

# Recycling Indium from Scraped Glass of Liquid Crystal Display: Process Optimizing and Mechanism Exploring

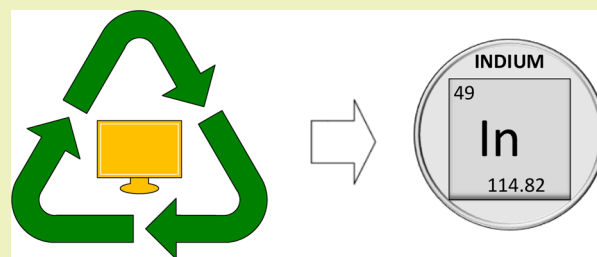
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## Supporting Information

**ABSTRACT:** With rapid growth in the use of liquid crystal display (LCD) and increasing concerns for environmental protection as well as conservation of scarce metals such as indium, the recycling of indium from waste LCDs is becoming a hot issue for current society. In this study, the leaching process for indium and exploration of its mechanism were carried out with full consideration of potential theory and experiments. The optimal parameters for the leaching process is controlled at a <math><75\ \mu\text{m}</math> sample size, 180 min retention time, 50 °C temperature,  $\text{H}_2\text{SO}_4$  for the leaching agent, 100 g/L initial concentration, and 1:1 liquid–solid ratio. The initial samples and leaching yields were examined with the necessary analytical techniques including SEM, XRF, EDS, XRD, FTIR, and ICP. We also found that the leaching process could largely modify raw material and enhance its activation for further recovery. All the obtained results and findings could contribute to affording a closed-loop recycling process for waste LCDs and sustainable development of indium industries.

**KEYWORDS:** Recycling, Indium, Liquid crystal display, E-waste, Process, Green chemistry



## INTRODUCTION

Liquid crystal displays (LCDs) have replaced cathode ray tubes (CRTs) and have been widely utilized as a fundamental component in most consumer electronics, including televisions, personal computers, laptop computers, tablet computers, smart phones, and eBook readers.<sup>1</sup> While the electronics are discarded with a 3–5 years lifespan, a huge amount of LCDs have been made obsolete worldwide.<sup>2</sup> China, for instance, will generate approximately 100 million units of e-waste of televisions, personal computers, and laptop computers from 2014 to 2020.<sup>3</sup> On the other hand, approximately 55% to 85% of global indium has been utilized in LCDs as indium–tin oxide (ITO) films.<sup>4</sup> It is reported that at least 80%—in fact, more than 90% in 2013—of the global indium production was used for ITO.<sup>5,6</sup>

Indium is a crucial and scarce metal and most commonly recovered from the zinc-sulfide ore mineral sphalerite.<sup>7</sup> According to the U.S. Geological Survey, the recoverable reserves of indium are 2800 t, and the confirmed reserves are roughly 6000 t. A rapid exhaustion of scarce natural indium elements (annual refinery production of 660–800 t) leads to an estimated availability of about 20 years until they will run out completely (Figure S1 of Supporting Information (SI)).<sup>8–10</sup> Additionally, exposure to hardly soluble indium compounds in ITO production and recycling plants can pose a new risk for public health such as lung damage.<sup>11–13</sup> To prevent this shortage and promote sustainable development of related industries, indium recycling from waste LCDs is an urgent necessity by means of an efficient approach.

The indium content in zinc ores varies from 10 to 20 g/t, which is far below its content (100–1400 g/t) in the LCDs of discarded mobile phones or televisions (SI Table S1). Indium recovery from the old scraps is not sufficiently economic to add significantly to secondary production.<sup>14</sup> Some technological processes in terms of indium recycling have been reported. Indium can be recovered from an acidic solution of ITO scrap using a simple hydrometallurgical approach and hot immersion techniques.<sup>15</sup> In order to recover indium from LCDs of mobile phones, hydrometallurgical processes,<sup>16,17</sup> pyrometallurgy,<sup>18</sup> and chloride induced vaporization are major potential candidates. A combined process with leaching using hydrochloric acid (HCl) and liquid–liquid extraction was employed for separation and concentration of indium.<sup>16</sup> By crushing ITO glass to micron size particles by high-energy ball milling, it is possible to extract a higher amount of indium at room temperature than that by conventional methods using only conventional shredding machines.<sup>19</sup> The direct smelting of LCD material in integrated smelters can ensure lower losses of indium than mechanical processing and hydrometallurgy. However, around 85% of the LCD panel comprises glass, which ends up in the slag after the smelting process. Consequently, this option means a disproportional high energy input to recover a very small amount of metal, what means an economic and environmental burden.<sup>20</sup>

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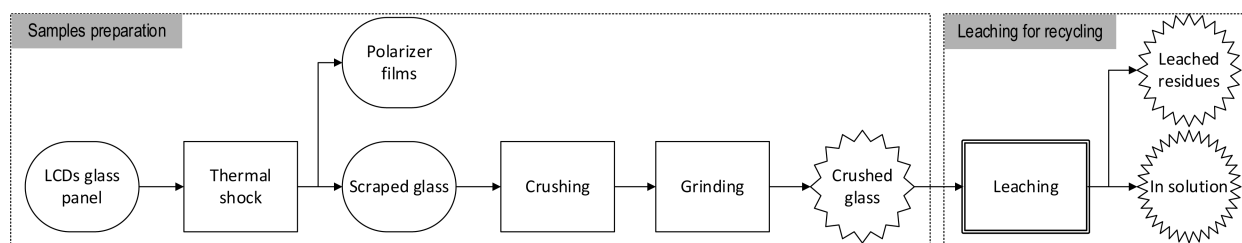


Figure 1. Proposed process of recycling indium from scrapped glass of LCDs.

Indium content in obsolete LCDs and several studies indicate acid leaching is appropriate for indium separation from the other materials.<sup>20</sup> In order to systematically and completely develop the indium recycling process from waste LCD, based on green chemistry,<sup>21</sup> this work was devoted to find the optimal leaching process and investigate its mechanism with full consideration of potential theory and experiments. The obtained results and findings could contribute to a closed-loop recycling process for waste LCDs and sustainable development of indium industries.

## EXPERIMENTAL SECTION

**Materials and Leaching Reactor.** The glass panel in LCDs consists of polarizer films, a glass substrate, an ITO-layer, and liquid crystals (SI Figure S2),<sup>22</sup> and the main physical materials in various components are presented at length in SI Table S2 and Figure S3. Scraped LCD glass pretreatment is usually indispensable to enriching the content of indium.<sup>23,24</sup> We collected the scraped LCD glass from the Tes-Amm Suzhou plant, whose ITO is a mixture of indium(III) oxide ( $\text{In}_2\text{O}_3$ ) and tin(IV) oxide ( $\text{SnO}_2$ ), roughly 80–90%  $\text{In}_2\text{O}_3$  and 10–20%  $\text{SnO}_2$  by weight. It was first crushed with a jawbreaker for 5-mm-size materials and then was grinded with a planetary ball mill to reinforce materials' activation.<sup>25</sup> Finally, the output product was sieved and cleaned using ultrasonography processing to obtain the samples for this study.<sup>26</sup> Then the samples of crushed glass was leached and recycled for indium solution (Figure 1). A leaching reactor was installed with a thermostatic bath and electronic relay (Figure 2).

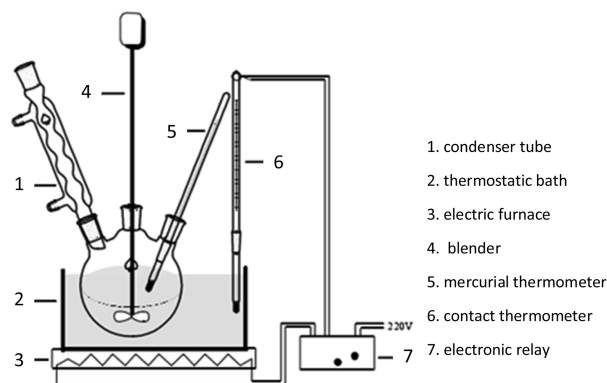
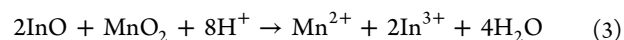
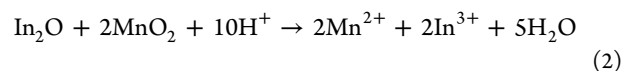
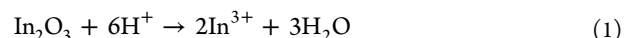


Figure 2. Leaching reactor in this study.

Table 1. Primary Factors and Levels in This Study

level	sample size ( $\mu\text{m}$ )	retention time (min)	temperature ( $^{\circ}\text{C}$ )	leaching agent	initial concentration (g/L)	liquid–solid ratio (g/L)	oxidant
1	>150	60	25	HCl	50	1:1	$\text{MnO}_2$ (1 g)
2	75–150	120	50	$\text{HNO}_3$	100	2:1	$\text{MnO}_2$ (2 g)
3	<75	180	100	$\text{H}_2\text{SO}_4$	200	3:1	none

**Experimental Measurement and Design.** Previous studies about thermodynamic considerations indicated that a complicated reaction and side-reaction commonly can occur while  $\text{In}_2\text{O}_3$  comes across acid (e.g., HCl). Chlorine, or three indium chloride species, i.e.,  $\text{InCl}$ ,  $\text{InCl}_2$ , and  $\text{InCl}_3$ , can be produced in the process (See SI Text1).<sup>27</sup> To accelerate the reaction rate and avoid a potential side-reaction of forming  $\text{Cl}_2$ , manganese dioxide ( $\text{MnO}_2$ ) is added to play a role as an oxidant, leading to a galvanic interaction with small amounts of  $\text{In}_2\text{O}$  and  $\text{InO}$  in samples. Therefore, potential reactions mainly involve the following.<sup>28</sup>



The mass content of indium and the recycling rate can be determined with eq 4 and eq 5.

$$\eta_0 = \frac{c_0 \times v \times 10^{-3}}{m_0 \times 10^3} \times 100\% \quad (4)$$

$$\eta = \frac{c \times v \times 10^{-3}}{m \times 10^3} \times 100\% \quad (5)$$

$$R = \frac{c \times v \times 10^{-3}}{m \times \eta_0 \times 10^3} \times 100\% \quad (6)$$

where  $\eta_0$  is the content of indium in crushed glass (%),  $c_0$  is identified with the concentration of indium in its digestion solution (mg/L), determined by ICP-AES,  $\eta$  is the content of leached indium in crushed glass (%),  $c$  is the leaching concentration of indium (mg/L),  $m_0$  and  $m$  are the masses of the samples (g),  $R$  is the recycling rate of indium (%), and  $v$  is the volume of leaching solution (mL).

Generally, the acids such as HCl,  $\text{HNO}_3$ , and  $\text{H}_2\text{SO}_4$  are regarded as potential candidates of leaching agents.<sup>19,29</sup> Primary factors and levels were chosen for orthogonal design (Table 1). Each experiment could use 5 g of sample for leaching operation, and all the experiments were repeated three times.

In order to discover the leaching mechanism between crushed glass and acid, a single-factor experiment for the particle size of samples, leaching time, and initial concentration

Table 2. Experimental Results and Analysis for Orthogonal Design

no.	liquid–solid ratio (g/L)	retention time (min)	temperature (°C)	sample size (μm)	leaching agent	initial concentration (g/L)	oxidant	R (%)
1	1:1	60	25	<75	HCl	50	1 g MnO <sub>2</sub>	54.00
2	1:1	120	50	75–150	HNO <sub>3</sub>	100	2 g MnO <sub>2</sub>	62.77
3	1:1	180	100	>150	H <sub>2</sub> SO <sub>4</sub>	200	none	54.46
4	2:1	60	25	75–150	HNO <sub>3</sub>	200	none	28.77
5	2:1	120	50	>150	H <sub>2</sub> SO <sub>4</sub>	50	1 g MnO <sub>2</sub>	61.15
6	2:1	180	100	<75	HCl	100	2 g MnO <sub>2</sub>	86.46
7	3:1	60	50	<75	H <sub>2</sub> SO <sub>4</sub>	100	none	81.38
8	3:1	120	100	75–150	HCl	200	1 g MnO <sub>2</sub>	59.38
9	3:1	180	25	>150	HNO <sub>3</sub>	50	2 g MnO <sub>2</sub>	14.77
10	1:1	60	100	>150	HNO <sub>3</sub>	100	1 g MnO <sub>2</sub>	27.69
11	1:1	120	25	<75	H <sub>2</sub> SO <sub>4</sub>	200	2 g MnO <sub>2</sub>	74.38
12	1:1	180	50	75–150	HCl	50	none	89.85
13	2:1	60	50	>150	HCl	200	2 g MnO <sub>2</sub>	27.23
14	2:1	120	100	<75	HNO <sub>3</sub>	50	none	11.38
15	2:1	180	25	75–150	H <sub>2</sub> SO <sub>4</sub>	100	1 g MnO <sub>2</sub>	60.92
16	3:1	60	100	75–150	H <sub>2</sub> SO <sub>4</sub>	50	2 g MnO <sub>2</sub>	44.15
17	3:1	120	25	>150	HCl	100	none	40.31
18	3:1	180	50	<75	HNO <sub>3</sub>	200	1 g MnO <sub>2</sub>	92.62
level 1 <sup>a</sup>	60.54	43.85	45.54	66.69	59.54	45.85	59.31	
level 2 <sup>a</sup>	46.00	51.54	69.15	57.62	39.69	59.92	51.62	
level 3 <sup>a</sup>	55.46	66.54	47.23	37.62	62.77	56.15	51.00	
max deviation	14.54	22.69	23.62	29.08	23.08	14.08	8.31	

<sup>a</sup>Levels 1, 2, and 3 are same as those in Table 1.

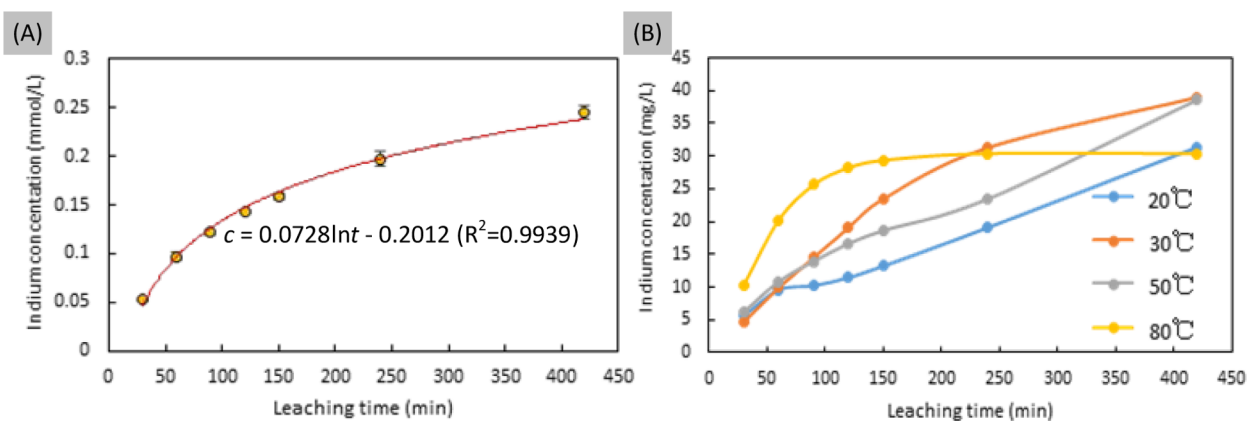


Figure 3. Change of indium concentration in solution with leaching time: (A) 25 °C; (B) multitemperature scenarios.

of the leaching agent was adopted to recognize the chemical kinetics. The relationship between leaching time and concentration in solution would be investigated. In this context, major measuring devices and those types in this work were also given in SI Table S3.

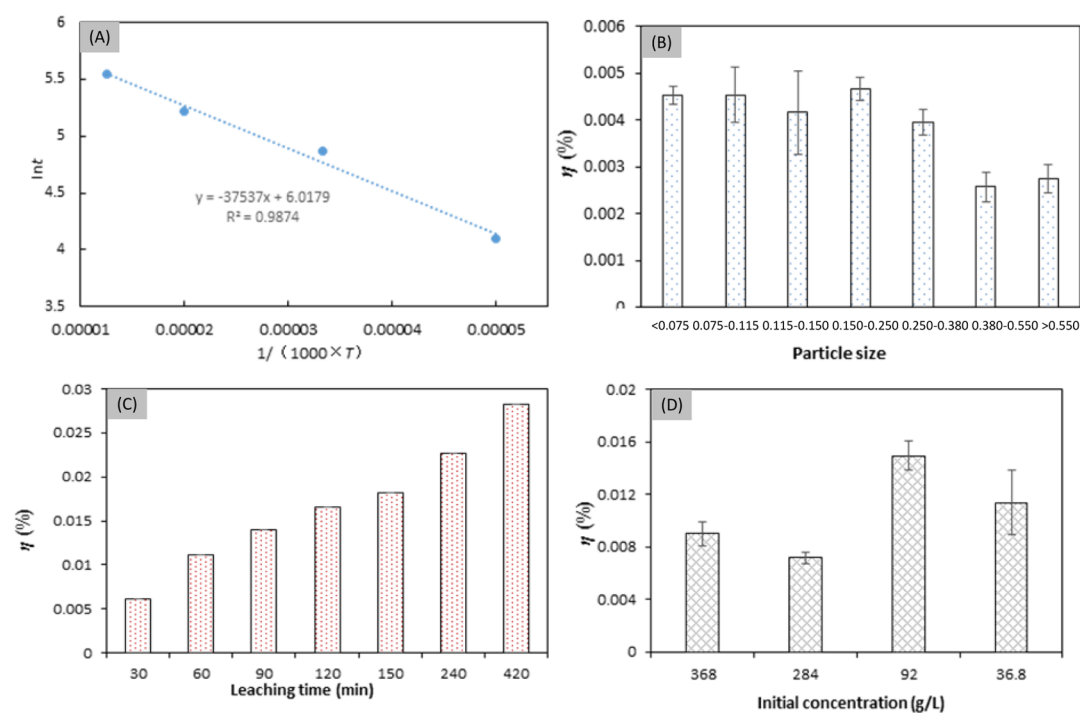
## RESULTS AND DISCUSSION

**Optimizing the Leaching Process for Indium.** Existing studies indicate that indium can convert into In<sup>3+</sup> or react with halogen elements to form precipitates in an acid solution. For instance, indium can react with HCl and produce InCl<sub>3</sub>.<sup>15</sup> To recover indium, a used ITO target or film can be first dissolved in an acid solution, and then the indium can be easily replaced by Zn, Al, or Mg for recovery. Here, 18 experiments were carried out to optimize the leaching process, illustrated in Table 2. The obtained results indicated that the optimal situation of the leaching process for good yields was a <75 μm sample size, 180 min retention time, 50 °C temperature, H<sub>2</sub>SO<sub>4</sub> as the

leaching agent, 100 g/L initial concentration, and 1:1 of liquid–solid ratio. Max deviation in Table 2 demonstrated that ranking order of the factors in the leaching process from big to small was particle size, temperature, leaching agent, retention time, liquid–solid ratio, initial concentration, and oxidant.

### Mechanism Exploring for Indium Leaching Process.

Temperature is commonly one of the important factors in a chemical reaction. We chose different temperatures including 20 °C, 25 °C, 30 °C, 50 °C, and 80 °C to uncover the mechanism of the indium leaching process. Despite any temperature, indium concentration in solution increased gradually with the growth of leaching time. Meanwhile, the rise in temperature would raise the indium concentration and save leaching time (Figure 3). In light of the relationship between leaching time and indium concentration (Figure 3A), a differential method in mathematics was adopted to tentatively estimate the reaction order. While  $k = 0.0728/t$  ( $k$  is instantaneous reaction rate, and  $t$  is leaching time), the reaction



**Figure 4.** (A) The relationship between reaction time and leaching temperature during the leaching process. (B) Effect of indium leaching from samples' particle size. (C) Effect of indium leaching under different leaching time. (D) Effect of indium under different initial concentration of H<sub>2</sub>SO<sub>4</sub>.

order was determined as 2.34 (see SI Text2). As a consequence, the reaction rate formula could be indicated as eq 7:

$$k = 1/(2.34 \times c^{2.34} \times t) \quad (7)$$

According to the data in Figure 3B, the leaching time to reach the same indium concentration could be easily determined under multitemperature scenarios. Here, the Arrhenius equation was utilized to examine the relationship between temperature and leaching time and thus achieve  $-3.75 \times 10^4$  and 6.02 for the slope and intercept of the straight line, respectively (Figure 4A). Then the activation energy and the pre-exponential factor of the leaching reaction were 312.1 kJ/mol and  $410.8 \times 10^9$  m<sup>3</sup>/(mol·min), respectively (see SI Text3), which obviously reveals that the leaching process could not occur in a regular situation of ambient temperature and atmospheric pressure.

In addition to temperature, other factors including the particle size of the samples, leaching time, and initial concentration of the leaching agent also can significantly affect the indium leaching process. Based on the obtained results of leaching process optimization, the agent H<sub>2</sub>SO<sub>4</sub> was chosen for further experiments. A single-factor experiment was utilized to distinguish their impact on the leaching process (Figure 4). No significant difference exists in various particle sizes owing to uniform distribution of ITO in scraped glass. The small size of particles generally led to a great possibility of indium leaching. The longer the leaching time, the bigger the concentration of indium in solution. When leaching time was 420 min, calculated indium content could reach 0.028%. In the case of the single-factor experiment with 180 min of leaching time, a 25 °C leaching temperature, H<sub>2</sub>SO<sub>4</sub> as the leaching agent, a little MnO<sub>2</sub>, and a 2:1 liquid–solid ratio, an interesting finding is that when the initial concentration of H<sub>2</sub>SO<sub>4</sub> was 92 g/L; the calculated indium content was obviously higher than those in

other initial concentrations. Too high and too low concentration of H<sub>2</sub>SO<sub>4</sub> could inhibit the leaching process.

**Analysis on the Physicochemical Changes of Prepared Samples after Leaching.** In order to further explore the leaching process of scraped glass, physicochemical changes were also determined in detail for raw material and leached residues. Under the optimal leaching process, the leached residues were collected, washed, and dried for further measurement. As shown in Table 3, almost all elements'

**Table 3. Comparison of Major Element's Content Determined XRF between Raw Material and Residue (Calculated as Chemical Product)**

element	raw material (%)	residue (%)	element	raw material (%)	residue (%)
SiO <sub>2</sub>	50.430	61.68	SO <sub>3</sub>	0.035	0.668
Al <sub>2</sub> O <sub>3</sub>	12.690	14.258	As <sub>2</sub> O <sub>3</sub>	0.953	0.336
BaO	22.131	7.349	NiO	0.059	0.176
SrO	5.291	6.27	ZnO	0.543	0.169
CaO	6.715	3.927	Na <sub>2</sub> O	0.291	0.072
Fe <sub>2</sub> O <sub>3</sub>	0.287	2.308	P <sub>2</sub> O <sub>5</sub>	0.070	0.064
MgO	0.140	1.79	K <sub>2</sub> O	0.021	0.015
Cr <sub>2</sub> O <sub>3</sub>	0.252	0.683	In <sub>2</sub> O <sub>3</sub>	0.057	none

content changed, and in particular, indium content had altered from 0.057% of raw material to none, which indicates that all indium in raw material could be substantially leached for recovery.

Same as the raw material, the leached residues were still in an amorphous state without being crystalline. However, lots of structure cells occurred in leaching residues, which lay at 25°, 36°, 42°, 45°, and 56° (Figure 5). SEM was also employed to find the disparity. As shown in Figure 6, there was no significant discrepancy between raw materials and leached residues: big-

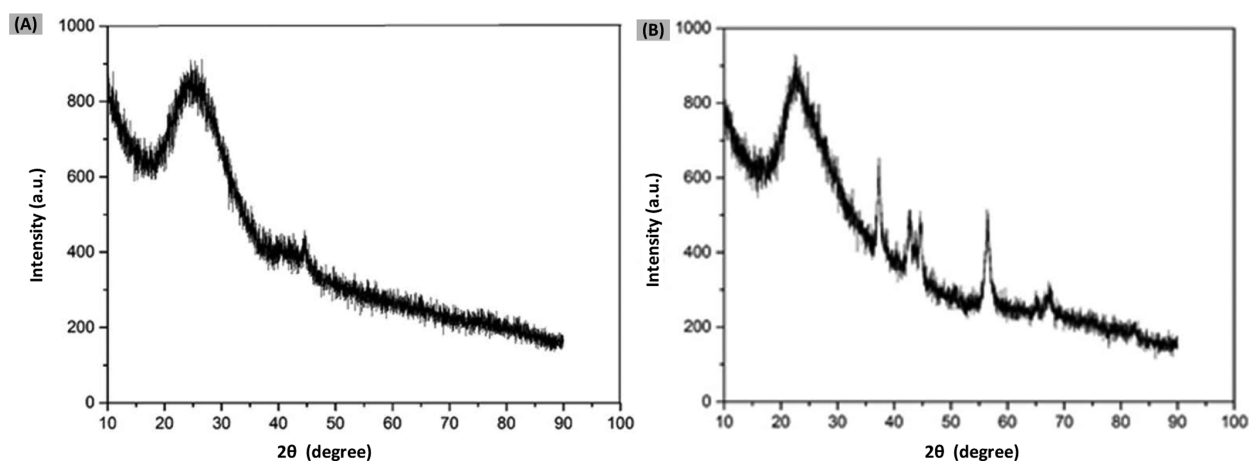


Figure 5. XRD analysis for leaching process: (A) raw material, (B) leached residues.

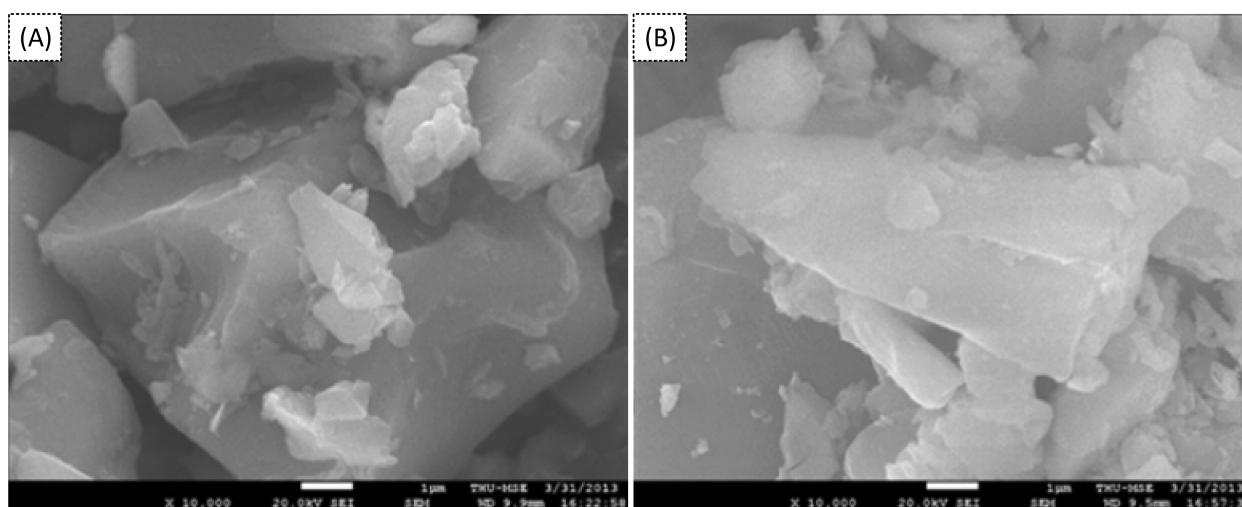


Figure 6. SEM images ( $\times 10\,000$ ) for leaching process: (A) raw material, (B) leached residues.

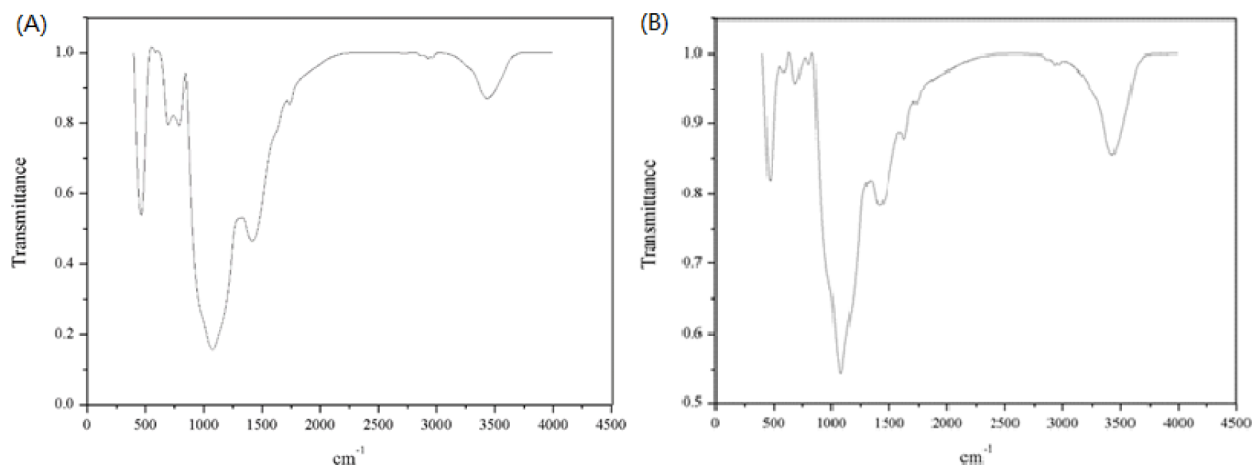


Figure 7. FTIR spectra for leaching process: (A) raw material, (B) leached residues.

and small-size particles were mixed together and varied in shapes. But fewer edges and corners appeared in the surface of particles. Additionally, the amount of peaks in FTIR spectra for leached residues increased (Figure 7), which could reveal that new functional groups occurred after the leaching process. The maximum peak still remained at  $1000\text{--}1200\text{ cm}^{-1}$  owing to the numerous existent C–N, C–O, or other functional groups.

More intensive peaks in Figure 7B arose at  $500\text{--}600\text{ cm}^{-1}$  and  $3400\text{--}3500\text{ cm}^{-1}$ , which could be explained with emerging C–Cl, C–I, O–H, or N–H in leached residues.<sup>30</sup> Figure 8 shows the TG-DSC curves of leached residues. Two distinct attenuations of mass appeared at  $220$  and  $410\text{ }^{\circ}\text{C}$ , demonstrating that a chemical reaction sensitive to the mass perhaps occurred during this temperature interval. All the

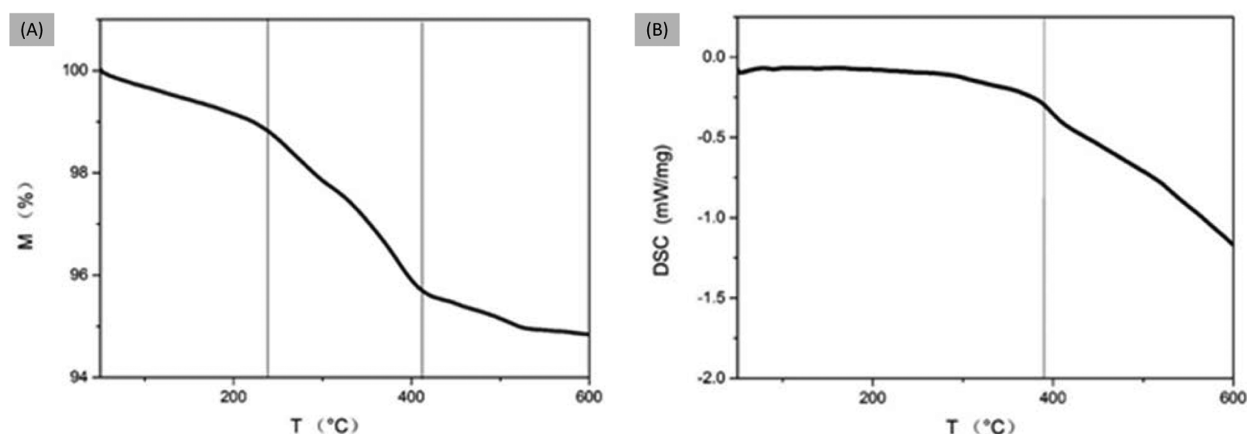


Figure 8. TG-DSC curves of leached residues: (A) TG, (B) DSC.

attained results further implied that the leaching process could modify raw material and enhance its activation for further recovery.

## CONCLUSION

The obtained results from theory and experiments show that scraped glass of LCDs can be leached using inorganic acid with an oxidant of  $\text{MnO}_2$ . The optimal parameters for the leaching process are controlled at a  $<75 \mu\text{m}$  sample size, 180 min retention time,  $50^\circ\text{C}$  temperature,  $\text{H}_2\text{SO}_4$  as the leaching agent, 100 g/L initial concentration, and a 1:1 liquid–solid ratio. The order, the activation energy, and the pre-exponential factor of the leaching reaction were 2.34, 312.1 kJ/mol, and  $410.8 \times 10^9 \text{ m}^3/(\text{mol}\cdot\text{min})$ , respectively, which reveals that the leaching process could not occur in a regular situation. Too high and too low of a concentration of  $\text{H}_2\text{SO}_4$  could not afford good yields in the leaching process. We also found that the leaching process could largely modify raw material and enhance its activation for further recovery. In prospect, the developed technique in this study contributes to a closed-loop process of indium recovery and can be fully integrated into e-waste recycling for metal recovery and pollution controlling.

## ASSOCIATED CONTENT

### Supporting Information

Figure S1–S3, Table S1–S3, and Text1–3. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acssuschemeng.5b00020.

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### Author Contributions

X.Z. wrote the manuscript, F.W. completed all the experiments and supplied the data, X.S. contributed her findings, and J.L. supervised the study. The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

### Notes

The authors declare no competing financial interest.

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