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Indium extraction from indium tin oxide (ITO) scraps using subcritical water

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ABSTRACT

Indium (In) extraction from indium tin oxide scraps was investigated using dilute acid solutions under subcritical condition. Three types of acids, including HCl, H_2SO_4, and HNO_3, were used and HCl yielded the highest extraction efficiency. The pronounced complex formation between In^{3+} and Cl^- was proposed to be involved in determining extraction efficiency. The extraction efficiency of In increased with HCl concentration and reached 95.41% ± 0.01% within 30 min at 0.75 M of HCl, 150°C, and final pressure of 20 bar. Pressure did not affect the efficiency as it varied from 10 to 60 bar. Efficiency as high as 94% was found at 100°C, implying that subcritical water extraction (SWE) was very effective in the range of 100°C–150°C. The present work demonstrates that SWE is an efficient, simple, and effective extraction technique for In extraction.

Keywords: Extraction; Indium; Indium tin oxide (ITO); Metal complexes; Subcritical water

1. Introduction

The rapid developments of electronic industry nowadays have led to mass production of varieties of electronic devices whose life cycle is shorter than conventional ones. When these devices become obsolete, they become electronic waste (e-waste). e-Waste is the fastest growing waste stream in the world, and significant amount is being produced annually [1,2]. Globally, more than 41.8 million metric tons of e-waste was discarded in 2014 and 65.4 million metric tons are forecasted to be disposed of in 2017 [3]. Waste liquid crystal display (LCD), used as screen of computer, cell phone, and television, is a typical e-waste that is accumulating rapidly [4]. Since rare metals are essential in manufacturing of electronic devices, their recovery and reuse have become a critical issue. A typical rare metal, indium (In) has been widely used in optoelectronic, semiconductor, and photovoltaic industry mainly in the form of indium tin oxide (ITO) [5]. The ITO, composed of mainly 90% In_2O_3 and 10% SnO_2, is used in the manufacturing process of LCD panels and accounts for nearly 80% of the total In supply [6]. However, In is a by-product commodity from primary ores of aluminum (Al) and zinc (Zn) with limited production. Therefore, there is a great interest in the development of recovery technology of In from wastes [7].

Extraction of In from wastes has been vigorously studied using conventional technologies, such as acid leaching [8,9], solvent extraction [10–12], and chlorination [2,11]. These processes involve strong acids, organic solvent, and long reaction time. In order to obtain high yield of In from its sphalerite ores, the pressurized acid leaching (PAL) method has been widely used in hydrometallurgical process. Li et al. [13] reported high recovery of In from sphalerite by PAL. Yet, this process required high operating cost and energy consumption [14]. Supercritical carbon dioxide (sc-CO_2) extraction coupled with chelating agent exhibited a good efficiency of In [15,16]. However, it became inefficient when in the absence of chelating agent. The phosphorus-containing chelating agent may cause environmental concern as well.

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Alternatively, subcritical water extraction (SWE), a pressurized hot water system, has been examined for extraction purposes, in which liquid water is used at temperatures above the atmospheric boiling point of water (100°C, 0.1 MPa) but below the critical point of water (374°C, 22.1 MPa) [17]. SWE has been shown effective and efficient for recovery of valuable resources from solid wastes with hydrolysis reaction [18], synthesis of biodiesel from vegetable oils and waste activated sludge [19], as well as metal extraction and fatty acid production [20]. It has been applied in removal of heavy metals from sea sand and sewage sludge [21], and soil remediation [22,23]. Recently, it has shown effective in extracting yttrium (Y) and europium (Eu) from waste cathode-ray tube (CRT) phosphor [24]. When applied for In extraction from waste LCD panel, high extraction efficiency was found [25]. However, In content in the waste is very low (716 mg/kg) and glues and organic films in the waste tend to interfere with the reaction. Thus, waste LCD panel may not be an attractive source for In recovery.

The mechanism of extraction is important to evaluate the extraction of In when in the presence of ligands. To date, there is no information available on recovery of In from ITO scraps using dilute acid solutions under subcritical condition associated with the mechanisms of metal complex. In the current study, a bench-scale apparatus was used to extract In from ITO scraps. Different types of acids under different concentration were examined. Effects of temperature and pressure on In extraction were investigated. In addition, results from SWE were compared with those from non-SWE in terms of extraction efficiency of In from ITO scraps as well.

2. Materials and methods

ITO scraps were obtained from the recycling process at UWin Nanotech Co, Ltd., Taiwan. ITO scraps were pulverized and screened through a 140 mesh sieve. The morphology and composition of the ITO scraps were assessed by the field emission scanning electron microscope equipped with energy-dispersive X-ray spectroscopy (FESEM-EDX, JSM-6390LV, JEOL). The sample was coated with gold for 100 s to enhance the conductivity. The acceleration voltage was set at 15 kV and the pressure in chamber was kept below 4.41×10^{-4} Pa. Total metal content was determined by aqua regia digestion, in which 3.0 g of dry sample was placed in the plastic bottle, and then 1.0 mL water was added to rinse the samples. Concentrated hydrochloric acid (HCl, Acros, Advantec) was then added (21 mL), followed by slow addition of 7.0 mL concentrated nitric acid (HNO_3, Acros, Advantec). This suspension was then allowed to react at room temperature for 16 h. The suspension later was heated to boiling point and reflux for 2 h. After cooled to room temperature, 0.5 M HNO_3 was used to wash the condenser. The suspension was transferred to a 100 mL volumetric flask and water was added to a final volume of 100 mL. It was filtered using a 0.2 mm mixed cellulose ester membrane (Advantec) and analyzed by the inductively coupled plasma-atomic emission spectrometry (ICP-AES, JY2000) at wavelengths of 230.606, 309.271, and 242.949 nm, for In, Al, and Sn, respectively. The detection limit of In, Al, and Sn was 63.0, 23.0, and 96.0 μg/L, respectively. In SWE experiments, all chemicals were analytical reagent grade.

Fig. 1 shows the bench-scale subcritical water extractor equipment used in this study. The reactor was made of stainless steel (SS 316) with total volume of 290 mL. The reactor had an external electric heater, a pressure gauge, a thermocouple (K-type), and a lid with screws. Magnetic stirrer was used for mixing. Measured amount of ITO scraps was mixed with acid solution to make 10 g/L suspension, which was placed in the 50-mL glass chamber. The insulated reactor was then sealed. N_2 gas was directed into the reactor to remove air and give an initial pressure at set value before heating. The reactor was heated from room temperature to the desired temperatures (100°C–150°C). The reaction time was 30 min, counting when the desired temperature was reached. The reaction time was chosen because preliminary experiments showed insignificant changes after 30 min. The pH of the suspension was measured by a pH meter (Thermo Scientific Orion Star A215) when reaction completed, and the reactor was cooled to room temperature. The suspension was filtered using a 0.2 mm mixed cellulose ester membrane (Advantec) and the metal concentration in the filtrate was then analyzed by ICP-AES. The solid residue was washed twice using deionized water and dried in an oven at 60°C for 24 h before analysis. Three acids (HCl, H_2SO_4, and HNO_3) at 0.75 M were first used and the reactions were conducted at 125°C and initial pressure of 10 bar. The effects of acid concentration (0.01–1 M), pressure (1–50 bar), and temperature (100°C–150°C) were then examined. All experiments were triplicated and the average value was taken.

3. Results and discussion

3.1. Characteristics of ITO scraps

FESEM image shows rod-shape ITO scraps with nonuniform size as described in Fig. 2(a). The EDX analysis revealed

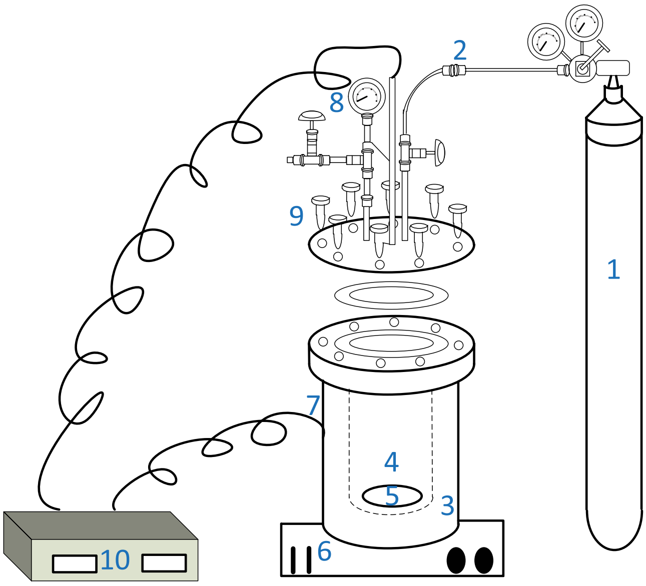

Fig. 1. Schematic diagram of the laboratory-scale of SWE equipment: (1) N_2 cylinder gas; (2) valve; (3) reactor; (4) glass chamber; (5) magnetic stirrer; (6) hot plate; (7) electrical heater; (8) pressure gauge; (9) screws; and (10) temperature controller.

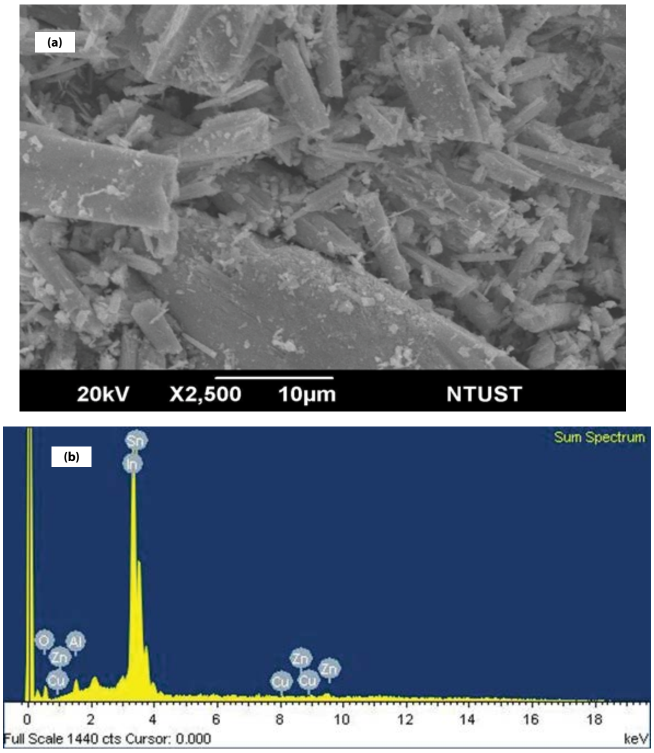

Fig. 2. (a) FESEM images of ITO scraps with magnification of 2,500× and (b) EDX analysis of ITO scraps.

that ITO scraps consisted of various metals such as In, Sn, Al, Zn, and Cu (Fig. 2(b)). Results of aqua regia digestion showed that the main elements included In (85.75%), Sn (12.04%), Al (1.62%), Zn (0.38%), and Cu (0.20%) as listed in Table 1.

3.2. Effects of acid type and acid concentration

The extraction efficiency is defined as the concentration of In in the extractant divided by the total concentration of In of ITO scraps. Three types of acids were used in the SWE of In, namely 0.75 M of HCl, 0.375 M of H_2SO_4, and 0.75 M of HNO_3. Table 2 shows that HCl solution yielded the highest extraction efficiency (92.15% ± 0.02%), followed by H_2SO_4 (83.86% ± 0.01%) and HNO_3 (81.84% ± 0.01%). Therefore, HCl solution was chosen for further experiments due to the higher extraction and lower cost amongst other mineral acids.

Acetyl acetone (AcAc) was added in the SWE to enhance the extraction. AcAc is commonly used as a chelating agent in the extraction of metals due to its ease of complex formation, high solubility, and the thermal stability of the metal complexes it forms [26]. In the preliminary experiment, it was found that the extraction efficiency of In increased from 88.25% to 94.10% upon the addition of 0.5 mL of AcAc, and the dose was adopted in the study. The possible mechanism of In extraction in the presence of AcAc was the formation of a symmetrical complex formation in which metal ion (In) is ionically bonded to the keto groups of AcAc in the acidic solutions as presented in Fig. 3, which was illustrated on the extraction of Cu in the presence of AcAc [27]. The effect of concentration of HCl on SWE of In from ITO scraps was studied and results are depicted in Fig. 4. When at 0.1 M of HCl and final pH of 2.21, ITO scrap was not significantly dissolved and only (23.07% ± 0.01%) of In was extracted. The extraction efficiency increased remarkably with increasing acid concentration, and reached (90.59% ± 0.02%) at 0.5 M HCl. It further increased to (94.10% ± 0.02%) and (98.27% ± 0.01%) at 0.75 and 1 M HCl, respectively. The metal concentration in the SWE extractant was 8,072.49 ± 52.20 mg/L for In, 1,126.14 ± 75.31 mg/L for Sn, and 150.51 ± 0.81 mg/L for Al, respectively. It was observed that acid concentration was an important factor affecting SWE of In. In this study, 0.75 M HCl could be a better choice of aqueous phase in the following experiments, since the high extraction efficiency was achieved (>94%). It was also intended to make the system less intense.

Table 1
Compositions of ITO scraps determined by aqua regia digestion

Element	Concentration (mg/L)	Mass (%)
In	8,214.67	85.75
Sn	1,153.61	12.04
Al	155.52	1.62
Cu	18.82	0.20
Zn	36.70	0.38

Table 2
Effects of acid type on SWE of In at solid concentration of 10 g/L, 125°C, and final pressure of 20 bar

Acid used	Extraction (%)	pH_i	pH_f
0.75 M HCl	92.15 ± 0.02	0.25 ± 0.01	0.45 ± 0.01
0.375 M H_2SO_4	83.86 ± 0.01	0.42 ± 0.01	0.56 ± 0.03
0.75 M HNO_3	81.84 ± 0.01	0.14 ± 0.01	0.24± 0.01

For comparison, conventional extraction of In by using 1 M HCl under temperatures from 30°C to 60°C was conducted using the same equipment. Experiments were carried out at identical solid concentration of 10 g/L for 3 h. Fig. 5 indicates that the extraction efficiency increased with increasing temperature. As much as 49.58% ± 0.05% of

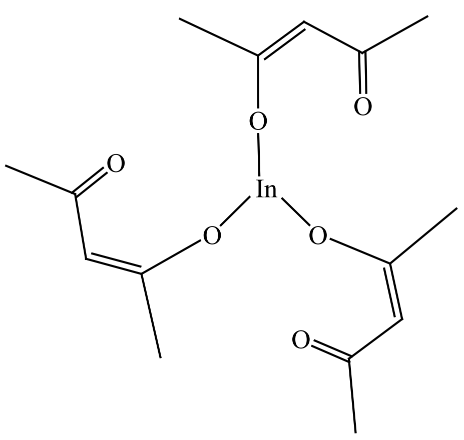

Fig. 3. Structure of In-acetyl acetone complex in an acidic medium.

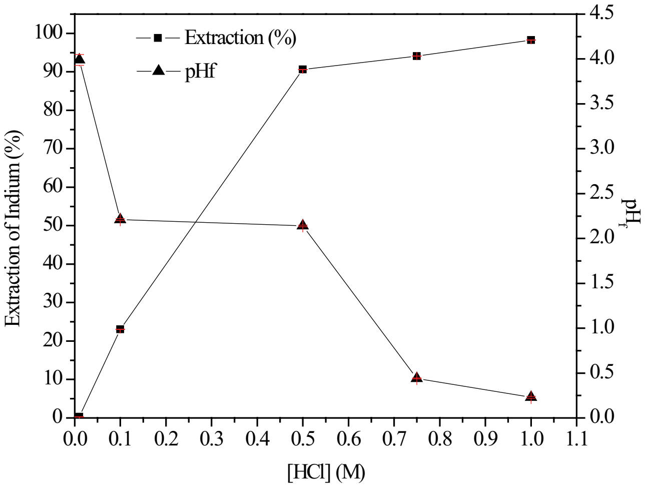

Fig. 4. Effect of HCl concentration on SWE of In at solid concentration of 10 g/L, 100°C, final pressure of 20 bar, 0.5 mL of AcAc, and reaction time of 30 min.

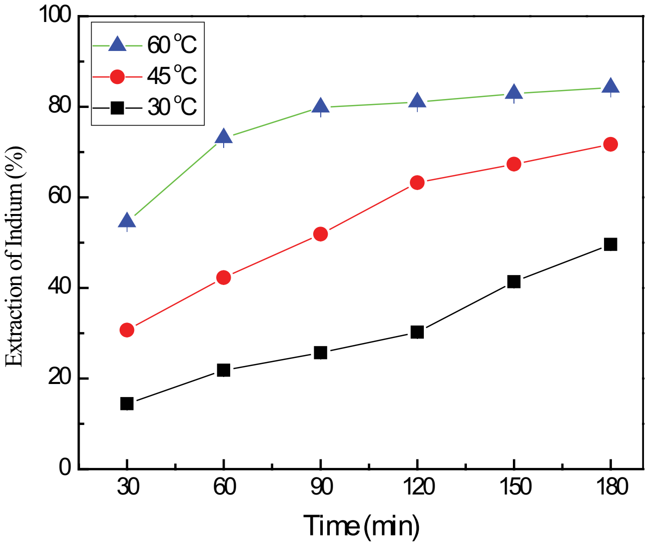

Fig. 5. Extraction of In using 0.75 M of HCl at solid concentration of 10 g/L and temperature of 30°C–60°C.

In was extracted at 30°C. At 60°C, the extraction efficiency increased to 84.25% ± 0.06%. Compared with SWE, conventional extraction for In by using HCl was not as efficient and a longer reaction time was required. It is clear that SWE has some advantages for extracting valuable metals from wastes.

3.3. Complex formation

It was observed that the initial pH of acid solution when using three different acids ranged from 0.14 to 0.42, while the final pH shifted to the range of 0.24–0.56. Even though SWE efficiency increased as HCl became more concentrated, it appeared that pH was not the sole parameter for SWE of In from ITO scraps (Fig. 4). The initial pH was 0.14 for 0.75 M of HNO_3 solution, and 81.84% of In was extracted. For comparison, the best extraction efficiency (92.15% ± 0.02%) was found using 0.75 M of HCl solution and the final pH was 0.45 ± 0.01. It implied that lower pH did not necessarily lead to higher efficiency.

Complex formation between In^{3+} and ligands, such as NO_3^-, SO_4^{2-}, and Cl^-, was then assessed. The equilibrium constants of metal complex are presented in Table 3 [28]. It was apparent that the metal complexes of In^{3+} and Cl^- have the highest equilibrium constants than those of In^{3+} and NO_3^-, and In^{3+} and SO_4^{2-}. The order of SWE efficiency of HCl > H_2SO_4 > HNO_3 was the same as the equilibrium constants of metal complexes. The higher affinity between Cl^- and In^{3+} was therefore proposed to play a key role, along with pH, in the extraction reactions. The speciation diagram of In^{3+} and ligands as affected by solution pH was assessed. Fig. 6(a) shows the speciation of In in aqueous solution in the presence of Cl^- as affected by pH. It was observed that In–Cl complexes were the dominant species for pH < 3.5. For example, when at the equilibrium pH (0.45 ± 0.01) of SWE using 0.75 M of HCl, the dominant species was all metal complexes, including 70% of $InCl_3$ and 30% of $InCl_2^+$. The speciation of In in aqueous solution in the presence of SO_4^{2-} was also investigated and the result is shown in Fig. 6(b). At the equilibrium pH (0.56 ± 0.03) of SWE of In using 0.375 M of H_2SO_4, In in complex form included $InSO_4^+$ (43%) and $In(SO_4)_2^-$ (4%), while the most dominant species was In^{3+} ions which constituted 53%. For In-NO_3 system, at the equilibrium pH (0.24 ± 0.01) of SWE of In using HNO_3, the main species was In^{3+} (38%), followed by $In(NO_3)^{2+}$ (37%), $In(NO_3)_3$ (17%), and $In(NO_3)_2^+$ (8%) (Fig. 6(c)). Therefore, it was proposed that complex formation was involved in In extraction from ITO scraps. In the following experiments, HCl was chosen as extraction agent.

3.4. Effect of temperature and pressure

The effect of temperature on extraction of In from ITO scraps was studied at 0.75 M of HCl, initial pressure of 10 bar and final pressure of 20–25 bar. Table 4 shows that as temperature increased from 100°C to 150°C, the extraction efficiency increased slightly from 94.10% ± 0.02% to 95.41% ± 0.01% with corresponding increase of In concentration from 7,730.40 ± 176.95 mg/L to 7,837.24 ± 41.69 mg/L. The concentration of Sn and Al increased from 377.17 ± 19.77 mg/L to 435.78 ± 2.26 mg/L, and from 145.96 ± 6.76 mg/L to 157.78 ± 0.96 mg/L, respectively. Results revealed that SWE of In from ITO scraps was very effective and the efficiency

Table 3
Complex reactions of In and their equilibrium constant [28]

Reaction	Log *K* at 25°C
$In^{3+}_{(aq)} + Cl^-_{(aq)} \leftrightarrow InCl^{2+}$	2.32 ± 0.05
$In^{3+}_{(aq)} + 2Cl^-_{(aq)} \leftrightarrow InCl_2^+$	3.62 ± 0.05
$In^{3+}_{(aq)} + 3Cl^-_{(aq)} \leftrightarrow InCl_3$	4.0 ± 0.2
$In^{3+}_{(aq)} + SO^{2-}_{4(aq)} \leftrightarrow In(SO_4)^+$	1.78
$In^{3+}_{(aq)} + 2SO^{2-}_{4\ (aq)} \leftrightarrow In(SO_4)_2^-$	1.88
$2In^{3+}_{(aq)} + 3SO^{2-}_{4\ (aq)} \leftrightarrow In_2(SO_4)_3$	2.36
$In^{3+}_{(aq)} + NO^-_{3(aq)} \leftrightarrow InNO_3^{2+}$	0.18
$In^{3+}_{(aq)} + 2NO^-_{3(aq)} \leftrightarrow In(NO_3)_2^+$	–0.3

Table 4
Effect of temperature on SWE of In at solid concentration of 10 g/L and 0.5 mL AcAc

[HCl] (M)	*T* (°C)	Extraction (%)	[In] (mg/L)	[Sn] (mg/L)	[Al] (mg/L)
0.75	100	94.10 ± 0.02	7,730.40 ± 176.95	377.17 ± 19.77	145.96 ± 6.76
0.75	125	94.71 ± 0.05	7,780.36 ± 44.42	395.65 ± 4.70	149.12 ± 3.58
0.75	150	95.41 ± 0.05	7,837.24 ± 41.69	435.78 ± 2.26	157.78 ± 0.96

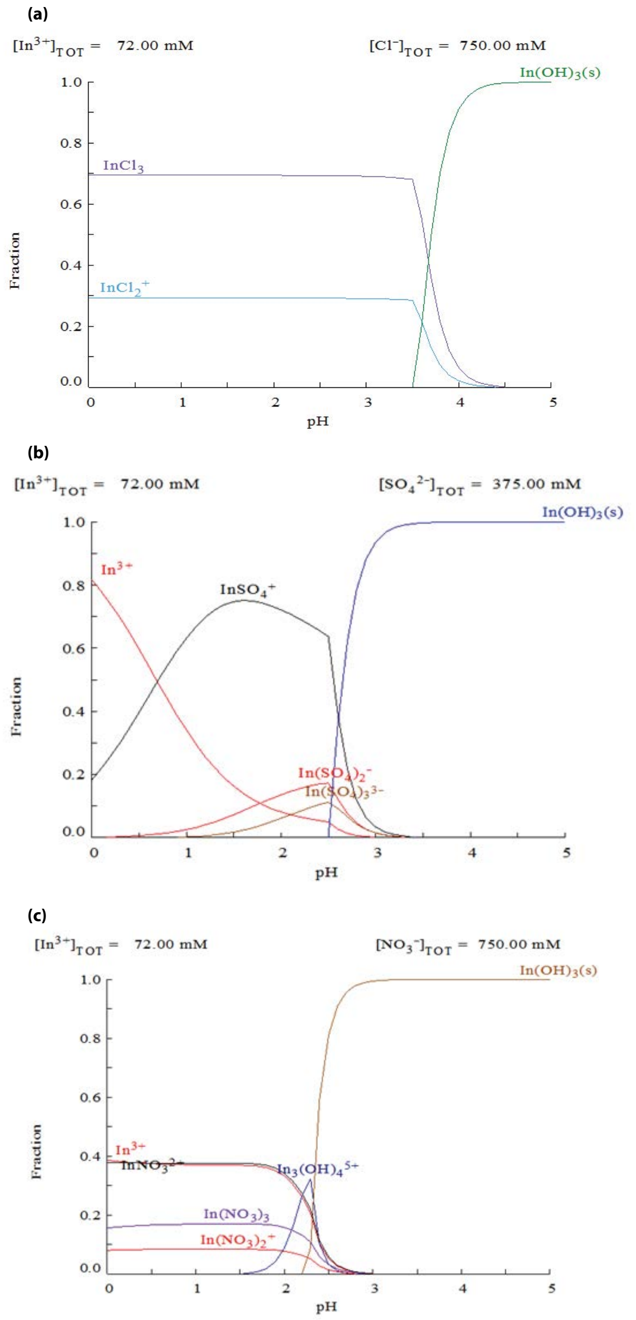

Fig. 6. (a) Speciation diagram of In in HCl solution as affected by pH at 25°C, (b) Speciation diagram of In in H_2SO_4 solution as affected by pH at 25°C, and (c) Speciation diagram of In in HNO_3 solution as affected by pH at 25°C.

as high as 94.10% ± 0.02% was obtained at 100°C. It has been indicated that SWE condition was in the temperature range between 100°C and 374°C where the concentration of hydronium (H_3O^+) and hydroxide (OH^-) are greatly influenced by the self-ionization of water molecules, thus enhance the fast mass transfer, high solubility, and diffusivity [29]. Therefore, it increases the extraction efficiency of In. However, the increase of temperature from 100°C to 150°C did not significantly increase the extraction efficiency because high extraction efficiency (94.10% ± 0.02%) of In was already achieved at 100°C. Hence, further increase of temperature to 125°C and 150°C did not significantly increase the extraction efficiency.

One of the parameters that may influence SWE of In is pressure, which could change the phases of water [30]. The effect of pressure on extraction of In from ITO scraps was examined at 0.75 M of HCl and 150°C. Four different initial pressures of 1, 10, 30, and 50 bar were selected for the experiments. It was observed that the final pressure increased from 1 to 10.17 ± 0.29 bar, and to 60.50 ± 0.50 bar from initial pressure of 50 bar (Table 5). The extraction efficiency did not change as final pressure varied, showing that pressure had no significant effect on In extraction. Actually, the pressure is mainly applied to maintain water in its liquid state [31]. Pressure also had insignificant effect on SWE for In from waste LCD panels [25,32]. Similarly, it had minor effect on immobilization of heavy metals under SWE treatment [23].

3.5. Comparison of extraction technologies

Recent study demonstrated that In of as high as 95% could be extracted from marmatite by PAL [33] (Table 6). Nevertheless, some problems such as high concentration of leaching agent, high operating cost, and safety risk of the zirconium autoclaves were pointed out. The method of SC-CO_2 extraction could reach 85% or 90.8% through proper selection of chelating agent. However, the main disadvantage may be the use of high amount of phosphorus-containing piperidinyldithiocarbamic acid (NCS_2H) as chelating agent which

Table 5
Effect of pressure on extraction of In at solid concentration of 10 g/L, 150°C, and 0.5 mL of AcAc

Initial pressure (bar)	Final pressure (bar)	Extraction (%)
1	10.17 ± 0.29	95.52 ± 0.05
10	20.33 ± 0.29	94.85 ± 0.01
30	40.17 ± 0.29	95.57 ± 0.08
50	60.50 ± 0.50	95.74 ± 0.03

Table 6
Comparison of In extraction using SWE and supercritical CO_2 (SC-CO_2) extraction

Method	Source	*T* (°C)	*P* (bar)	Solution	In (%)	Reference
SC-CO_2 extraction	Acidic solution	70	138	NCS_2H: In(III) = 10:1	85	[15]
SC-CO_2 extraction	Etching wastewater	80	207	HFOD: In(III) = 3:1	90.8	[16]
SWE	Waste LCD panel	360	N/A	7% HCl	83 (CF glass) 7 (TFT glass)	[25]
NaOH-enhanced SWE	Waste LCD panel	160 (CF glass) 220 (TFT glass)	N/A	0.1 N NaOH + 7% HCl	99 (CF glass) 95 (TFT glass)	[32]
SWE	ITO scraps	100	20	0.75 M HCl	94.10	This work

HFOD: fluorinated a-diketone; CF: color filter; TFT: thin-film transistor.

may result in environmental concern [15]. In comparison, higher extraction efficiency was generally obtained by using SWE than SC-CO_2. When SWE was applied to In recovery from waste glass of LCD panel, very high temperature was required for the removal of the organic materials which were attached to the glass [25]. Much higher energy consumption may render the process uneconomical. In case NaOH was used in SWE for In recovery, the presence of 0.1 N of NaOH gave efficiency of 95% at 220°C and 99% at 160°C [32]. Yet, the method may have the drawback of very low In content in the waste glass. The present study shows the merits of SWE when applied to ITO scraps because of the high efficiency, simplicity, short reaction time, and no need for organic solvent. The environment-friendly method deserves further investigation to make it suitable for industry-scale production.

4. Conclusions

The extraction of In from ITO scraps using dilute acid solution under SWE was examined. The extraction was very efficient as the extraction efficiency reached >94.10% at moderate temperature (100°C), mild acid strength (0.75 M of HCl), and short reaction time of 30 min. The SWE efficiency increased with HCl concentration and temperature, while pressure did not affect the efficiency. Through assessment of speciation, it was proposed that the complex formation reactions between In^{3+} and Cl^- contributed to better performance of HCl over H_2SO_4 and HNO_3. This study shows SWE a promising technology for extraction of In from ITO scraps, which is simple, effective, and efficient.

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