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Title: Processing an Ultramafic Ore Using Fibre Disintegration by Acid Attack

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Keywords: Ultramafic deposits, chemical/mechanical treatment, fibre disintegration, flotation, collectorless flotation

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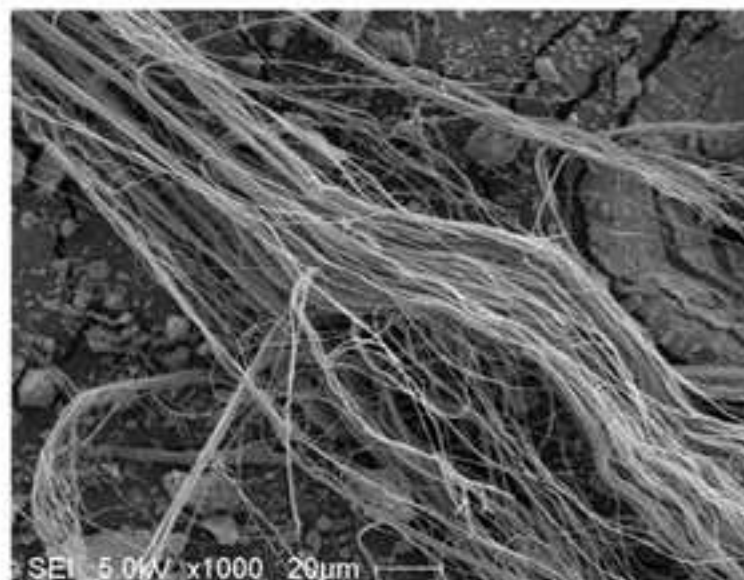
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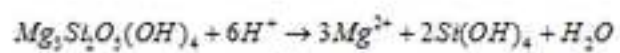
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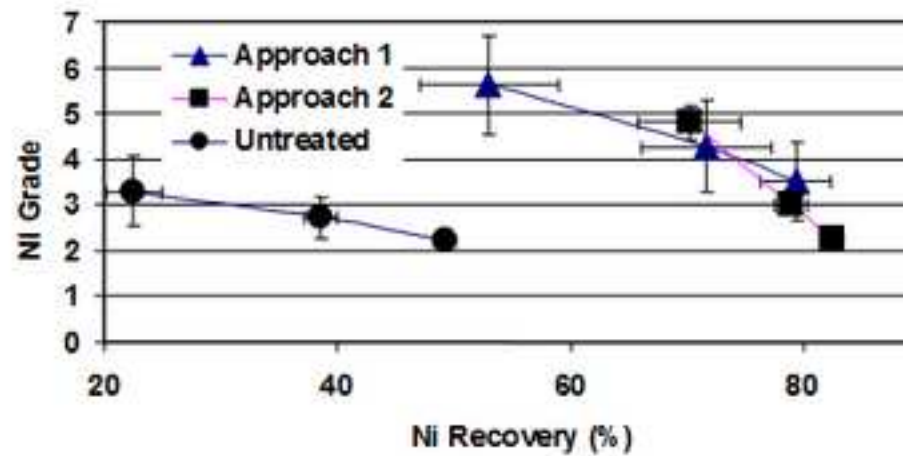
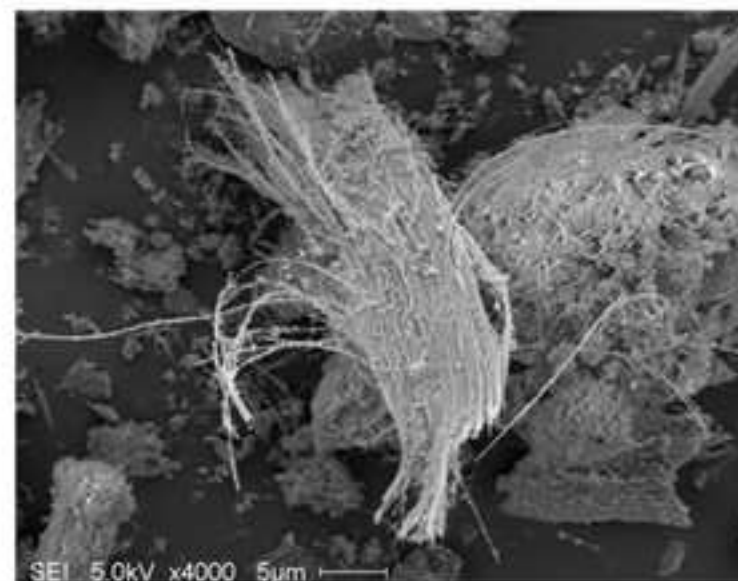
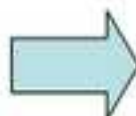
Salah Uddin



Chrysotile fibre



Grinding media



Research highlights:

- A processing route has been designed for serpentine rich ultramafic Ni-ore.
- Process based on fibre disintegration by combined chemical/mechanical treatment.
- Low pH slurry after the treatment effects collectorless flotation.
- Significant improvement in Ni-metallurgy and MgO rejection was achieved.
- A mechanism of fibre disintegration is proposed.

Processing an Ultramafic Ore Using Fibre Disintegration by Acid

Attack

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Abstract

Ultramafic ores are a potential major resource of nickel. For the Thompson area deposits of northern Manitoba, Canada, a processing challenge is the serpentine polymorph chrysotile, the dominant gangue mineral. Due to its fibrous nature chrysotile causes physical entanglement and increased pulp viscosity which reduces selectivity in flotation. The proposed approach involves disintegration of the fibres by a combination of chemical (H_2SO_4) and mechanical (grinding) treatment adapting technology explored for CO_2 sequestration. The use of H_2SO_4 also led to collectorless flotation traced to elemental sulphur formation, and release of magnesium increased solution ionic strength sufficient to eliminate the need for frother. Significant improvement in Ni grade-recovery with higher rejection of MgO-bearing gangue was achieved compared to untreated ore. Structural changes in the fibres were followed using spectroscopic techniques. A mechanism of fibre disintegration is suggested.

Keywords: Ultramafic deposits, chemical/mechanical treatment, fibre disintegration, flotation, collectorless flotation

1. Introduction

There are two major sources of Ni: sulphide ores and laterites. Sulphide ores range from massive (90% sulphide, up to 10% Ni) to sulphide matrix (20-50% sulphide) to disseminated (0.5% Ni). Massive and sulphide matrix ores are characterized by high pyrrhotite content that often represents the principal separation challenge in flotation of the main Ni mineral, pentlandite. Flotation typically employs combinations of xanthate collector, MIBC frother, soda ash pH modifier (Pietrobon et al., 1997; Fuerstenau and Somasundaran, 2003), carboxy-methyl-cellulose (CMC) dispersant (Edwards et al., 1980; Wellham et al., 1992) and occasionally diethylenetriamine (DETA) as pyrrhotite depressant (Xu et al., 1997). Canadian examples are the ores of the Sudbury district, Raglan in northern Quebec and Voisey's Bay, Labrador. These sulphide ores represent the bulk of current supply of nickel and often yield significant copper and by-products such as platinum-group elements.

Laterites result from extensive weathering of sulphide ore and the Ni is present in a variety of Ni silicate minerals in a largely iron oxy-hydroxide matrix (Dalvi et al., 2004). The main current processing option is pressure acid leaching.

As a result of decreasing massive/sulphide matrix ore reserves and the technical challenge and cost of treating laterites, increasing attention is directed to disseminated ultramafic deposits. The name reflects the high content of magnesium-iron silicate gangue minerals (referred to as MgO minerals) derived from weathering of primary minerals such as olivine (Hurlbut and Sharp, 1998).

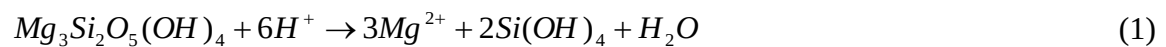
Several hundred million tonnes of inferred Ni resource is hosted in ultramafic rocks in the ‘Thompson Nickel Belt’ in north central Manitoba (Dai et al., 2009). This is the deposit of interest in this study. Similar deposits are in northern Quebec, Western Australia, north central China, Zimbabwe and Finland (Mani et al., 1997).

The processing problem in common with these potential ores is the fibrous nature of some of the MgO minerals, for example chrysotile. The fibres produce entanglement and viscous slurries which hamper grinding and flotation. High rejection of MgO minerals is required to avoid increasing slag melting temperature in Ni smelting. Physical removal of fibres, e.g. by cycloning, has resulted in improved rejection of MgO (Senior and Thomas, 2005; Dai. et al., 2009). The purpose of this paper is to propose an alternative route to fibre removal based on disintegrating the fibres by chemical-mechanical means.

2. Background

The dominant mineral in Thompson ultramafic deposits is the serpentine polymorph chrysotile (Mani et al., 1997). It comprises sheets of brucite (magnesium hydroxide) covalently bonded to sheets of tridymite (polymorph of quartz). The mismatch in spacing between Mg and Si atoms makes chrysotile curl into hollow tube-like fibres (Brindley and Brown, 1980; Wypych et al., 2005).

Recently, serpentine minerals have been investigated for CO₂ sequestration (Seifritz, 1990; Park and Fan, 2004; Fujii et al., 2010). By acid attack, Mg can be leached from the brucite layer (reaction 1) to react with CO₂ to form environmentally benign and stable MgCO₃:



The structure remaining after leaching consists of hydrated amorphous silica without elasticity and dimensional stability that can be easily broken mechanically (Hargrevass and Taylor, 1946; Stumm, 1992). Park and Fan (2004) showed that extraction of Mg is enhanced by combining acid attack with grinding which facilitates the exposure of the inner Mg layers to the acid.

This CO₂ sequestration work suggested a possible treatment to effect fibre disintegration prior to flotation. A previous paper considered HCl (Uddin et al., 2010), as used by Park and Fan (2004). In this paper we examine H₂SO₄ which is arguably the practical option, the acid being widely available as part of SO₂ abatement at smelters.

In the case of HCl, the slurry pH rose with time eventually (ca. 12 hrs) allowing the addition of soda ash and the setting of other conditions typical of Ni sulphide ore flotation. This was not the case with H₂SO₄, the slurry remaining buffered at ca. pH 2. Xanthate collectors tend to decompose at low pH (Iwasaki and Cooke, 1958), but the use of sulphuric acid opens the possibility of collectorless flotation: Heiskanen et al. (1991) found that pentlandite (along with pyrrhotite) floated collectorlessly at pH 3-5. Sulphuric acid provides the oxidizing conditions to promote formation of polysulphide (metal deficient sulphide) and elemental sulphur which are the hydrophobic species responsible for flotation (Heyes and Trahar, 1977; Gardner and Woods, 1979; Rao and Leja, 2003). The oxidization (anodic) reactions are represented in (2) and (3) with oxygen acting as electron acceptor to provide the reduction (cathodic) reaction (4) and complete the redox couple. In addition, the low pH prevents released metal ions from precipitating as hydroxides which can depress flotation.

Anodic reactions:



Cathodic reaction:



Another potentially useful outcome is that released Mg^{2+} ions may increase the ionic strength of the pulp liquor sufficient to inhibit bubble coalescence (Craig et al., 1993; Hofmeier et al., 1995; Laskowski et al., 2003) and reduce bubble size, normally the function of adding frother. It has been shown that an ionic strength > 0.4 gives similar bubble size and flotation response as ca. 10 ppm MIBC (methyl-iso-butyl-carbinol), a typical sulphide flotation frother and dosage (Quinn et al., 2007). The proposed treatment using sulphuric acid, therefore, may not only disintegrate fibres but also produce a flotation condition requiring no collector or frother.

3. Experimental

3.1. Ore mineralogy

The sample was supplied by Vale and comprised mostly serpentine (63%) and olivine (12%) with minor dolomite and Mg chlorite. X-ray diffraction identified the major serpentine mineral as clinochrysotile (Fig. 1). Naturally hydrophobic talc content was low ($<1\%$). The main iron minerals were pyrrhotite (5%) and magnetite (6%). The principal Ni sulphide mineral was pentlandite with minor violarite, mackinawite and millerite giving a grade of ca. 0.6% Ni.

3.2. Flotation without acid treatment

Grinding used a 15 x 15 cm ceramic ball mill with 40%v/v charge of 3, 1.8 and 1 cm diameter zirconium oxide balls. A 100 g sample was slurried with 800 mL water; and 1 wt% soda ash and 0.05 g CMC were added prior to grinding for 1 hr. A sample of supernatant was taken for assaying (ICP-OES) after grinding to determine dissolved species. The slurry was then transferred to a Denver flotation cell.

For flotation, the slurry volume was adjusted to 1 L (i.e., slurry density is 10 wt% solids). Soda ash was added to bring pH to 10. As collector, 0.004 g of purified potassium-amyxanthate (PAX) was used with a conditioning time of 5 minutes (based on scaling down the aeration time used by Heiskanen et al. (1991)). Impeller speed was 1500 rpm. Frother MIBC (0.002 g) was added and conditioned for a further 5 minutes prior to introducing air (flow rate 5 L/min). Three concentrates were taken at 1, 2 and 4 minutes. They along with the tails, were filtered, oven dried, weighed and assayed (ICP-OES). This procedure is based on Ni sulphide flotation practice.

3.3. Flotation with acid treatment

The same grinding mill was used but this time the 100 g sample was slurried with 800 mL of 15 wt% sulphuric acid (H_2SO_4). The acid concentration was selected based on the previous work (Uddin et al., 2010). There were two approaches taken to encourage oxidation and collectorless flotation: 2-hr aging in the ball mill followed by addition of DF250 (polypropylene methyl ether) frother (0.002 g) (MIBC did not produce adequate froth in this case); or, 5-min aeration (5 L/min) in the Denver cell with flotation of the first two concentrates without frother and with 0.002 g DF250 addition prior to collecting the third concentrate. The common conditions were: grinding time 1 hr, slurry volume

adjusted to 1 L for flotation, and three concentrates (5, 10 and 15 minutes) collected. For both approaches, the experiments were conducted three times. The flowsheet is shown in Fig. 2.

3.4. Illustration of fibre disintegration

In these tests 1 g of ore ground (1 hr) without acid was transferred to an 8 x 8 cm ceramic mill with 1-cm zirconium oxide grinding media (40%v/v charge). The sample was ground for a further hour in the presence of 5 mL HCl or H₂SO₄, equivalent to ca. 500 wt% acid. A higher concentration of acid than in the flotation tests was needed to achieve detectable physical changes in the fibres. After grinding, the samples were filtered, dried and analyzed.

3.5. Analytical methods

X-ray diffraction (American Instruments Inc., Cu target at 20 mA, 40 kV) was used to determine mineral phases. Fourier transformation infrared spectroscopy (FT-IR; Model 1600, Perkin Elmer) was used to identify changes in molecular structure. Disks were prepared by mixing 200 mg KBr and 3-4 mg of sample. Electron beam analysis (Philips XL30 FEG-SEM with Genesis EDS/X-ray microanalysis system) was used to image physical changes. Powdered samples were mounted on carbon tape and wafer-coated with gold/palladium (Anatech Hummer VI sputtering system).

A test was performed to extract hydrophobic species from concentrate produced in approach 2 using toluene at 70-80°C (Becze et al., 2009). Extracted material was precipitated by evaporation and analyzed with SEM-EDS and Raman spectroscopy. For the latter, spectra of the extract and a standard elemental sulphur sample (Sigma-Aldrich)

were collected using an inVia Raman microscope (Renishaw). Laser excitation was provided by a polarized He-Ne laser operating at 632 nm.

3.6. Bubble size distribution

Acid treatment produced a pulp liquor of ionic strength ca. 0.85 (considering SO_4 , Mg, Fe and Ni ions in solution). To simulate and determine the effect on bubble size, a solution containing 4 g/L Mg (as MgSO_4 , ionic strength ca. 0.7) was prepared and placed in a 110 x 10 cm bubble column with a rigid, vertical cylindrical porous sparger. Bubble size distribution (BSD) was determined using the McGill Bubble Size Analyzer (MBSA) (Hernandez-Aguilar and Finch, 2005) at a gas superficial velocity of 0.5 cm/s. Images were collected and processed using a 10-bit digital camera driven by commercial software (Northern Eclipse, Empix Imaging Inc.). Typically, 500 bubble images were processed for each test. The BSD data are presented as number mean (d_{10}) and Sauter mean diameter (d_{32}).

4. Results

4.1. Effect of acid treatment

An indication of fibre disintegration is the extraction of Mg. Table 1 shows the 15 wt% H_2SO_4 extracts ca. 25% of the Mg yielding a solution concentration ca. 6-7 g/L Mg. At the same time, ca. 4% Ni and 12% Fe were lost to solution. It was noted that slurry flowability increased after acid treatment.

4.2 Flotation

The slurry pH was stable after grinding with H_2SO_4 (ca. pH 2). Fig. 3 compares the froth appearance of the treated and untreated sample after the first minute of flotation (approach 1). For treated sample the froth had the metallic sheen typical of a sulphide float compared to the dull appearance for the untreated sample.

Fig. 4 shows the Ni grade vs. recovery for both approaches. The results are similar: about 80% Ni recovery at 2.5-3.5% Ni grade was achieved with the treated sample (enrichment ratio ca. 4.5); a significant improvement compared to the untreated case which achieved only 50% Ni recovery by concentrate 3. This corresponds to increased selectivity against the MgO minerals: Fig. 5 indicates that at 90% MgO rejection (i.e., recovery to tails) the treated sample yielded ca. 80% Ni recovery while the untreated sample gave only ca. 45% Ni recovery. This limited separation for untreated ore is seen by others (Dai et al., 2009).

The main gangue contaminant in the concentrate was pyrrhotite (Po) as indicated in the XRD pattern (Fig. 6). From the Fe assay and assuming no other Fe-bearing mineral in the concentrate besides pentlandite, the pyrrhotite content at 80% Ni recovery was ca. 30%. The collectorless flotation at ca. pH 2 was evidently selective against MgO but not Po, as the work of Heiskanen et al. (1991) would support.

4.3 Illustrating fibre disintegration

This test was done at the higher acid concentration. Fig. 7 shows FT-IR spectra over two frequency ranges, 500 to 2000 cm^{-1} (left-hand figure) and 3000 to 4000 cm^{-1} (right-hand figure). The major absorption bands are identified based on the literature (Nyquist and Kagel, 1971; Sugama et al., 1998). The band at 3680 cm^{-1} with the shoulder

at 3640 cm^{-1} represents the stretching vibration of hydroxyl in $\text{Mg}(\text{OH})_2$. The 3410 and 1640 cm^{-1} bands relate to stretching and bending modes of H_2O and the bands at 1080 and 970 cm^{-1} corresponds to Si-O-Si and Si-O⁻ stretching, respectively. The important features of the samples ground with acid include reduction of band intensity at 3680 and 3640 cm^{-1} , growth of bands at 3410 and 1640 cm^{-1} , gradual conversion of bands at 1080 and 970 cm^{-1} into shoulders and emergence of bands at 1220 and 810 cm^{-1} .

X-ray diffraction patterns are shown in Fig. 8. There is apparent attenuation of the chrysotile-related peaks (marked by arrows) in the samples ground with acid. The SEM image (Fig. 9A) reveals the long fibres in the ground untreated sample while acid-treated samples – even at the higher magnification – show few of these long interconnected fibres being instead dominated by short, apparently broken fibres (Figs. 9B and 9C). Higher magnification is used to visualize one of the short fibre bundles (Fig 9D).

4.4. Bubble size

Fig. 10 shows bubble size in tap water and in 4 g/L Mg solution with H_2SO_4 added to pH 2.8. Bubble coalescence was inhibited and considerable decrease in bubble size (Sauter mean reducing from ca. 4 mm to ca. 0.6 mm) was observed in the salt solution compared to tap water.

4.5. Evidence of elemental sulphur

SEM-EDS analysis of the substance extracted from the concentrate (approach 2, concentrate 1) is shown in Fig. 11. The crystalline-looking material is evidently sulphur. Raman spectrum confirms sulphur by comparison with a standard sample (Fig. 12). X-ray photoelectron spectroscopy (XPS) studies (not shown) were performed to identify

polysulphide species. But, probably due to low concentration, decoupling polysulphide peaks from elemental sulphur in the spectra was not possible.

5. Discussion

The positive impact on mineral separation of the acid/grind treatment is evident in the improved Ni grade-recovery and rejection of MgO minerals compared to the untreated case. The treatment was adapted from work to enhance CO₂ sequestration which employ acid attack on serpentine minerals to release Mg (to react with CO₂).

The ca. 25% release of Mg here infers fibre disruption and the sensed increase in slurry flowability (reduced viscosity) implies the targeted improvement in rheological character was achieved. The cost was 4% Ni loss, although it is possible some of the dissolved Ni came from minerals other than Ni sulphides which are otherwise unrecoverable by flotation.

Chrysotile is one of the strongest asbestos-type minerals (Kogel et al., 2006). It is not easily broken by grinding but with sufficient acid breakage is demonstrated (Fig. 9). In the FT-IR spectra (Fig. 7) the reduction of the Mg-OH band at 3680 cm⁻¹ and transition to a shoulder of the Si-O band at 970 cm⁻¹ on acid treatment can be interpreted as breakage of the external Mg(OH)₂ layers on the serpentine; and the growth of the bands at 3410, 1640 and 1220 cm⁻¹ can be interpreted as hydration of the exposed inner siliceous structure. Apparent attenuation of the chrysotile-related line intensities in the XRD pattern after acid treatment (Fig. 8) is also evident in the literature (Sugama et al., 1998).

Fig. 13 suggests the possible breakage mechanism resulting from acid attack: cracks are initiated by extraction of Mg from the surface followed by rapid crack growth

in this ‘corroding’ environment. That fibre breakage was not evident at the 15 wt% acid flotation condition is attributed to limited breakage which is difficult to locate in a small amount of sample. The higher acid strength tests clearly show breakage and the mechanism in Fig. 13 seems plausible.

Leaching of Mg will also alter surface charge on serpentine. Poor separation of pentlandite from serpentine has been attributed to hetero-coagulation of the minerals which carry opposite charge, serpentine positive and pentlandite negative, at alkaline flotation pH (Edwards et al., 1980). Loss of Mg will tend to reduce the charge on serpentine (Tartaj et al., 2000) making electrostatic interaction with pentlandite less likely. At the high ionic strength of the leach solution measuring particle surface charge is difficult so this possibility has not been verified.

The acid treatment induced flotation without collector or frother. The collectorless response was traced to formation of elemental sulphur that is known to be promoted at low pH (Heiskanen et al., 1991). Just 5-minute aeration created the collectorless response. The ‘frotherless’ flotation was related to the ionic strength of the leach liquor exceeding ca. 0.4 where it is known that bubble size reduction at least equivalent to conventional frothers is realized with Mg salts. Bubble size reduction is a necessary condition to increase flotation kinetics and hence capacity of the flotation machine. High ionic strength may have other effects related to particle and even bubble charging but these are considered secondary to the impact on bubble size. The fact that collector and frother are not required offers a cost offset to the proposed treatment. The product of flotation while encouragingly low in MgO minerals is high in pyrrhotite.

Pentlandite/pyrrhotite separation is a common problem processing Ni-sulphide ores and various technologies exist that will require reagent additions.

A conceptual circuit based on the findings and assuming a supply of H_2SO_4 is presented in Fig. 14. Apart from the potential to resolve problems associated with fibrous minerals the proposed route turns two liabilities, SO_2 (captured as H_2SO_4) and MgO-rich tails (possible candidate for CO_2 capture), into potential assets.

6. Conclusions

Sulphuric acid/grinding treatment gave marked improvement in pentlandite-MgO mineral separation in flotation of an ultramafic ore. The improvement is attributed to disintegration of sufficient serpentine (clinochrysotile) fibres to reduce entanglement and pulp viscosity that otherwise hinder selective flotation. Fibre disintegration was indicated by significant (ca. 25%) Mg loss to solution and supported by electron beam and spectroscopic analyses. About 4% Ni is lost to solution. The treatment induced flotation without collector, shown to be due to elemental sulphur formation; and without frother, shown to be due to high ionic strength of the leach solution.

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Table 1
Metallurgical balance (average of the three tests): leach and flotation, approach 1 (A) and approach 2 (B). (Note: composition of ‘mass loss’ is inferred from grams mass loss and solution assay.)
A) Approach 1: 2-hr aging and frother (DF250) addition.

	Mass	Assay (%)			Distribution (%)		
	(g)	Ni	Mg	Fe	Ni	Mg	Fe
Leaching							
Sol (L, g/L)	1	0.03	6.89	1.60	-	-	-
Mass Loss	14.13				4.2	26.6	12.2
Flotation							
Con 1	6.80	5.24	9.75	28.34	52.9	3.4	13.2
Con 2	5.93	2.38	14.28	23.83	18.6	4.6	9.3
Con 3	4.69	1.25	13.56	18.55	7.6	5.1	6.4
Tail	68.43	0.17	21.68	10.21	16.5	60.2	58.7
Head	100	0.67	23.46	12.30	100	100	100

B) Approach 2: 5-min aeration, no frother for cons 1 and 2.

	Mass	Assay (%)			Distribution (%)		
	(%)	Ni	Mg	Fe	Ni	Mg	Fe
Leaching							
Sol (L, g/L)	1	0.02	6.35	1.36	-	-	-
Mass Loss	10.87	-	-	-	3.8	25.3	11.1
Flotation							
Con 1	9.27	4.79	8.56	31.64	70.1	3.1	26.2
Con 2	7.30	0.76	14.74	13.20	8.6	3.6	8.6
Con 3	6.52	0.35	18.16	8.31	3.6	2.6	4.8
Tail	66.01	0.13	21.41	8.26	13.6	65.1	49.1
Head	100	0.63	23.80	11.12	100	100	100

Fig1

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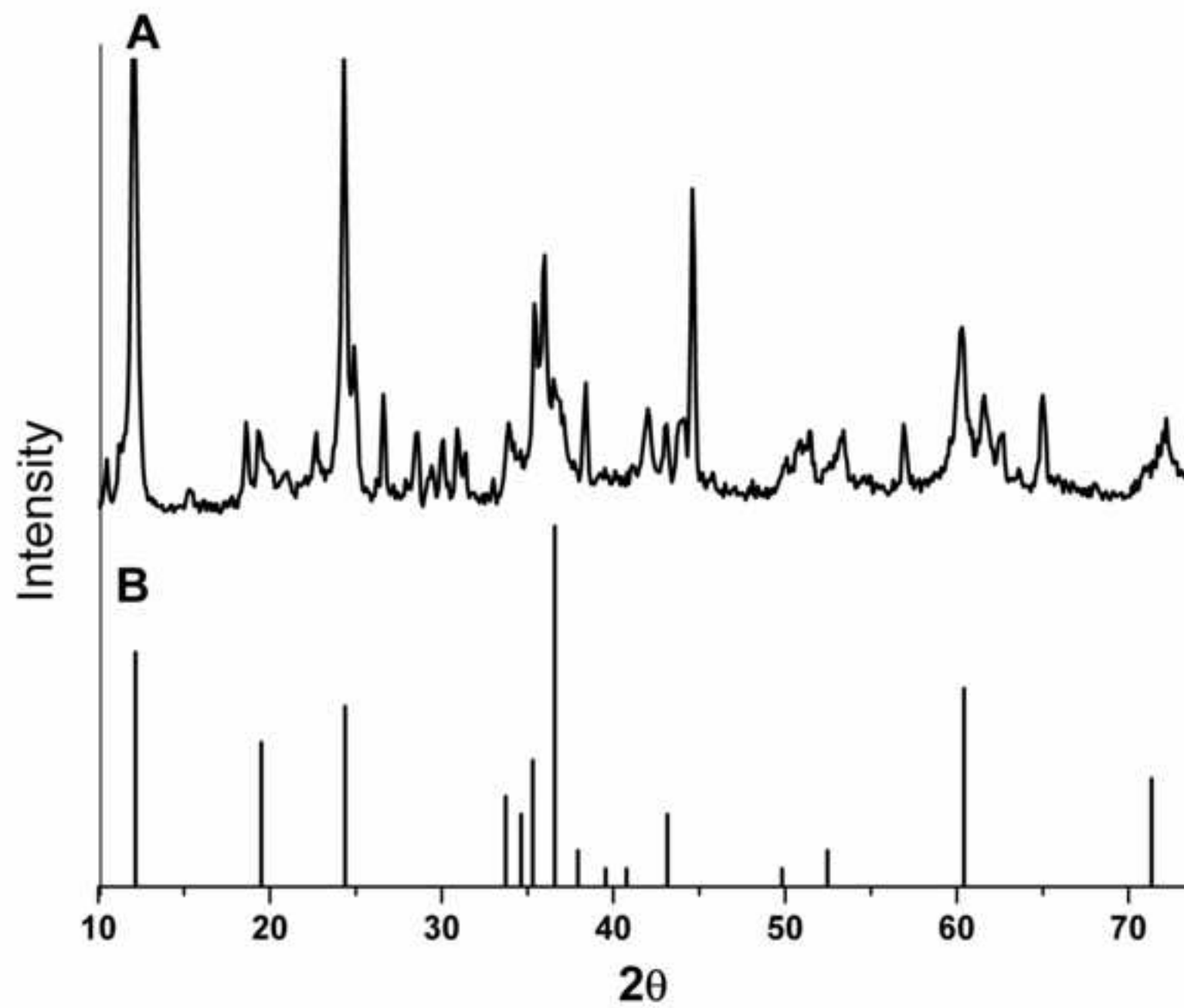


Fig2

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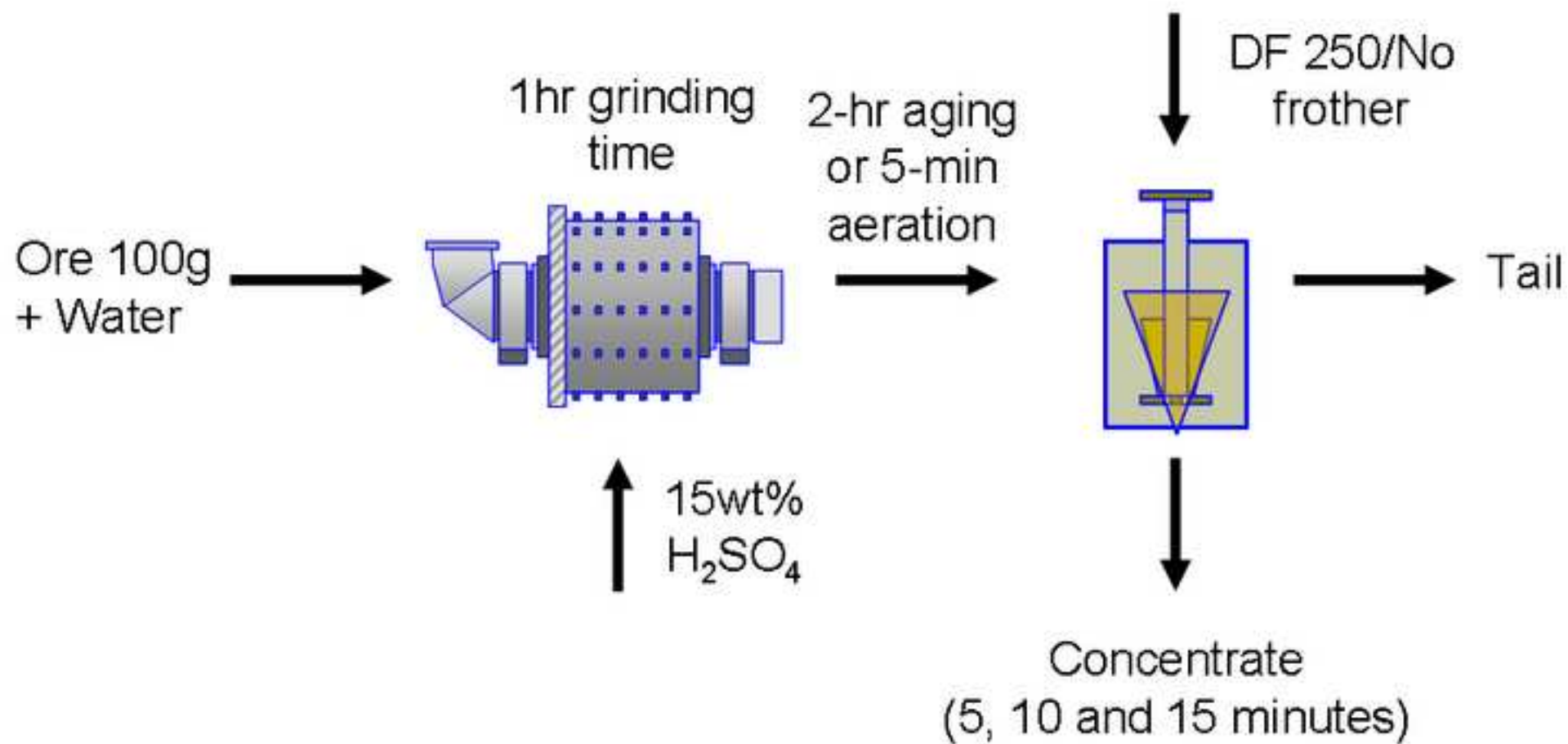


Fig3

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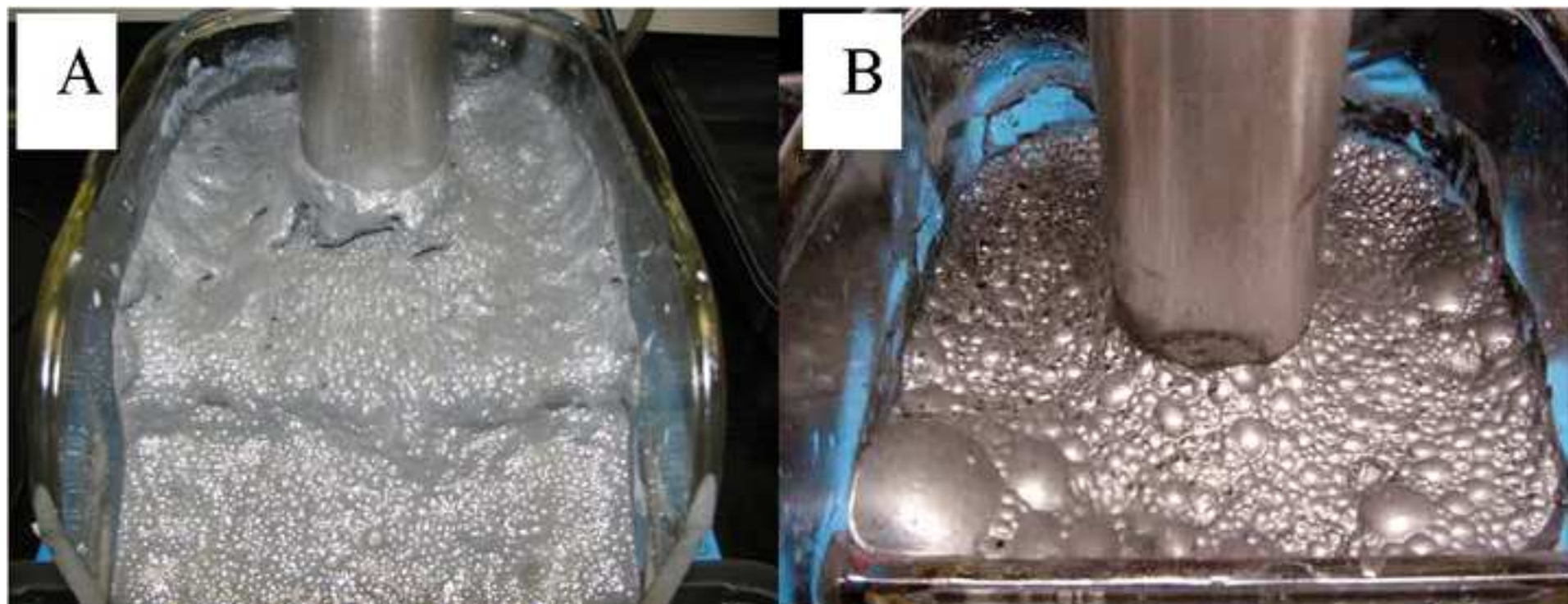


Fig4

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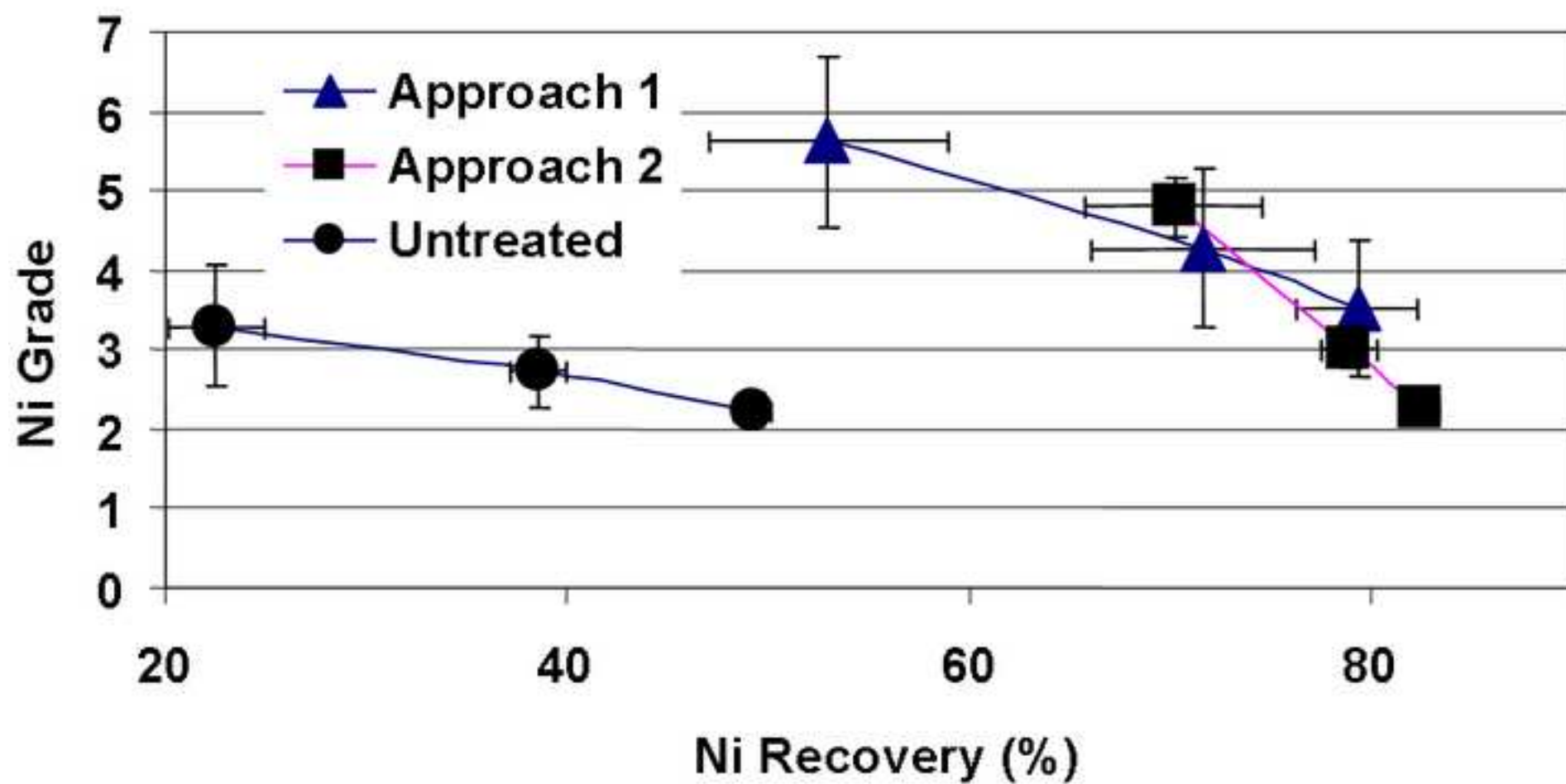


Fig5

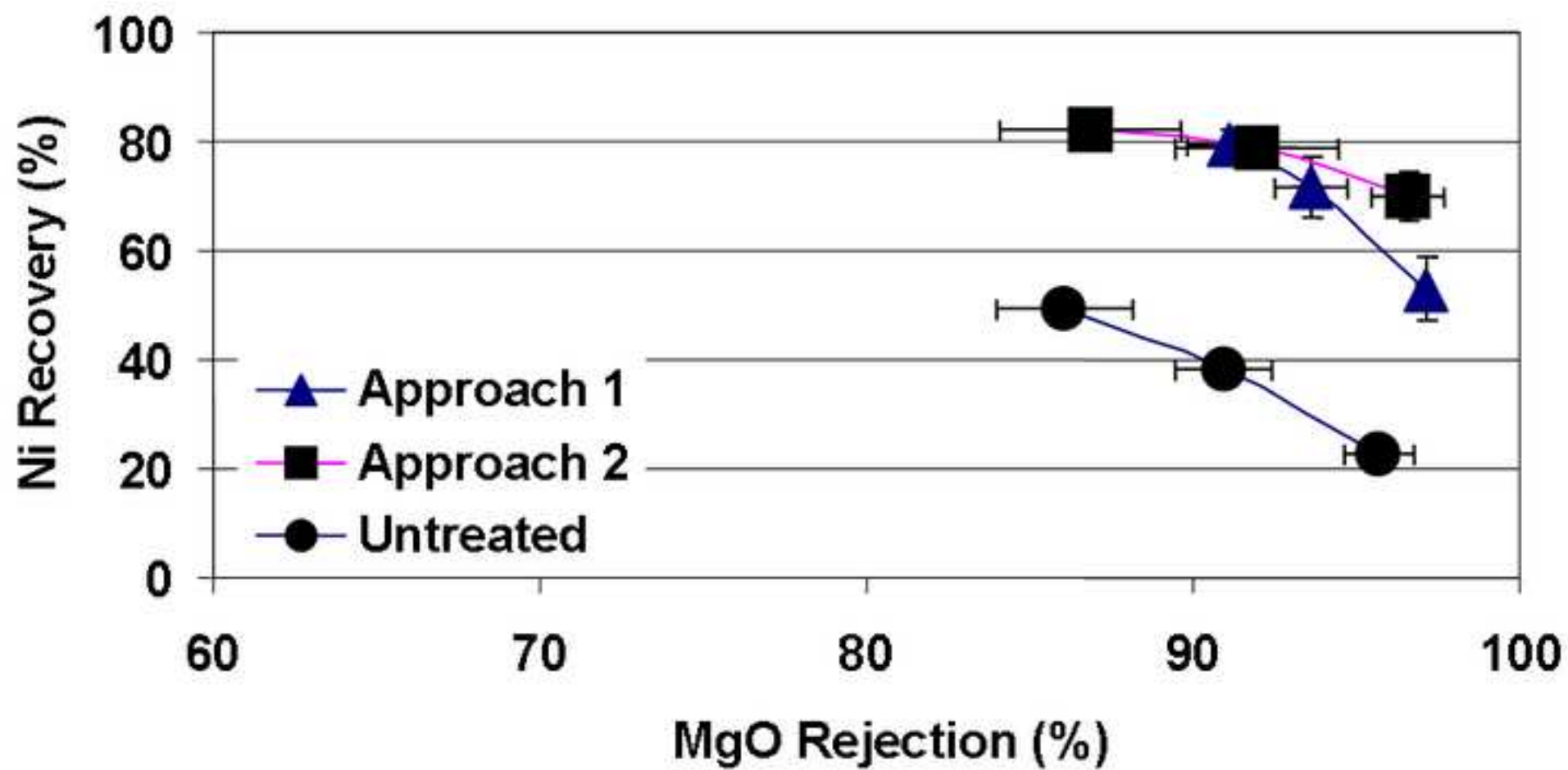
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Fig6

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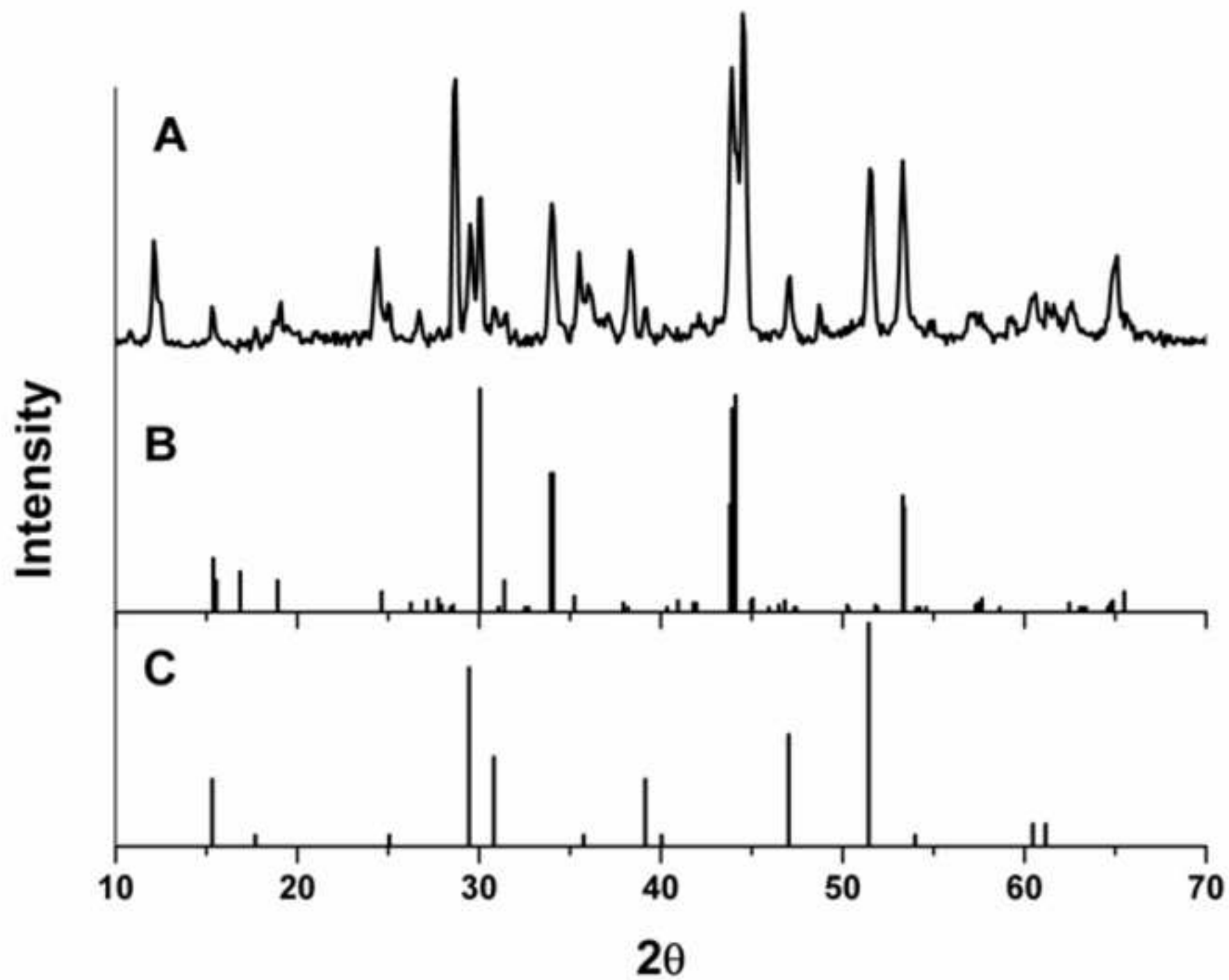


Fig7

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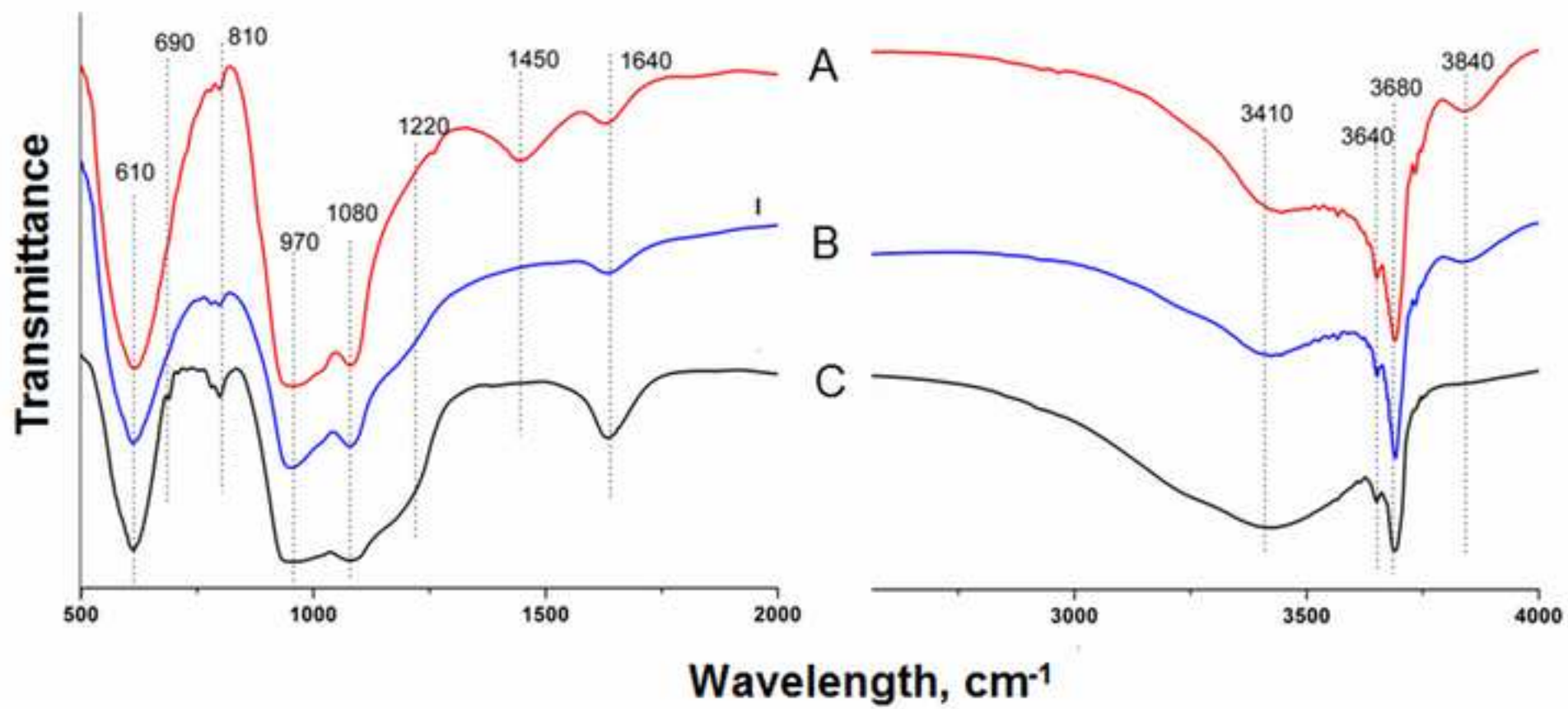


Fig8

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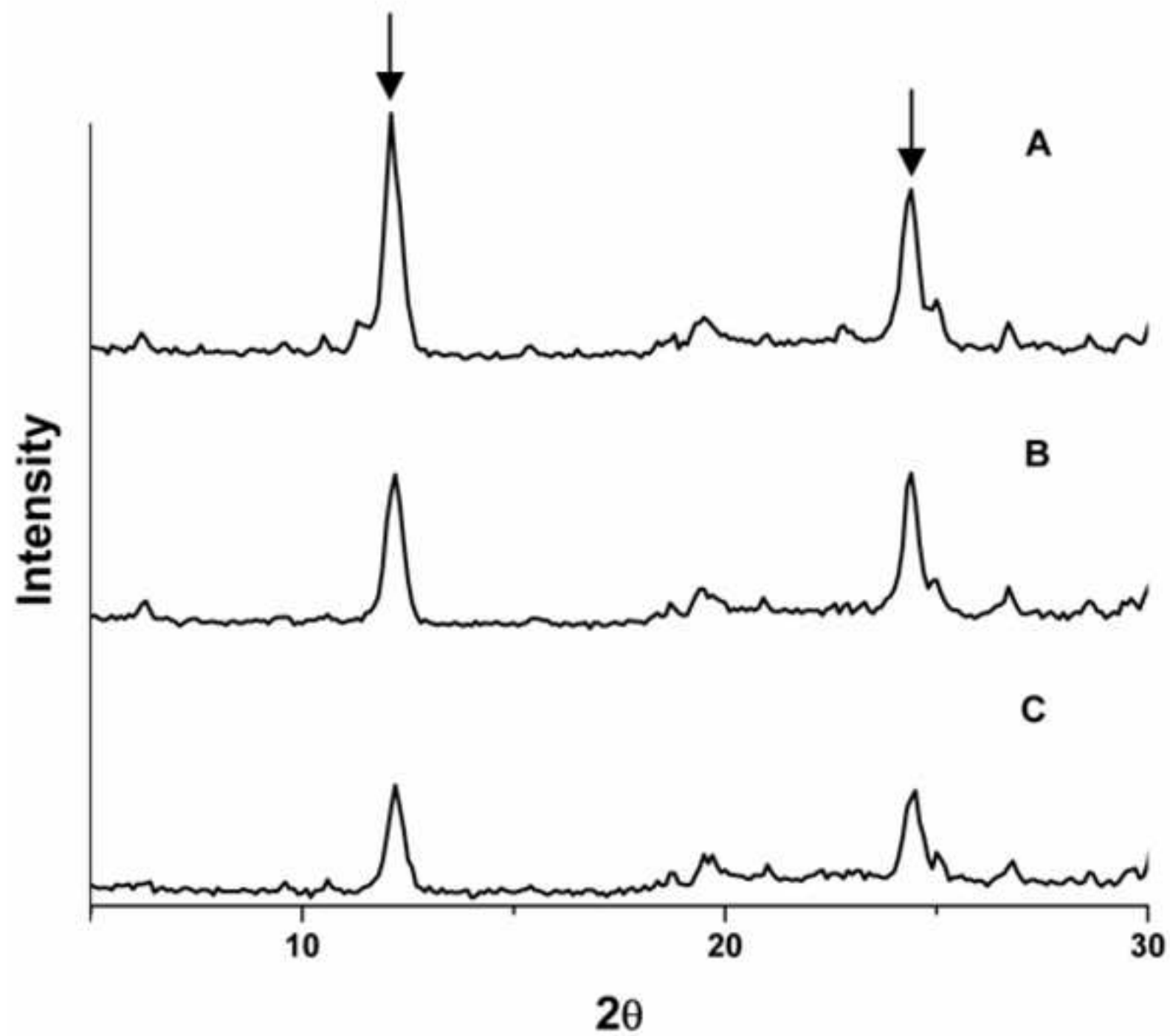


Fig9

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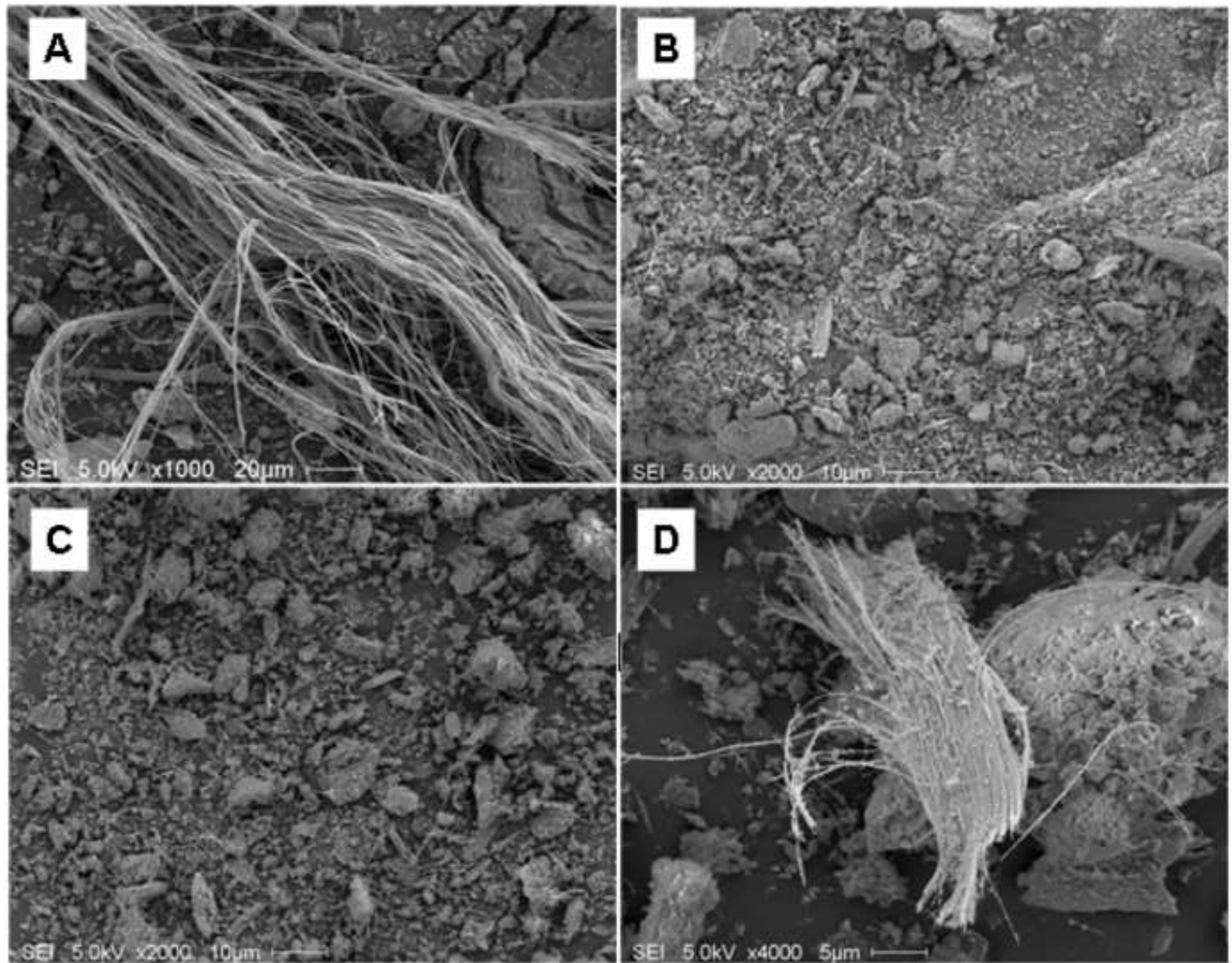
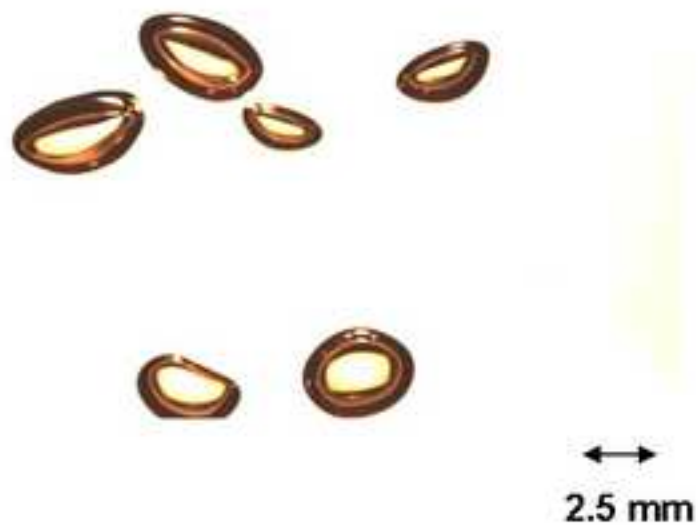


Fig10

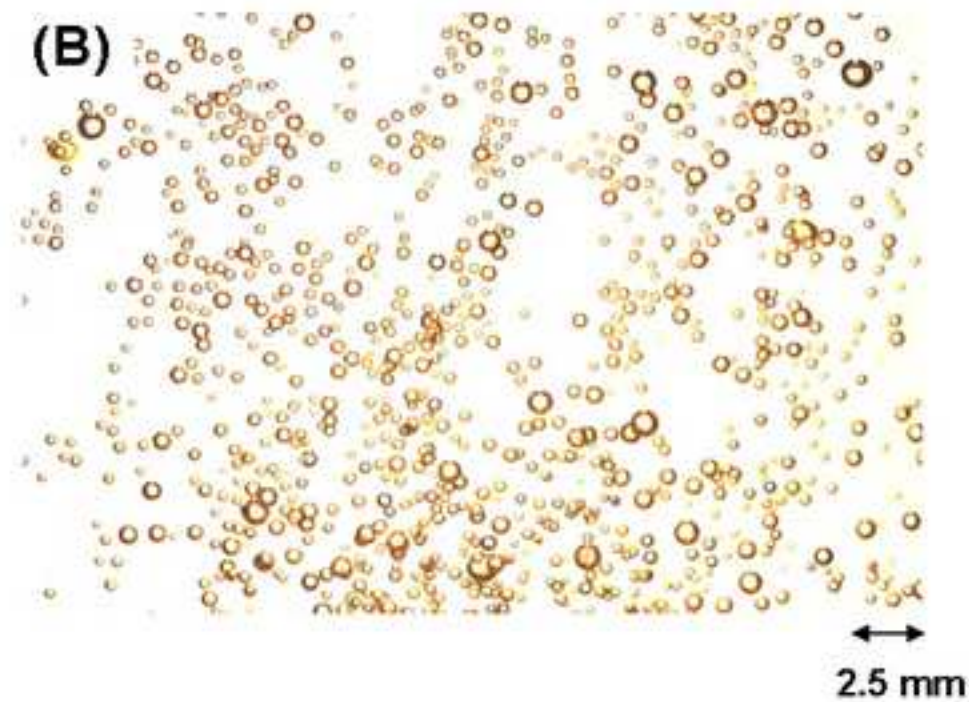
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(A)



$$d_{10} = 3.94 \text{ mm}; d_{32} = 4.49 \text{ mm}$$

(B)



$$d_{10} = 0.55 \text{ mm}; d_{32} = 0.63 \text{ mm}$$

Fig11

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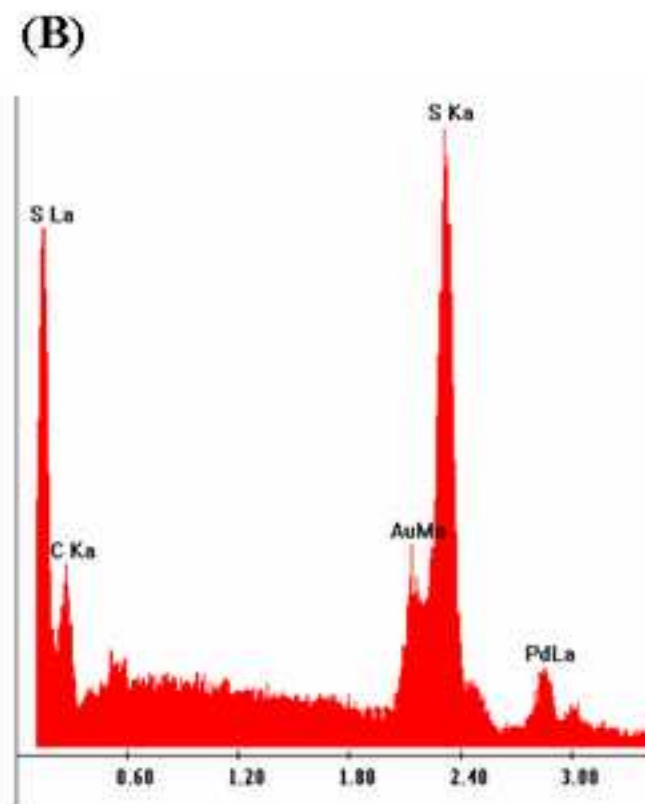


Fig12

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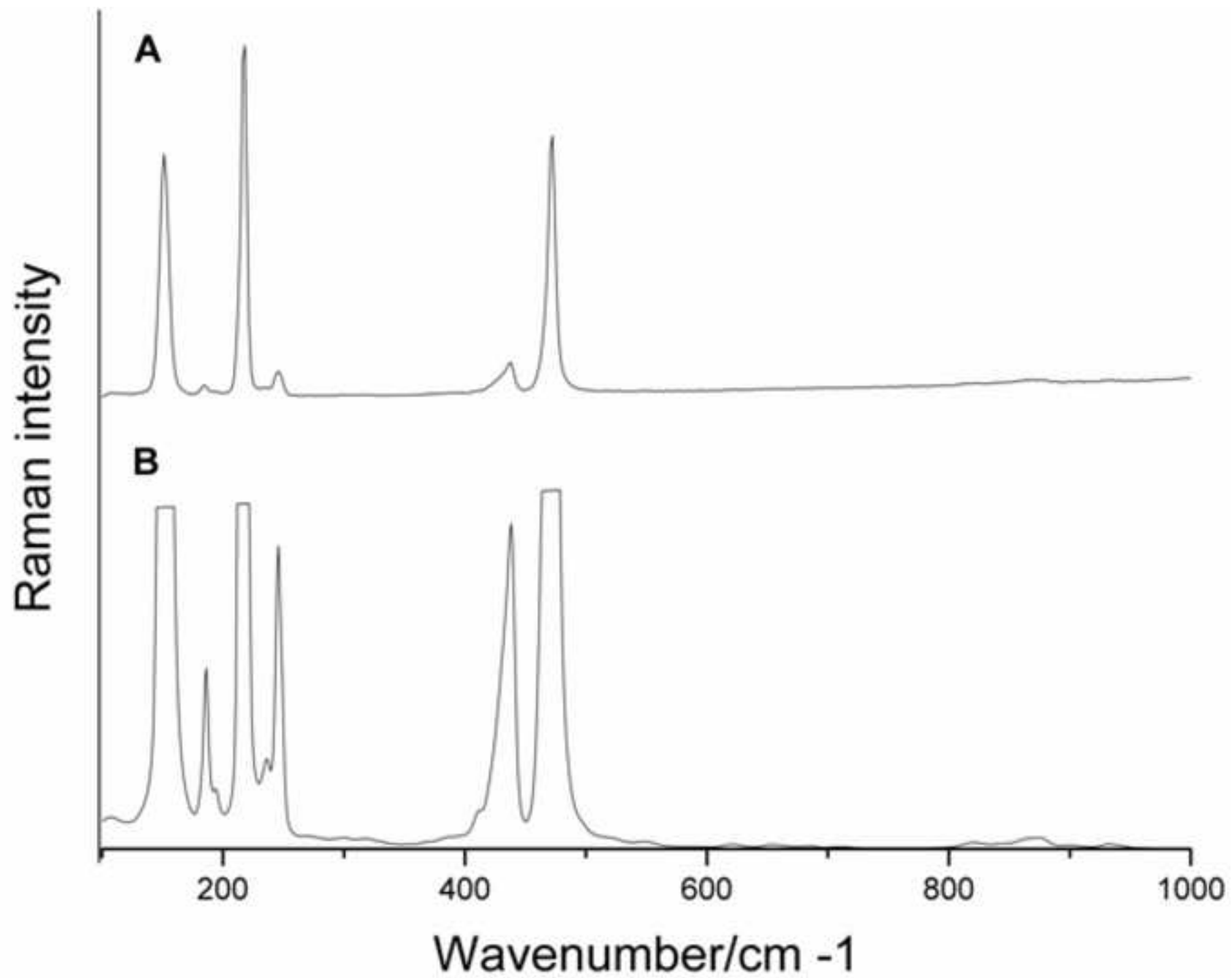
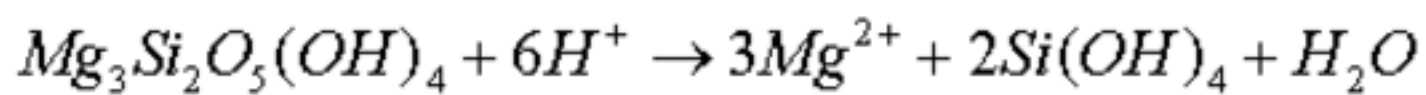


Fig13

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Chrysotile fibre

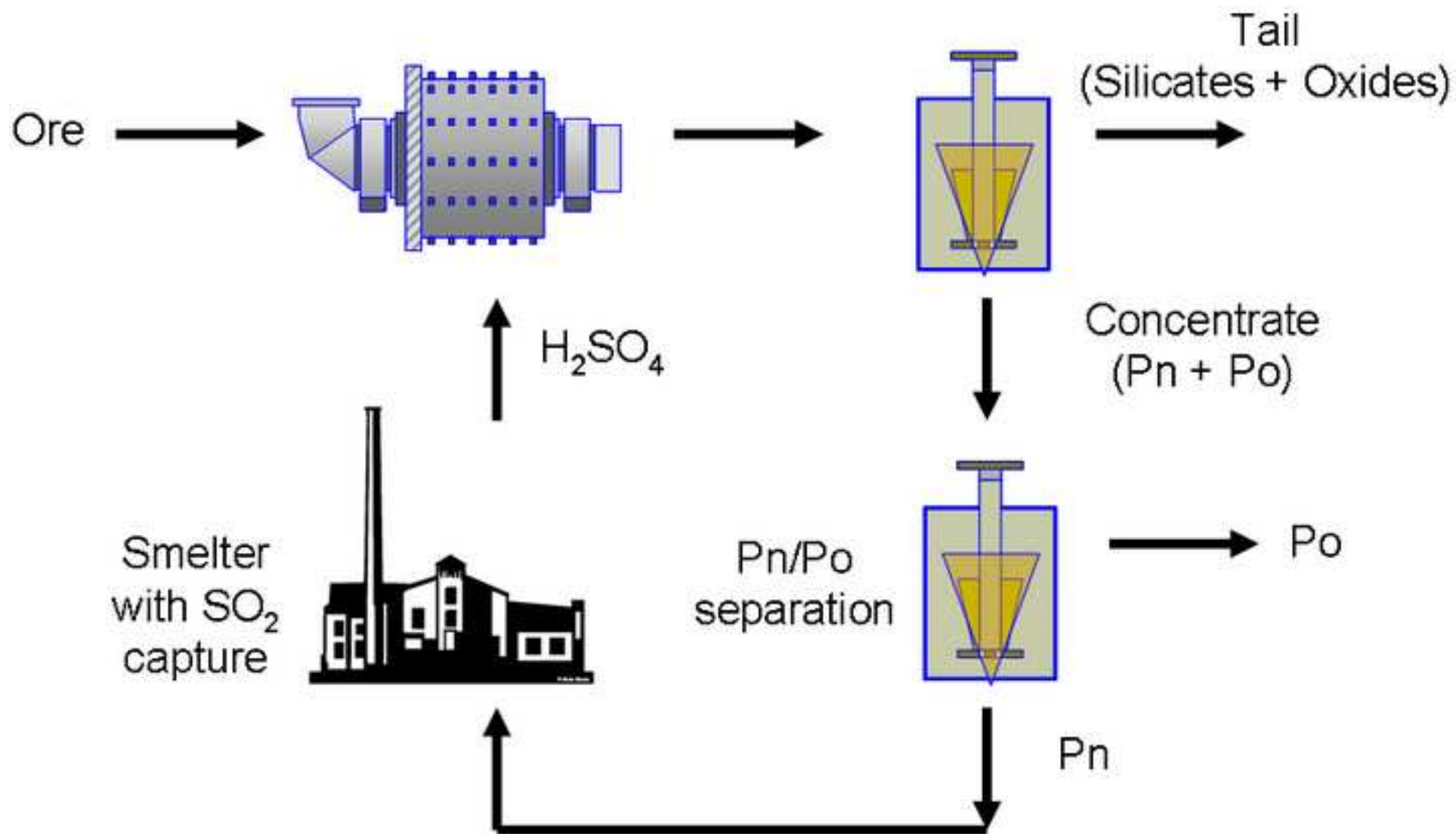


Grinding media



Fig14

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