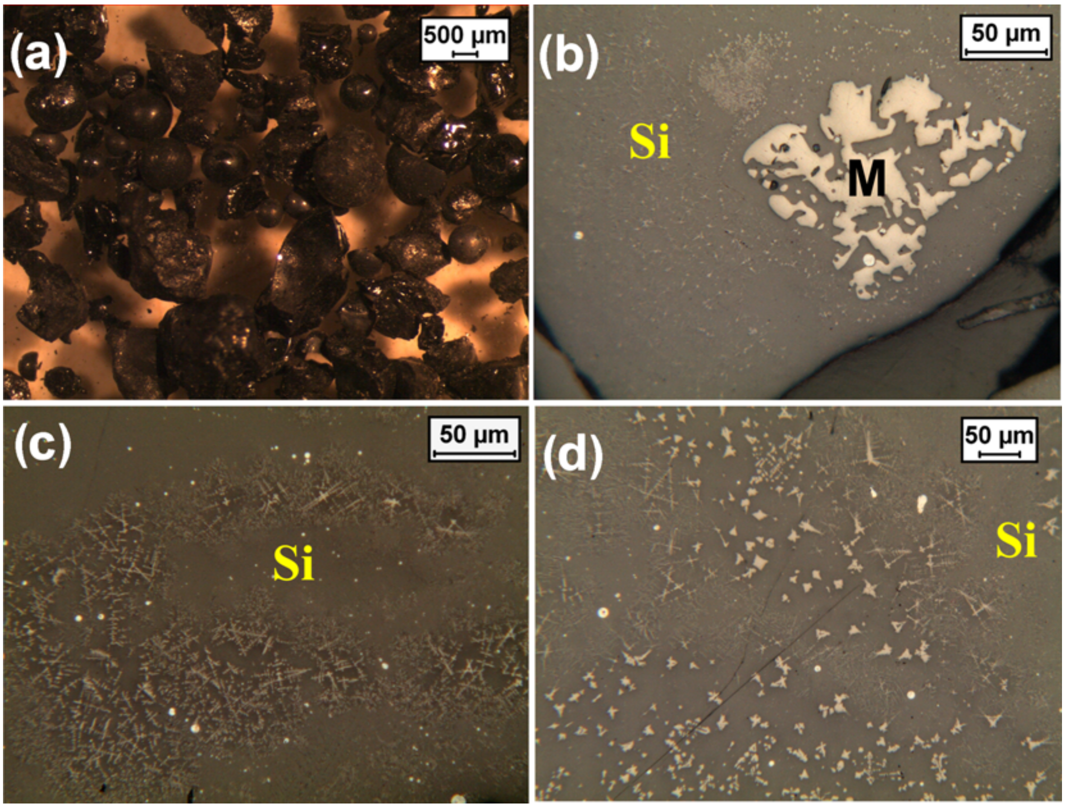

Fig. 2. (a) Black spherical ball shaped particles along with other shaped granules. (b) Patches of magnetite (M) occur with the silicates (Si). (c) The magnetite exhibit thin lath shape within the silicates (Si). (d) Magnetite occurring as stars within the silicates (S). "b, c and d" are the reflected light photomicrographs.

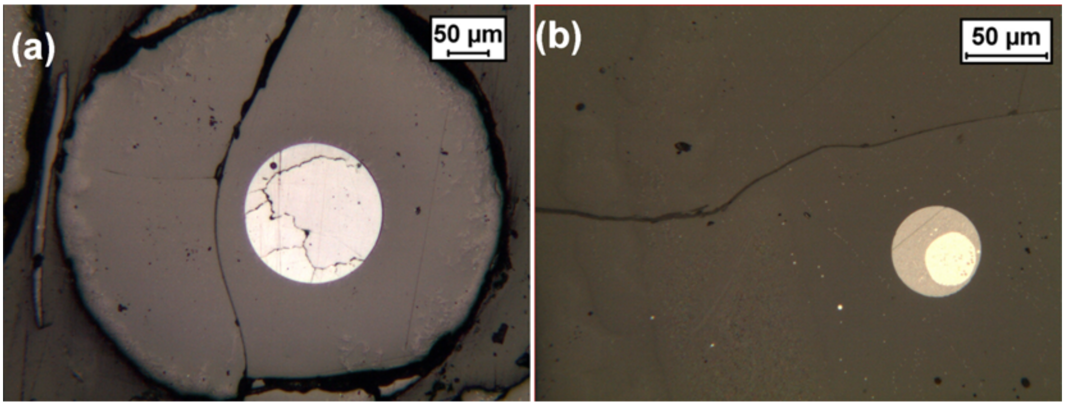

Fig. 3. (a) The metallic phases occur as spherical (white) within the silicates. (b) Two different compositional types of spherical metals occur one in another as inclusion.

and 2.0% Al_2O_3. The higher amounts of iron and silica validate the mineralogical finding that the sample contains magnetite and fayalite.

Mineralogical studies with the help of optical microscope indicated that the copper slag sample consisted of black ball shaped granules (Fig. 2(a)). Reflected light microscopic studies revealed that the sample consisted of three different phases: magnetite, fayalite (that is in agreement to XRD) and two different spherical shaped metallic phases. In the sample, magnetite was seen to be present as patches, as fine laths showing dendritic texture and stars of various shapes and sizes within the fayalite phase. On the other hand, the fayalite phase was present as patches that contained some spherical metallic phases (present as round white dots) in addition to the inclusions of magnetite (Fig. 2(b), 2(c) and 2(d)). Fig. 3 provides more insights into the appearance of some of the metallic phase's occurring within the slag sample. As can be seen from Fig. 3(a), the metallic phase (mostly spherical) was present as inclusions within the patches of fayalite or spherical shaped fayalite. The metal spherical balls were mostly of two types that were clearly distinguished

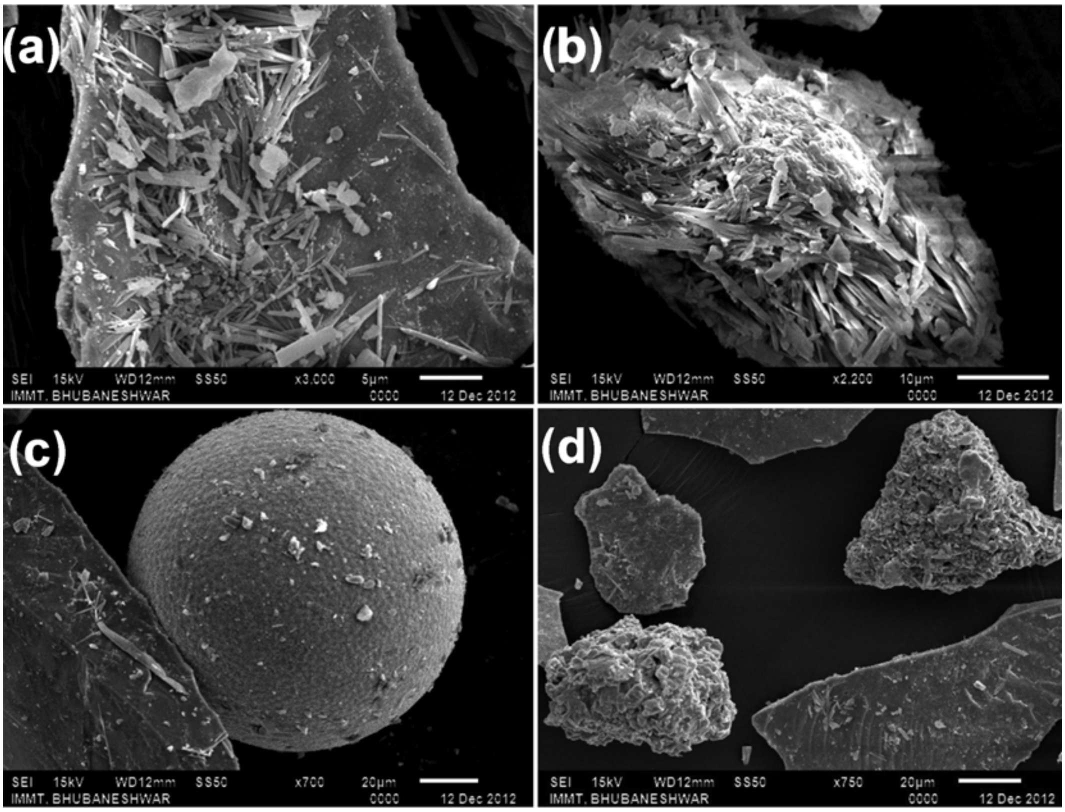

Fig. 4. Scanning electron photomicrographs of the copper slag. (a) Acicular needle type fayalite crystals embedded in a glassy phase. (b) Acicular needle type fayalite coagulated forming an agglomerate of the crystals. (c) Spherical ball shaped materials. (d) Slag showing flat, porous and platy shaped grains.

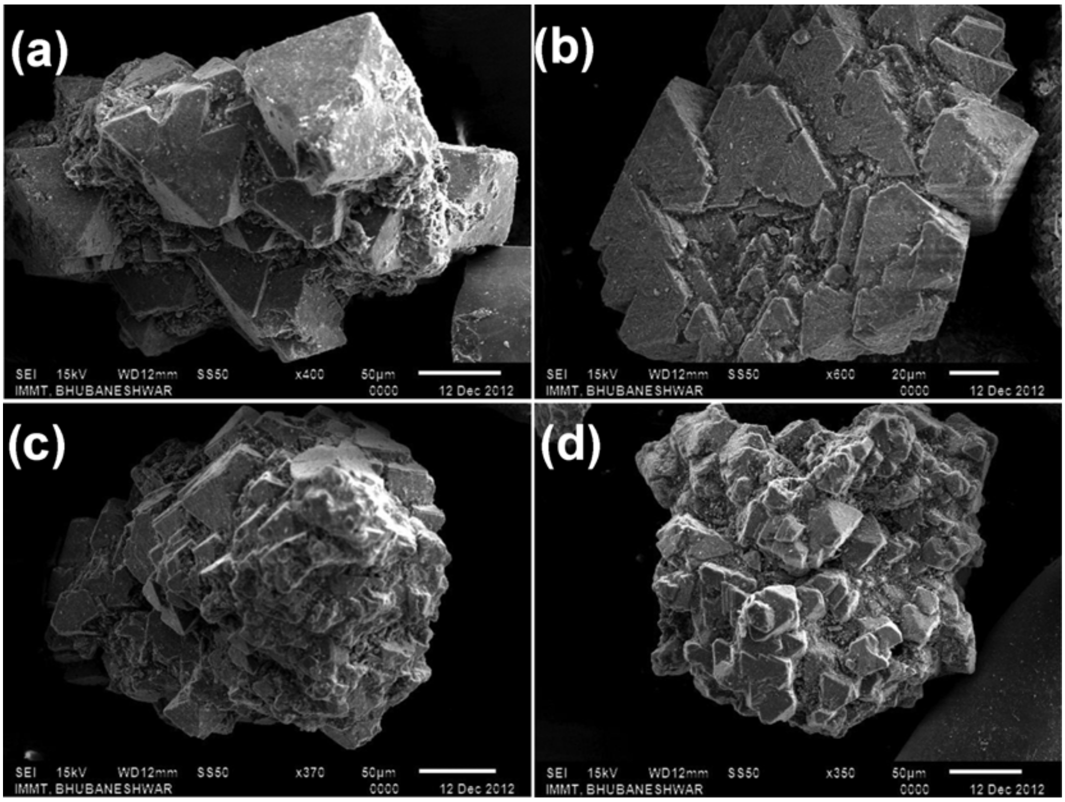

Fig. 5. Scanning electron photomicrographs of the copper slag. (a) Perfect euhedral. (b) Octahedron. (c) Trigon. (d) Cube crystals.

by their color under the optical microscope. They were seen to be present in an inclusive form of one in the other or separately (Fig. 3(b)).

Studies on the morphological features of the slag sample used SEM (JOEL JSM-6510 model). The electron microscopic studies on the slag sample indicated that the sample contains different types of particles (Fig. 4 and Fig. 5). Several shapes including round, spherical, flat or plate shaped with rods, tabular, euhedral were observed that exhibited complete solid structures (Fig. 4). Furthermore, the sample also contained acicular needle type fayalite crystals either embedded in a glassy phase or as acicular needle type fayalite crystals coagulated forming agglomerate of the crystals (Fig. 4(a) and 4(b)). Fig. 4(c) indicates the spherical nature of the particles that verified the formation of slag from the melt. On the other hand, the rod shaped as well as euhedral crystals indicated slag formation from the melt under very slow cooling process. The majority of the slag sample resembled flat, platy shaped grains with few indicating porous structures (Fig. 4(d)). As can be seen in Fig. 5, some grains exhibited perfect euhedral shaped crystals that were conglomerated to form grains of different sizes and shapes like the octahedron, trigon and cube crystals. These crystals were juxtaposed having some voids in between them.

Mineral chemistry was determined by spot analysis of minerals using an energy dispersive spectrometer (EDS) attached to the QEMSCAN. The different phases like spherical metallic phase, magnetite as well as the fayalite phases were analyzed. The backscattered electron (BSE) image generated by SEM is shown in Fig. 6. Four different areas were analyzed by the system and have been numbered

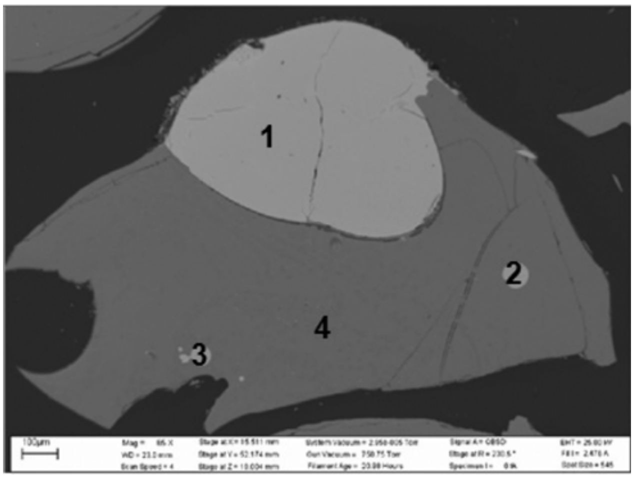

Fig. 6. Back scattered electron image of the SEM. The numbers in the image indicate the spots where the analysis has been carried out and its numerical data is presented in Table 1.

numerically in Fig. 6 that reflected their mineral chemistry. The EDS spectrum with the elemental chemistry of fayalite phase is specified in Fig. 7. From the analysis, it was clear that even the silicates present in slag contained around one percent of copper in them, which could be ascribed to their adsorption in the molten state (Fig. 7).

Further, to bring in better insights into the elemental analysis of the metallic and fayalite phases in the BSE image, the data have been summarized in Table 1. The results of different metallic phases (point

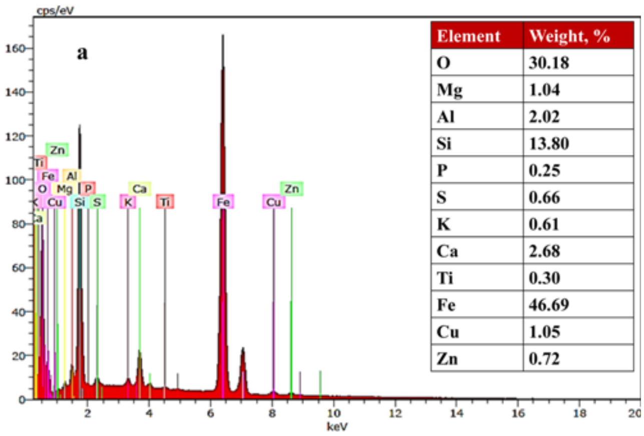

Fig. 7. EDS spectrum of fayalite as indicated in the point analysis of number 4 of Fig. 6.

Table 1. Elemental analysis of the metallic and Fayalite phase in the BSE image

Elements	Weight %			
	1 (Metal)	2 (Metal)	3 (Metal)	4 (Fayalite)
O	3.42	4.40	4.43	30.18
S	20.73	22.40	21.98	0.66
Fe	6.80	7.29	6.88	46.69
Ni	0.46	-------	-------	-------
Cu	66.48	64.38	64.98	1.05
Mg	-------	-------	-------	1.04
Al	-------	-------	-------	2.02
Si	-------	-------	-------	13.8
P	-------	-------	-------	0.25
K	-------	-------	-------	0.61
Ca	-------	-------	-------	2.68
Ti	-------	-------	-------	0.30
Zn	-------	-------	-------	0.72

"--------" Indicates not detected

analysis) indicated that it contains 64.38% to 66.48% Cu, 6.80% to 7.29% Fe and 20.73% to 22.40% S (Table 1). The metallic phase was also seen to contain 3.42% to 4.43% oxygen, which can be attributed to its reaction during slag formation stage. The point analysis of the spherical shaped phases (as observed in Fig. 3) most possibly can be assigned to bornite (Cu_5FeS_4). However, in the present case, the metallic phase constitutes slightly higher copper percentage with less iron and sulfur percentage when compared to natural bornite which has 11.13% Fe, 63.31% Cu and 25.56% S.

2. Physical Beneficiation Tests

The objective of the beneficiation studies was to recover additional copper values from the copper slag sample. Elaborate investigations in mineral characterization (discussed in section 3.1) confirmed the liberation characteristics of the copper values from the associated gangues and indicated that copper values were disseminated within the iron and silicate phases. Now, this phenomenon poses a very little scope to recover copper values by any physical beneficiation means. However, to ascertain beneficiation response,

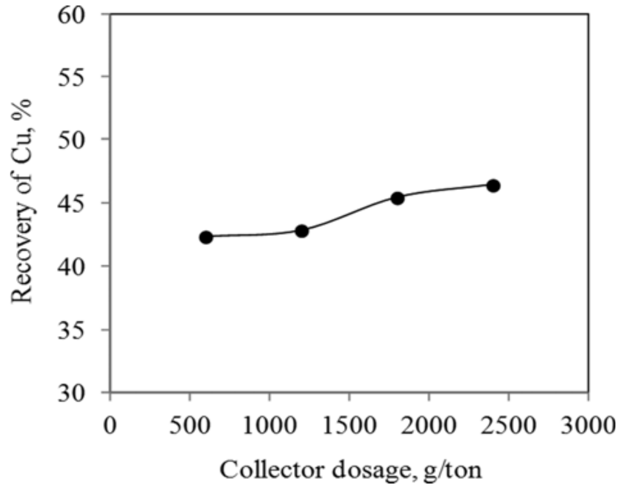

Fig. 8. Percentage recovery of copper with respect to variation in collector dosage (g/ton).

flotation experiments were carried out with various reagent combinations to recover additional copper values from the given sample. The influence of collector dosage (sodium isopropyl xanthate) on the flotation of copper slag is shown in Fig. 8. The figure indicates that increasing the collector dosage from 600 to 2,400 g/t, the Cu recovery increases from 42.3% to 46.4% and the grade improves to 2% to 3% of the feed containing 0.6 % Cu. Note that the influence of dispersant and frother dosage on flotation was not encouraging, and the response of any reagent combinations with regard to the recovery of copper values was very poor (data not shown). The poor response can be attributed to the locking of the copper values within the iron and silica matrices that made the sample less amenable to copper recovery even at ultra-fine particle sizes.

3. Effect of Microbial Leaching and Optimization of Parameters for Copper Recovery

3-1. Effect of pH

Efficient dissolution of metal values using meso-acidophilic microorganisms from various ores and industrial wastes is dependent upon controlled pH conditions [27]. The optimum growth conditions for meso-acidophilic bacteria range within a pH range of 1.5-2.5 to produce the necessary Fe (III) iron to act as an indirect leaching reagent, whereas the bacteria itself plays the direct role [28,29]. The dissolution of fayalite and copper (Cu^o, Cu_5FeS_4) from the slag sample can be explained based on the chemical reactions discussed by Carranza et al. [30] and Zhao et al. [31] as shown below.

Dissolution of Fayalite:

$$Fe_2SiO_4 \rightarrow SiO_4^{4-} + 2Fe^{2+} \quad (1)$$

$$SiO_4^{4-} + 4H^+ \rightarrow H_4SiO_4 \quad (2)$$

$$H_4SiO_4 \rightarrow H_2SiO_3 + H_2O \quad (3)$$

$$H_2SiO_3 \rightarrow SiO_2 + H_2O \quad (4)$$

Dissolution of Cu^o:

$$Cu^o + 2Fe^{3+} \rightarrow Cu^{2+} + 2Fe^{2+} \quad (5)$$

Dissolution of Bornite (Cu_5FeS_4):

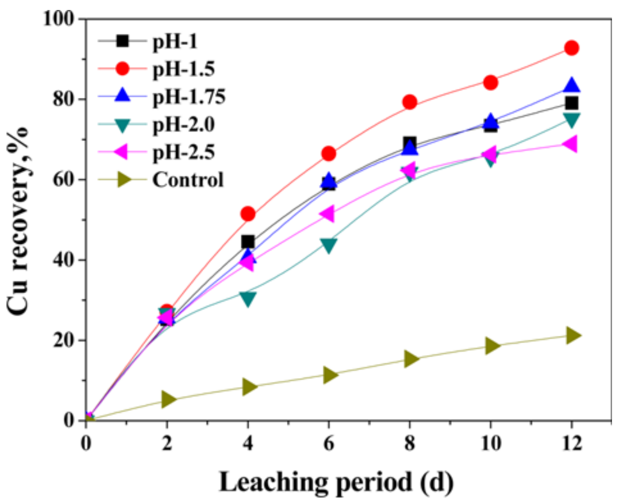

Fig. 9. Effect of variation of pH on copper recovery from slag as a function of time (days).

$$Cu_5FeS_4+O_2+H^+ \rightarrow 5Cu^{2+}+2Fe^{2+}+2H_2O \quad (6)$$

Fig. 9 depicts the copper recovery patterns from the slag sample under varying pH conditions in the presence of the meso-acidophilic bacterial consortium. It was observed that a maximum of 93.3% Cu recovery could be achieved in 12 days of meso-acidophilic leaching at pH-1.5 (Fig. 9). The maximum copper recovery as observed with varying pH values, i.e., 1.0, 1.75, 2.0 and 2.5 was 79.1%, 83.12%, 75.23% and 68.9%, respectively. Interestingly, recovery of copper from the slag sample showed marginal variations at pH values ranging between 1.75-2.5 (Fig. 9). At a lower pH of 1.0 or even less, some interesting observations were marked. The bioleach solution slowly started to have a dense appearance from day 7 onwards till the end of the experiment, which might be ascribed to the dissolution of silica at lower pH values ≤1.0 [7]. Possibly, some copper leached at pH-1.0 from the slag sample might have been adsorbed with few clumps of reddish-like agglomerates, which were noticed upon complete drying of the bioleached residues at the end of the experiment (picture not shown). This might have contributed to a lower recovery of copper along with formation of jarosites. From pH-1.5 onwards, no visible agglomerates or similar precipitates were obtained upon drying post leaching of the slag sample.

3-2. Effect of Varying Fe (II) Iron Concentration

Since the bacterial activity is dependent on Fe (II) concentration in the media, it is necessary to optimize the concentration of its use. As can be seen from Fig. 10, a maximum of 96.83% Cu could be recovered using 3 g/L initial Fe (II) concentration at pH -1.5. A maximum of 90.75%, 84.99% and 73.73% Cu recovery was noticed with 1 g/L; 5 g/L and 7 g/L varying initial Fe (II) concentrations. On the other hand, in the absence of any supplemented Fe (II) iron, i.e., the $9K^{-}$ media, copper recovery was seen to be affected when compared to its presence in the media. A maximum of 60.02% Cu recovery could be achieved in the absence of Fe (II) iron within 12 days of bacterial leaching. In the control set nearly 20% copper recovery was achieved.

3-3. Effect of Inoculum Variation

The concentration of inoculum (v/v) in the bioleaching medium has a significant role to play in metal dissolution process [27]. As can be seen from Fig. 11, an initial 15% (v/v) inoculum concentration in the medium yielded a maximum of 96.43% Cu recovery in 12 days of leaching. The maximum copper recoveries noticed with 5%, 15%, 20% and 25% (v/v) initial inoculum concentrations were 57.98%, 93.56%, 76.12% and 75.4%, respectively. In contrast, the copper recovery recorded from the control set was nearly 20%. Further, with the increase of inoculum concentration in the media, the rate of jarosite formation was proportionately faster (due to a quicker conversion of Fe (II) to Fe (III) at higher inoculum concentrations) with its simultaneous precipitation over the material (due to increase in pH). Vestola et al. [32] also observed a similar kind of phenomenon with a 0.35% Cu containing final slag and 0.02% Cu containing converter sludge sample after 7 and 14 days of bioleaching.

3-4. Effect of Pulp Density (PD)

Pulp density (w/v) is one of the important physicochemical parameters in the microbial leaching process when considered for a large scale operation for the recovery of metals from ores [1,13,27].

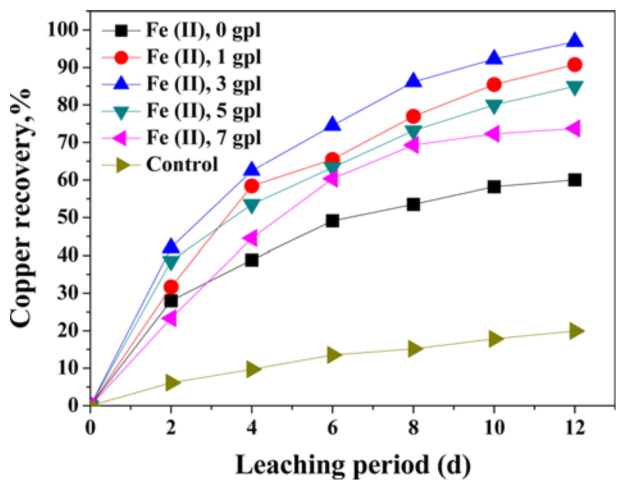

Fig. 10. Effect of variation in Fe (II) concentration on copper recovery from slag as a function of time (days).

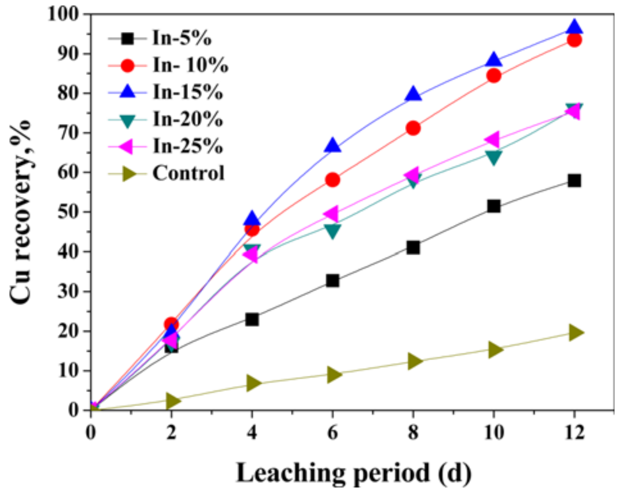

Fig. 11. Effect of variation in bacterial inoculum volume on copper recovery from slag as a function of time (days).

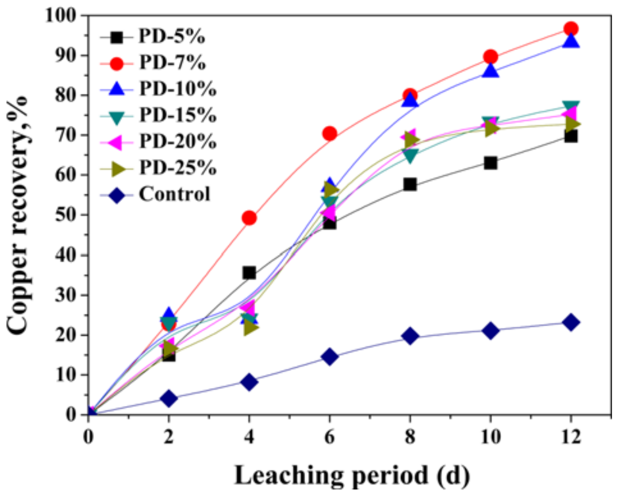

Fig. 12. Effect of variation in pulp density (w/v) on copper recovery from slag as a function of time (days).

As can be seen from Fig. 12, a maximum of 96.66% Cu recovery was achieved in 12 days of leaching at 7% pulp density (w/v) of slag in the bioleaching medium. The maximum copper recovery as observed with different pulp densities: PD-5%, PD-10%, PD-15%, PD-20% and PD-25% (w/v) slag were 69.8%, 93.25%, 77.3%, 75.2%, and 72.78%, respectively. It is very interesting to note that marginal variations in recovery of copper were obtained at PD's ranging from 15-25% (w/v). It was, therefore, clear that a higher pulp density above 7% does not bear a significant effect on copper extraction using the mixed bacterial consortium. It can be anticipated that recovery rates of copper may vary in bench scale or scale up studies. These findings might be attributed to the toxic effects of higher dissolution of metal concentrations upon the microbial populations in the bioleaching medium or formation of any such agglomerates or jarosite precipitation or a combined effect of all the above. Under control conditions, the maximum copper recovery observed was less than 25%.

4. Analytical Insights of Slag Post Bioleaching

To shed more light on the mineralogical aspects of the slag sample post meso-acidophilic bioleaching, detailed studies were undertaken by scanning electron microscope (SEM), field emission scanning electron microscope (FE-SEM) attached with energy dispersive X-ray spectrometer (EDAX).

SEM characterization of the microbiologically leached copper slag indicated that the samples lost their structure while some of the cubic grains still persevered. The cubic grains were ascribed to magnetite, which is hard to leach out because of its spinel and cubic structure. Further, the samples exhibited massive coagulated texture with porous structures after leaching (Fig. 13(a) and 13(b)). The weathered structure of the slag showed typical petals of a flower shape (Fig. 13(c) and 13(d)). This phenomenon clearly demonstrated the efficacy of the mixed meso-acidophilic bacterial consortium on copper recovery from the slag sample. After convincing results from SEM, field emission scanning electron microscopic studies (FE-SEM Zeiss Supra-55 model) coupled with EDAX analysis at

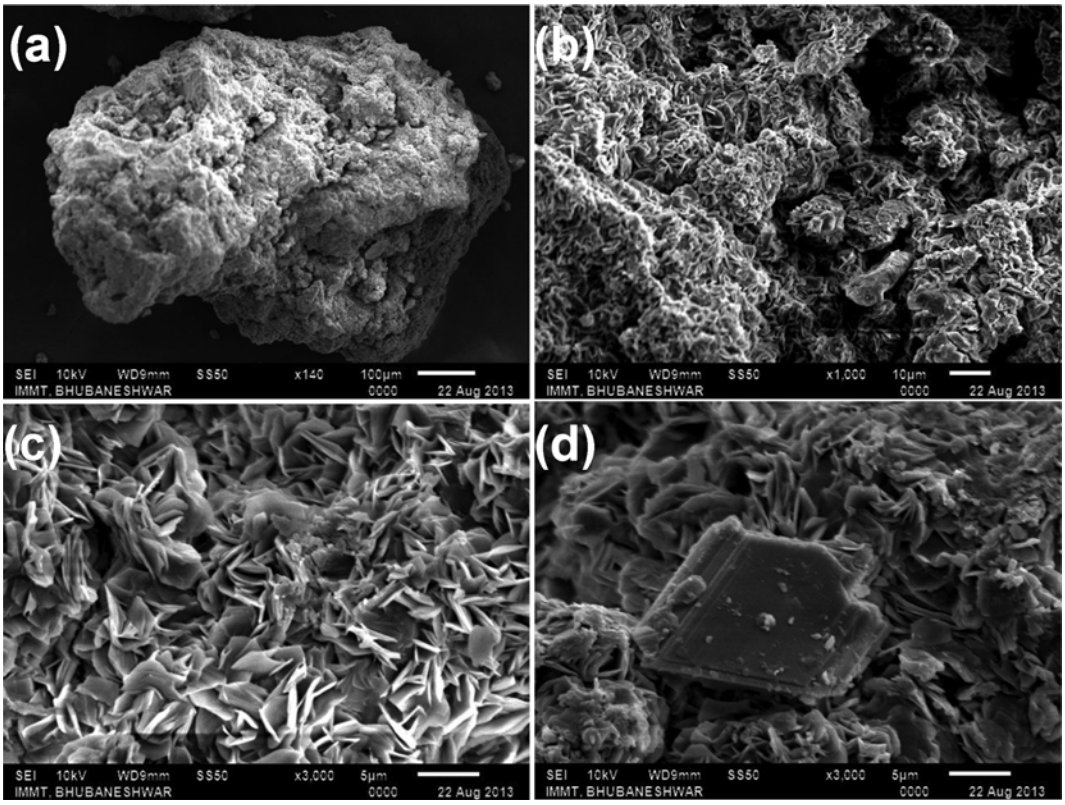

Fig. 13. (a) Massive and patchy nature of the bioleached slag residue. (b) Fibrous, needle and petal shape granules after bioleaching. (c) Petals of a flower type of the granules as seen in the bioleached residue. (d) Remnants of cubic crystals observed in the bioleached residue.

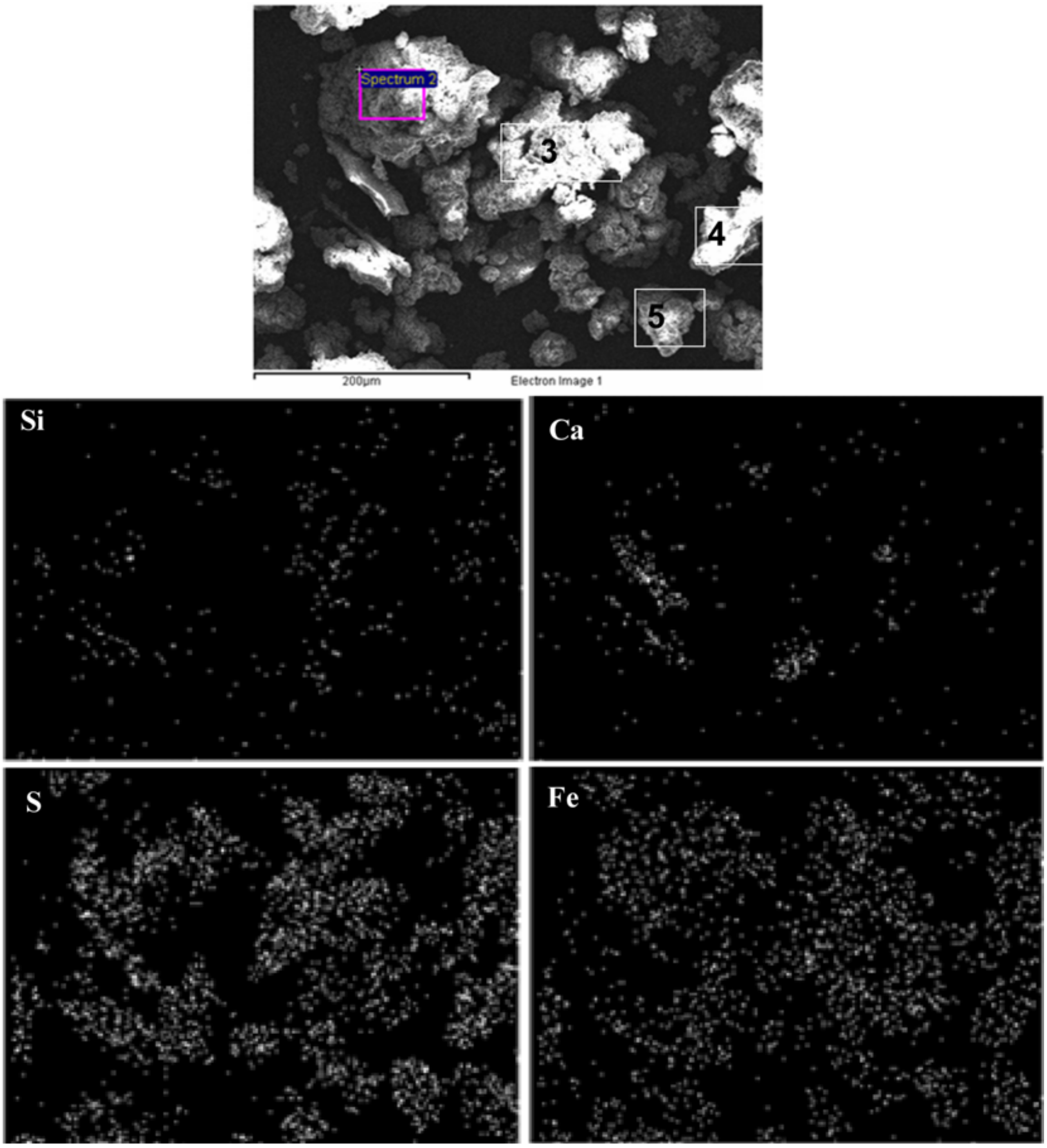

Fig. 14. Elemental mapping of the bioleached residue. Top photomicrograph is the BSE image. Silica (Si), Calcium (Ca), Sulphur (S) and iron (Fe) decipher the elemental mapping that has been carried out.

nanometer scale provided us with some more interesting insights into the copper dissolution from the slag sample. Both elemental mapping as well as area analysis of the grains of the bio-leached residue was carried out using FESEM (Fig. 14). As can be seen from the elemental mapping patterns in Fig. 14, the bioleached residues were devoid of copper. Further EDAX analysis of some selected spectra (Spectrum 2 and 3) as shown in Fig. 15 also confirmed the FE-SEM elemental mapping studies. In other words, a detailed instrumental analysis of the bioleached residue through SEM, FE-SEM elemental mapping and EDAX studies was in confirmation of the results obtained during the microbial leaching and optimization studies discussed in section 3.3.

CONCLUSIONS

Optical and electron microscopic studies revealed that mineralogically the slag sample contains fayalite and magnetite as the major phases, minor amounts of sulfides and traces of metal phases containing copper. Copper was identified to be dispersed within the iron and silicate phases. Physical beneficiation of the slag sample resulted in poor copper recovery when compared with a mixed meso-acidophilic bacterial leaching test. The flotation studies showed that it is possible to achieve a copper grade of 2-3% with 42-46% recovery values. On the other hand, the meso-acidophlic bacterial leaching studies resulted in 96% copper recovery in 12 days of leaching with optimized conditions at pH of 1.5, 15% (v/v) bacterial inoculum in media, 3 g/L initial Fe (II) concentration and 7% (w/v) pulp density. Further, mineralogical analysis by means of FE-SEM elemental mapping and EDAX studies provided better insights into the copper recovery patterns from the bioleached slag residue, which clearly confirmed the efficacy of the microbial leaching over physical beneficiation.

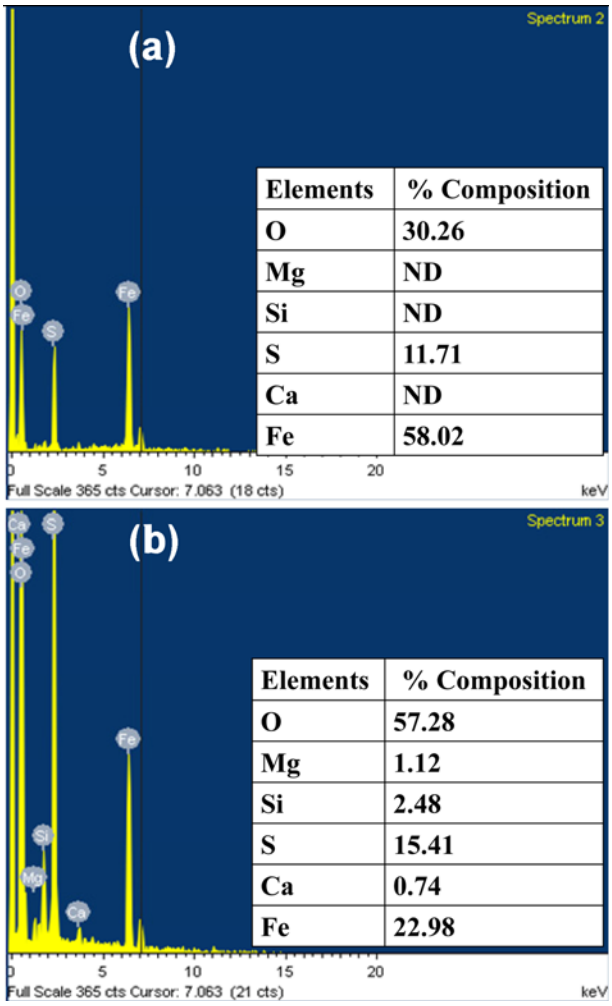

Fig. 15. Energy dispersive X-ray spectroscopy analysis of bioleached copper slag residues.

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