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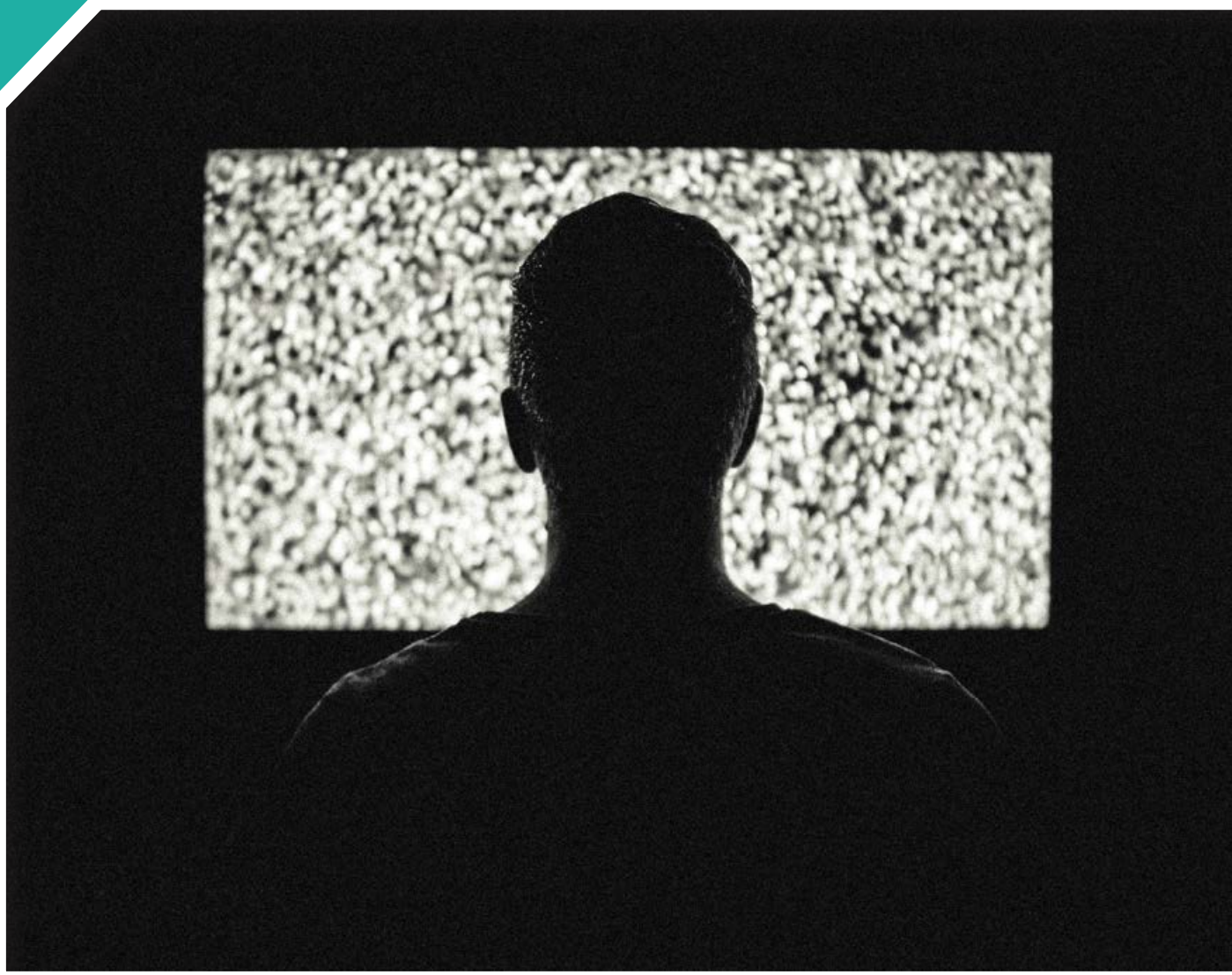
Investigation of Indium Recovery from End-of-life LCDs

Authors: Chinnam Rama Krishna, John Mulcahy and Lisa O'Donoghue



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- Office of Communications and Corporate Services

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Investigation of Indium Recovery from End-of-life LCDs

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EPA Research Report

Prepared for the Environmental Protection Agency

by

University of Limerick

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Cover image: Silhouette of man in front of TV (https://www.pexels.com/photo/night-television-tv-video-8158/).

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Executive Summary

The LCDVal project aimed to illustrate the potential indium value contained in liquid crystal displays (LCDs) accessible via recovery techniques, therefore turning liquid crystal panel waste fractions into a resource for critical raw materials. The objectives and targets were to:

- unlock the volume of indium from waste LCDs and increase the range and yields of recovered raw materials;
- illustrate the automation potential of the recovery process;
- push Ireland to the forefront in the area of raw material-processing technologies;
- increase the economic viability and investment security of the processing operations.

Liquid crystal displays contain a liquid crystal glass panel component. This liquid crystal glass panel is composed of two glass sheets with liquid crystals placed between the sheets. Indium is present in the form of a coating on the internal surfaces of the glass panels of the LCD and is combined in an alloy of indium tin oxide (ITO). In order to achieve the objectives, the potential for indium recovery from LCDs was investigated by exploring pre-treatments (to remove ITO) and various hydrometallurgical techniques (to recover indium from the separated ITO). The pre-treatments considered for investigation were bulk grinding of the LCD glass panel with indium coating, surface grinding to remove ITO from the LCD glass panel and laser pre-treatment to evaporate the ITO coating.

The output materials from the pre-treatments – indium-containing powders in the case of bulk and surface grinding techniques and indium on an absorption media in the case of the laser technique – were subjected to metallurgical testing to identify the parameters that would cost-effectively yield recovered indium of good quality.

Analysis of the initial indium concentrations on the LCD panels showed large variations, from 38 to 800 mg/kg of glass. The glass front generally contained more indium then the glass back. The best-performing pre-treatments were considered to be the surface grinding technique and the laser ablation processes, as both removed ITO directly with minimal contaminants being collected. The surface grinding process yielded a powder that predominantly consisted of ITO; this has the advantage of introducing fewer contaminants to the acid bath than the bulk grinding technique during the hydrometallurgical step.

The laser technique has the potential to be a very high throughput process; however, the ITO has to be captured on an absorption media, which subsequently must be submerged into the acid bath and may act as a contaminant. However, the potential to deposit or capture the ITO on bath-friendly media readily resolves this and offers a high degree of flexibility with regard to processing options.

The potential of automating both the laser and the surface grinding techniques was considered in detail. Similar process set-ups would be required by both. The advantage in automating these processes is that the polariser film of the panel does not require removal in order to access the ITO. This has a significant impact on the time and quality of these processes compared with the other options available (e.g. drect acid digestion and bulk grinding). The laser technique has the potential to achieve very high throughputs, with a minimum of approximately 240 individual panels of 120 panel pairs per hour. Up to 96 g per hour of indium is collectable with one laser (assuming 800 mg per device); using multiple lasers the process can be scaled up for fully automated closed-loop indium recycling from LCDs. This study shows that the laser processing technique has significant potential for use in large-scale indium recovery.

1 Introduction

1.1 Project Statement

The LCD Val project aimed to demonstrate the indium content in liquid crystal displays (LCDs), accessible using recovery techniques, therefore turning waste LCD waste fractions into a resource for critical raw materials (CRMs).

1.2 Objectives and Targets

The objectives and target of this study were to:

- unlock the volume of indium from waste LCDs and increase the range and yields of recovered raw materials;
- illustrate the automation potential of the recovery process;
- push Ireland to the forefront in the area of raw material-processing technologies;
- increase the economic viability and investment security of the processing operations.

Liquid crystal glass panels are composed of two glass sheets with liquid crystal placed between the sheets. Indium is present in the form of a coating on the internal surfaces of the glass panels of an LCD and is combined in an alloy of indium tin oxide (ITO).

In order to achieve the objectives, the potential for indium recovery from LCDs was investigated by exploring pre-treatments (to remove ITO) and various hydrometallurgical techniques (to recover indium from the separated ITO). Figure 1.1 summarises the pre-treatments considered for investigation: bulk grinding of the LCD glass panel with the ITO coating, surface grinding to remove the ITO from the LCD glass panel and laser pre-treatment to evaporate the ITO coating.

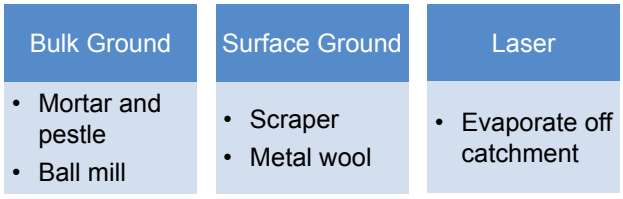


Figure 1.1. Pre-treatment options for the removal of ITO from the LCD glass panel.

The output materials from the pre-treatments – ITO-containing powders in the case of the bulk and surface grinding techniques and ITO on an absorption media in the case of the laser technique – were subjected to metallurgical testing to identify the parameters that would cost-effectively yield recovered indium of good quality (Figure 1.2).

In the study, the glass panels were separated from each LCD and washed clean of liquid crystals before the pre-treatments and hydrometallurgical testing. Chapter 2 details the experimental set-up and procedures carried out.

1.3 Relevance to Policy

Indium is one of the strategically important materials that have been characterised as critical by several industrialised countries (Hatayama and Tahara, 2014; Graedel and Reck, 2016). In 2010, indium was classified as a CRM by the European Commission (EC, 2010). The concentration of indium in the Earth's crust is one-sixth that of gold (He *et al*., 2014). Indium has no ores of its own; it is primarily produced as a by-product of zinc and lead production in a concentration of between 10 and 20 mg/kg (Takahashi *et al*., 2007). Up until 2025, the yearly demand for this material is estimated to grow by 70% relative to 2015 (Licht *et al*., 2015). In 2011, 1220 t indium was primarily mined worldwide, from which 660 t indium was refined (Licht *et al*., 2015). In 2013 and 2014, the output of refined indium increased to 800 t and 820 t, respectively (US Geological Survey, 2015). Over

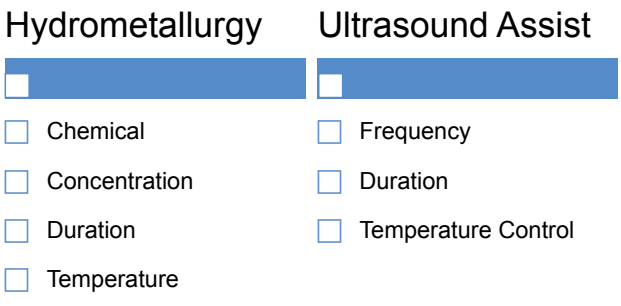


Figure 1.2. The parameters investigated during hydrometallurgical testing in order to establish an optimised process for indium recovery.

55% of the worldwide production of indium is used in the ITO target industry, which is a major component in the production of LCDs (Li *et al*., 2011; Pradhan *et al*., 2018). Hence, indium recovery from LCDs is strategically important.

Liquid crystal displays are used in applications ranging from small machine displays up to 100-inch television screens. In Ireland in 2015, 8321 t of televisions and 148 t of monitors containing LCD screens were collected (EPA, 2015). It is estimated that 25 million m^2 of LCDs have entered the European waste stream (Kopacek, 2009). Given that many LCDs have a short lifespan, a large number of LCDs are made redundant each year and require proper disposal (70,000 t in 2013). The Waste Electrical and Electronic Equipment (WEEE) Directive (EU, 2012) and Restriction of Hazardous Substances (ROHS) Directive (EU, 2011), which all EU Member States have to implement, stipulate that components containing mercury and liquid crystals must be removed from LCDs and avoided in production when possible. A challenge with LCD televisions is the separation of the LCD glass panel and the cold cathode fluorescent lamp (CCFL) tubes (containing mercury). This challenge was undertaken by the University of Limerick in a previous Environmental Protection Agency (EPA)-funded project, which led to the formation of a spin-out company Votechnik and a European pilot action project called ReVolv (http://revolvproject.eu/), which developed and launched a commercial-level technology for LCD depollution involving removal of the LCD glass panel and CCFLs in an automated process. This project now focuses on researching efficient options to recover the indium from LCD glass panels.

2 Hydrometallurgy Experimental Methodology

2.1 "As-is" Samples: Hydrometallurgy Methodology

An LCD glass panel in an as-is state underwent standard hydrometallurgy to remove ITO directly by acid digestion (Figure 2.1). These samples were used as control samples in order to evaluate the effectiveness of the pre-treatments and alterations to the hydrometallurgical process. The polariser film that is present on the external surfaces of the glass panels of the LCD was removed manually and the surface of the glass was cleaned using acetone. The liquid crystals were also cleaned before testing as-is samples. It was important to remove the polariser from the glass surface to minimise its effects, if any, on the subsequent dissolution of ITO in acids during the hydrometallurgical step. It is important to note that in LCD glass panels the polariser is on the outer surface of the glass and the ITO is on the internal surface; thus, the polariser removal process has no direct influence on the ITO. The individual glass sheets were labelled glass front (GF) and glass back (GB), representing their original location within the LCD device, with the GF being the viewing side and the GB being the rear side. The GF and GB were cut into samples of 2 cm × 2 cm using a carbide tip pen (see Figure 2.1) and were preserved in airtight packaging until used for experiments.

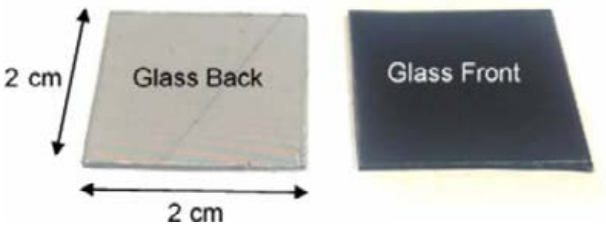


Figure 2.1. Photograph of the GF and the GB.

The as-is samples were tested in aqua regia (AR; ratio of HCl : HNO_3 = 4 : 1) for indium dissolution from ITO. The time of dissolution was varied or dissolution was carried out under the influence of ultrasound (US) waves.

2.1.1 Hydrometallurgy: shaker digestion for as-is samples

Leaching experiments were performed by acid dissolution of GF and GB samples using different techniques. GF and GB samples were placed in separate jars containing acids of different concentrations, as indicated in Table 2.1. These jars were placed into a pre-heated incubator at 70°C with shaking at 200 rpm for the different timescales indicated in Table 2.1 and were then taken out of the incubator to cool to room temperature. The solution in each sample was gathered into a centrifuge tube and stored for further analysis using inductively coupled plasma optical emission spectrometry (ICP-OES). In ICP-OES, the indium concentration in the acid is measured and extrapolated as a measurement of the indium (mg) per kg of sample.

2.1.2 Hydrometallurgy: US-assisted digestion for as-is samples

As-is samples were placed in different beakers containing the acid concentrations indicated in Table 2.2. These beakers were placed directly into an ultrasonic sonicator at room temperature (Soniprep 150, 28 KHz) and the ultrasonic probe (titanium alloy) was dipped into the acid solution. Ultrasonic waves were applied at 15 µm amplitude and 28 KHz for the timescales indicated in Table 2.2. During the experiment the temperature of the acid solution was monitored. For measurements of greater than

Table 2.1. Parameters used for shaker dissolution of ITO

Sample	AR concentration (%)	Sample size (cm)	Volume of acid (ml)	Temperature (°C)	Mixing speed (rpm)	Time (hours)
GF	1, 2, 2.5, 5, 10 and 15	2 × 2	20	70	200	0.5, 2, 6, 12, 24, 48
GB	1, 2, 2.5, 5, 10 and 15	2 × 2	20	70	200	0.5, 2, 6, 12, 24, 48

Table 2.2. Parameters used for ultrasonic dissolution of ITO

Sample	AR concentration (%)	Sample size (cm)	Volume of acid (ml)	Frequency (KHz)	Increase in temperature of AR with US	Time (minutes)
GF	1, 2, 2.5, 5, 10	2×2	20	28	Yes	0.5, 1, 3, 5, 10, 15
GF	1, 2, 2.5, 5, 10	2×2	20	28	Yes	0.5, 1, 3, 5, 10, 15

See Figure 3.8 for temperature profiles of AR with US.

5 minutes, paper tissue was wrapped around the beaker opening to reduce acid evaporation. At the end of the sonication process, the solution and glass samples were collected for further analysis using ICP-OES, scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX).

The temperature of the different AR concentrations was measured at regular intervals of ultrasonic application time. All concentrations of acids showed similar temperature trends in relation to ultrasonic application time. At 15 minutes of sonication solutions reached a maximum temperature of approximately 90°C because of cavitation.

2.1.3 Sample code explanation

The samples tested were given specific IDs, which included the type of test carried out (as-is samples), the LCD sample used (M1, M2, M3, M4 or M5), which side of the sample was investigated (GF or GB), the type of dissolution carried out [leaching (Le) or US], the acid used and concentration (AR; 1%, 2%, 2.5%, 5%, 10% or 15%) and how long the dissolution process was carried out for (time in minutes). The following provides an example of the IDs used:

- Asis_M2GF_Le_1%AR_30min [Asis (method)_ M2GF (LCD sample and side of sample)_Le (type of dissolution used)_1%AR (concentration and type of acid)_30 min (time of dissolution)].

2.2 Bulk Grinding Pre-treatment: Hydrometallurgical Methodology

A mortar and pestle technique was used to bulk grind the GF and GB control samples (Figure 2.2). Before bulk grinding was performed, the polariser film was removed and liquid crystals were cleaned. The GF and GB samples were crushed for approximately 5 minutes, which resulted in powders containing fine and coarse particles. The obtained powders were filtered using different particle size sieves (53 µm, 125 µm). This filtration process derived three particle sizes – < 53 µm, < 125 µm and < 250 µm – which were used for further analysis.

The three different particles size samples, as well as a sample that was not filtered, were analysed under SEM to measure the average particle size and to perform EDX mapping. ImageJ software was used for the particle size analysis. The powders obtained from filtering were dissolved in AR in a shaker or under the influence of US waves.

2.2.1 Hydrometallurgy: shaker digestion for bulk ground samples

Please refer to section 2.1.1 for methodology. Table 2.3 details the parameters used for the hydrometallurgical process for the bulk ground samples.

Figure 2.2. Mortar and pestle used to crush the GB and GF samples.

Table 2.3. Parameters used for the hydrometallurgical process for the bulk ground samples

Sample	AR concentration (%)	Particle size (µm)	Volume of acid (ml)	Temperature (°C)	Mixing speed (rpm)	Time (hours)
GF	1, 2, 2.5, 5, 10 and 15	<53, <125, <250 and pre-filtered sample	20	70	200	0.5, 2, 6, 12, 24, 48
GB	1, 2, 2.5, 5, 10 and 15	<53, <125, <250 and pre-filtered sample	20	70	200	0.5, 2, 6, 12, 24, 48

Table 2.4. Parameters used for the US-assisted hydrometallurgical process for the bulk ground samples

Sample	AR concentration (%)	Particle size (µm)	Volume of acid (ml)	Frequency (KHz)	Increase in temperature of AR with US	Time (minutes)
GF	1, 2, 2.5, 5, 10	<53, <125, <250 and pre-filtered sample	20	28	Yes	0.5, 1, 3, 5, 10, 15
GB	1, 2, 2.5, 5, 10	<53, <125, <250 and pre-filtered sample	20	28	Yes	0.5, 1, 3, 5, 10, 15

2.2.2 Hydrometallurgy: US-assisted digestion for bulk ground samples

Please refer to section 2.1.2 for methodology. Table 2.4 details the parameters used for the US-assisted hydrometallurgical process for the bulk ground samples.

2.2.3 Sample code explanation

The bulk ground samples tested were given specific IDs, which included the type of test carried out (bulk grinding), the particle size, the LCD sample used (M1, M2, M3, M4 or M5), which side of the sample was used (GF or GB), the type of dissolution carried out (Le or US), the acid used and concentration (AR; 1%, 2%, 2.5%, 5%, 10% or 15%) and how long the dissolution was carried out for (minutes). The following provides an example of the IDs used:

- Bulk_53um_M3GF_Le_1%AR_30min [bulk (method)_53um (particle size)_M3GF (LCD sample and side of sample)_Le (type of dissolution used)_1%AR (concentration and acid)_30min (time of dissolution)].

2.3 Surface Grinding Pre-treatment: Hydrometallurgical Methodology

The surface grinding technique involved removal of the ITO and dye on the GF or removal of the ITO alone on the GB by abrasion. During surface grinding the polariser was not removed and the liquid crystals were removed by washing. The aim of this technique was to remove the ITO and dye without influencing or chipping the glass surface. Different abrasion techniques were tested, with the extraction time, yield and efficiency measured. The techniques that resulted in the highest yields were selected for further analysis. The surface grinding samples were tested in AR for indium dissolution from ITO. These experiments were conducted by varying the time of dissolution of the samples in a shaker or carrying out dissolution under the influence of US.

2.3.1 Hydrometallurgy: shaker digestion for surface ground samples

Please refer to section 2.1.1 for methodology. Table 2.5 details the parameters used for the hydrometallurgical process for the surface ground samples.

2.3.2 Hydrometallurgy: US-assisted digestion for surface ground samples

Please refer to section 2.1.2 for methodology. Table 2.6 details the parameters used for the US-assisted hydrometallurgical process for the surface grinding samples.

Table 2.5. Parameters used for the hydrometallurgical process for the surface ground samples

Sample	AR concentration (%)	Sample size	Dissolution time (hours)	Volume of acid (ml)	Temperature (°C)	Mixing speed (rpm)
GF	1, 2, 2.5, 5, 10 and 15	2 cm × 2 cm LCD glass panel used to produce powder	0.5, 2, 6, 12, 24, 48	20	70	200

Table 2.6. Parameters used for the US-assisted hydrometallurgical process for the surface ground samples

Sample	AR concentration (%)	Sample size	Dissolution time (minutes)	Volume of acid (ml)	Frequency (KHz)	Increase in temperature of AR with US
GF	1, 2, 2.5, 5, 10	2 cm × 2 cm LCD glass panel used to produce powder	0.5, 1, 3, 5, 10, 15	20	28	Yes

2.3.3 Sample code explanation

The surface ground samples tested were given specific IDs, which included the kind of test carried out (surface grinding), the surface grinding technique used [Metal wool (MW) or scraper], the LCD sample used (M1, M2, M3, M4 or M5), which side of the sample was used (GF or GB), the type of dissolution carried out (Le or US), the acid and concentration used (AR; 1%, 2%, 2.5%, 5%, 10% or 15%) and how long the dissolution was carried out for (minutes). The following provides an example of the IDs used:

- Surface_MW_M1GF_Le_1%AR_30min [Surface (method)_MW (surface grinding technique)_M1GF (LCD sample and side of sample)_Le (type of dissolution used)_1%AR (concentration and acid)_30 min (time of dissolution)].

2.4 Laser Process Pre-treatment: Hydrometallurgical Methodology

The GFs and GBs were cut into 10 cm × 10 cm samples using a silicon carbide tip pen. These samples were subjected to laser pre-treatment for ITO removal using different configurations (Figure 2.3).

Two external laboratories were used to assist with the laser ablation trials. A laser power of 50 W was used with a scan mode from a fixed distance of approximately 50 cm. Tests were conducted to assess the ability of the laser to remove ITO from the GF and GB samples. The residue generated from the GF and GB samples after laser ablation was captured using different media such as cotton wool, filter paper, glass, tissue paper and water. The direction of laser ablation of the GF and GB samples is indirect, as explained in Figure 2.4. In these experiments the polariser was not removed and the experiments were conducted with and without liquid crystals present. The ITO was dissolved in AR.

2.4.1 Hydrometallurgy: shaker digestion for laser-processed samples

Please refer to section 2.1.1 for methodology. Table 2.7 details the parameters used for the hydrometallurgical process for the laser-processed samples.

2.4.2 Sample code explanation

The laser-ablated samples were given specific IDs, which included the side of the sample (GF or GB), whether or not liquid crystals were present (with/without_LC), the sample used (LED) and the capturing medium used (cotton wool, filter paper or glass). The following provides an example of the IDs used:

- GF_with_LC_LED_wool [GF (side of the sample)_with_LC (with or without liquid crystals)_LED (sample used)_wool (the capturing medium)].

2.5 Characterisation Procedure and Parameters

Characterisation of the GF and GB samples was performed using SEM, EDX and ICP-OES techniques. All of the samples that were analysed

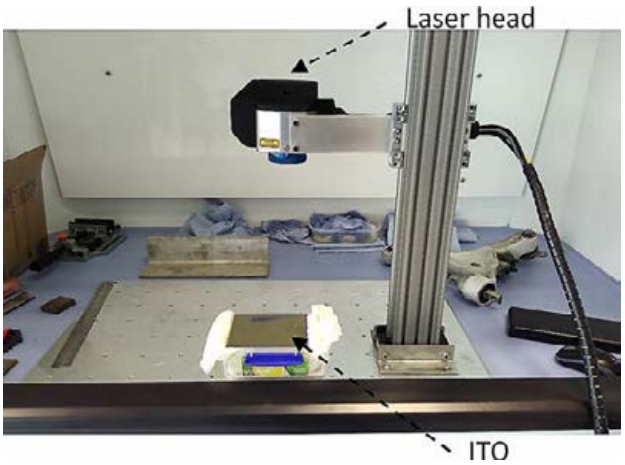


Figure 2.3. Laser set-up used for removal of ITO from GF and GB samples.

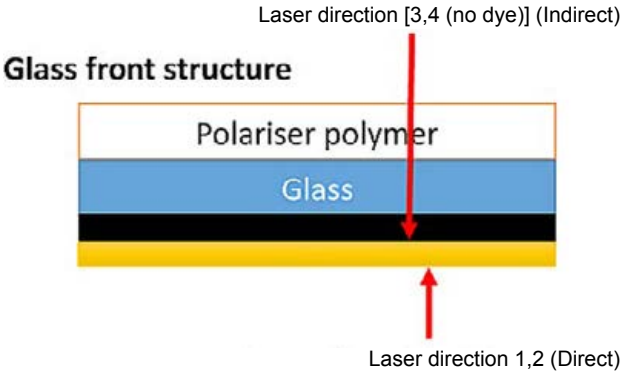


Figure 2.4. Schematic of laser ablation of ITO.

Table 2.7. Parameters used for the hydrometallurgical process for the laser-processed samples

Sample	AR concentration (%)	Medium	Volume of acid (ml)	Temperature (°C)	Mixing speed (rpm)	Time (hours)
GF	10	Cotton wool, filter paper and glass	35 and 50 (cotton)	70	200	24
GB	10	Cotton wool, filter paper and glass	35 and 50 (cotton)	70	200	24

with SEM were gold coated for 30 seconds to achieve electrical conductivity. SEM images of the microstructure were captured using secondary electrons. The same areas were subjected to elemental mapping using the EDX technique. The power used for SEM and EDX varied depending on the type of sample being analysed. Attempting to capture certain features and carry out elemental mapping at 20 kV resulted in bright spots and image drifting, also known as charging, which did not allow image capturing and mapping, whereas other samples did not charge at 20 kV. Therefore, for the samples that were charging the voltage was reduced to 10 KV.

An Agilent Technologies 5100 inductively coupled plasma optical emission spectrometer was used for metal analysis. Samples were prepared in a 1 M HNO_3 solution. This 1 M HNO_3 solution was also used for the dilutions of the standard solutions and as a calibration blank. The calibration curve was constructed by fitting through the origin using standard solutions of 10, 20, 40, 60 and 80 g/l of indium standard. The following analytical line (in nm) was used for calculations: In 325.609.

3 Hydrometallurgical Results

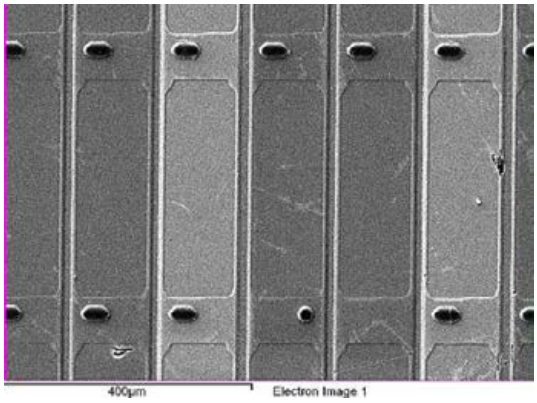
3.1 Characterisation of the Liquid Crystal Panels

All of the samples of LCD glass panels were used as is and were prepared for characterisation. The samples were analysed using SEM secondary electrons. Figure 3.1a shows a typical sample micrograph (C2-M1-S1-GF sample). The sample was mounted in resin to analyse the cross-section of the screen, as shown in Figure 3.1b. From the cross-section it was possible to measure the thickness of the colour dye and ITO as ~2.3 µm. The same kind of measurements were performed on the GB sample (C2-M1-S1-GB; Figures 3.1c and d). On the GB cross-section, no measurable coating was observed.

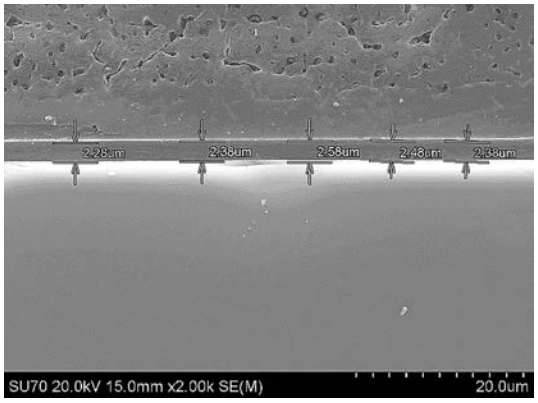
The GF sample was characterised using EDX point analysis, as shown in Figure 3.2. The first three point scans were carried out on the region where indium can be expected, i.e. on the colour dye, and the remaining three point scans were carried out on the glass surface. The point scans revealed the presence of indium in the colour dye region and no indium on the glass surface (Table 3.1).

The GB sample was similarly characterised using EDX point analysis, as shown in Figure 3.3. The point scans again revealed the presence of indium in the surface region, with no indium present in the glass region (Table 3.2).

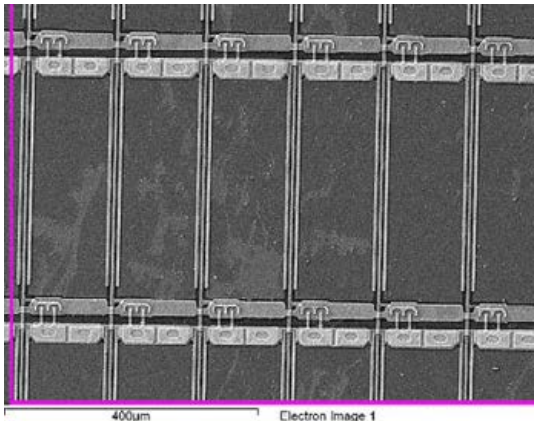
a. GF Surface: C2-M1-S1-GF SEM



b. GF Cross-Section: C2-M1-S1-GF-XS SEM



c. GB Surface: C2-M1-S1-GB SEM



d. GB Cross-Section: C2-M1-S1-GB-XS SEM

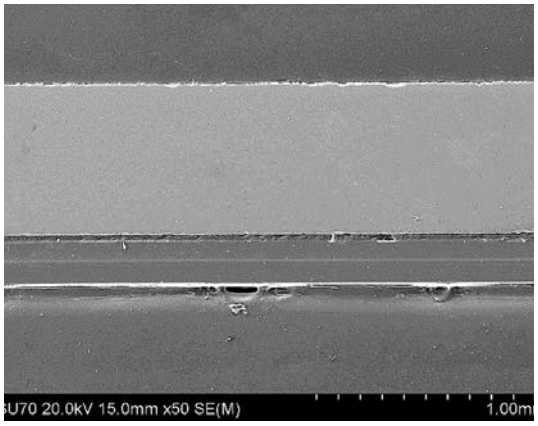


Figure 3.1. SEM images of the control samples: (a) GF surface; (b) GF cross-section; (c) GB surface; and (d) GB cross-section.

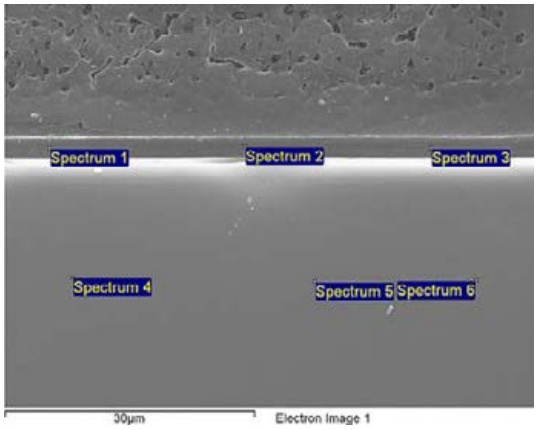


Figure 3.2. C2-M1-S1-GF microstructure used to perform the EDX point analysis. Spectra 1–6, shown on the microstructure, are the points used for analysis.

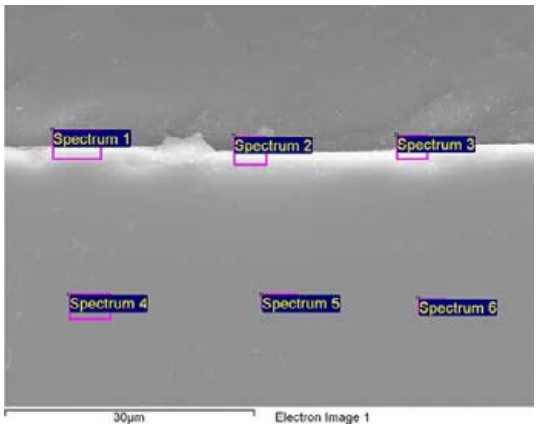


Figure 3.3. C2-M1-S1-GB microstructure used to perform the EDX point analysis. Spectra 1–6, shown on the microstructure, are the points used for analysis.

Table 3.1. EDX point analysis data for Figure 3.2, showing the elemental composition for various elements (wt%)

Spectrum	Magnesium	Aluminium	Silicon	Chlorine	Calcium	Copper	Indium	Tin
1	0.2	2	6.98	0.4	1.2	0.9	5.3	0.6
2		2.1	7.63	0.4	1.3	1	6.3	0.5
3	0.2	1.9	6.67	0.2	1.1	0.3	3.6	0.4
4	0.8	8.9	30.6		5.3			
5	0.7	8.8	30.5		5.2			
6	0.7	8.8	30.4		5.2			

Table 3.2. EDX point analysis data for Figure 3.3, showing the elemental composition for various elements (wt%)

Spectrum	Magnesium	Aluminium	Silicon	Sulfur	Chlorine	Calcium	Indium	Tin
1	0.12	1.38	8.58	0.38	0.15	0.63	1.27	0.15
2	0.23	2.43	9.34	0.38	0.17	0.94	0.90	0.10
3	0.11	1.32	7.56	0.60	0.19	0.53	1.51	0.19
4	0.77	8.64	28.81			4.80		
5	0.83	8.53	28.84			4.83		
6	0.75	8.53	29.00			4.86		

The GF sample was also characterised using EDX mapping to evaluate the ITO coating uniformity on the colour dye (Figure 3.4). The EDX analysis detected the presence of aluminium, silicon, chlorine, calcium, nickel, copper, bromine and indium. As the ITO layer is made of indium and tin, the other elements are understood to come from the colour dye or from the glass substrate. The rectangular boxes of ~150 µm are colour pixels and the vertical and horizontal lines are electrodes (see Figure 3.4a and c). The indium layer was found to be uniformly coated on the surface for the GF sample; this was also the case for the rest of the GF samples: C2-M2-S1-GF, C2-M3-S1-GF, C2-M4-S1-GF and C2-M5-S1-GF.

The GB of the sample was characterised using EDX mapping to test the ITO coating uniformity on the glass layer (Figure 3.5). The EDX analysis detected the presence of aluminium, silicon, calcium, molybdenum, indium and magnesium. Indium was found to only

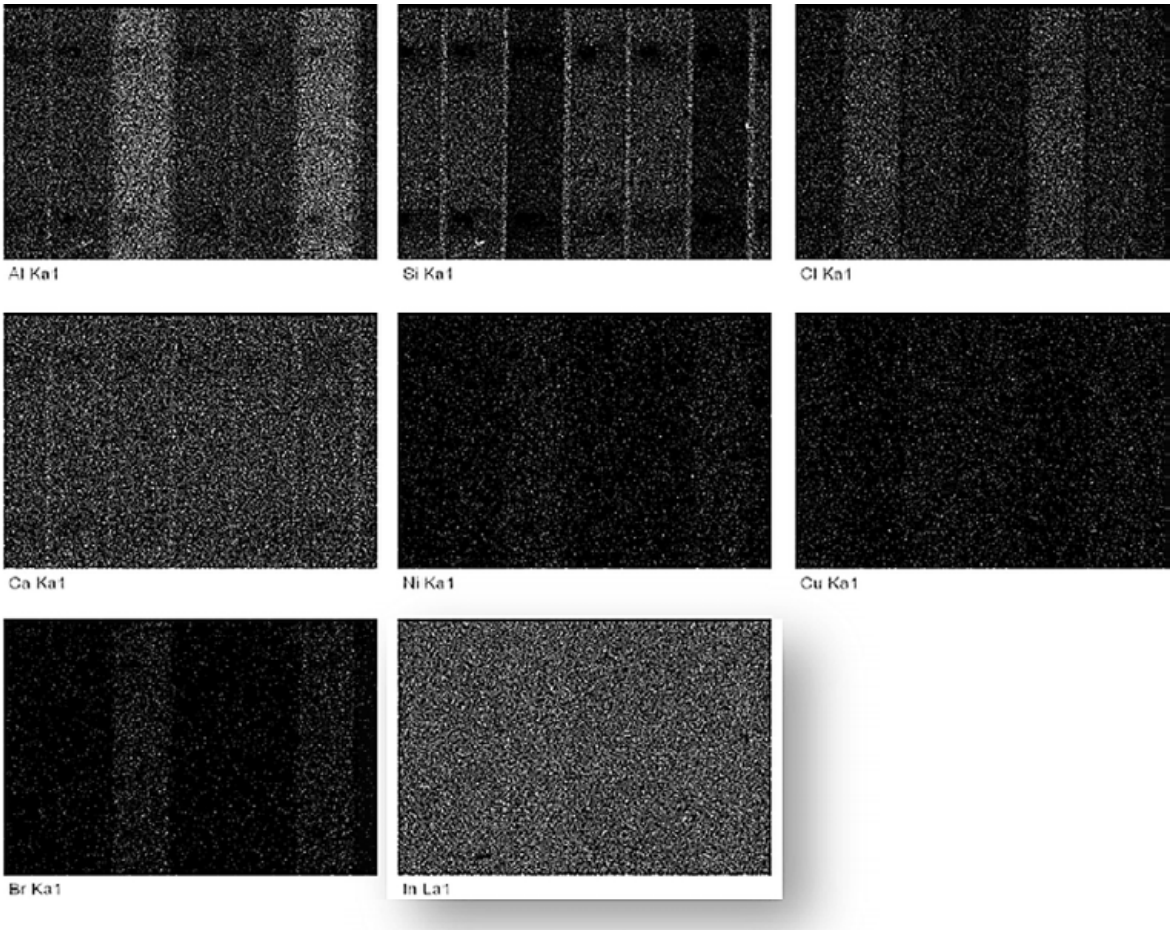


Figure 3.4. EDX area mapping of the C2-M1-S1-GF SEM image. The EDX mapping confirms the presence of indium, along with other elements such as Aluminium, silicon, chlorine, calcium, nickel, copper and bromine. Normalisation was not carried out on these data to extract the wt% of indium.

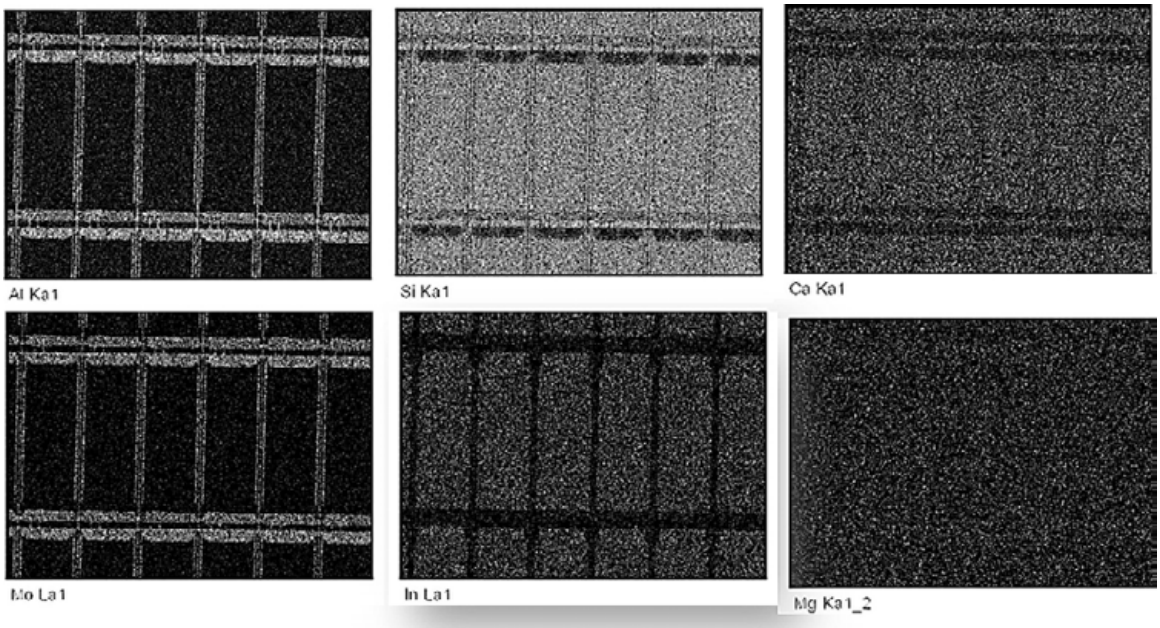


Figure 3.5. EDX area mapping of the C2-M1-S1-GB SEM image. The EDX mapping confirms the presence of indium, along with other elements such as magnesium, aluminium, silicon, calcium and molybdenum. Normalisation was not carried out on these data to extract the wt% of indium.

uniformly coat the surface of the rectangular boxes, with it being absent from the horizontal and vertical electrodes. Similar results were found for the rest of the GB samples: C2-M2-S1-GB, C2-M3-S1-GB, C2-M4-S1-GB and C2-M5-S1-GB.

The XPS measurements were also conducted on all of the samples and the indium content was measured per square meter of GF and GB. The average indium content of the GF is considerably higher than that of the GB. The GF has an average indium content of ~0.82 g/m^2 whereas the GB has an average indium content of ~0.25 g/m^2 (Figure 3.6). An average 32-inch LCD display has a viewing area of approximately 0.283 m^2; therefore, the equivalent indium content per display is approximately 232 mg according to these samples.

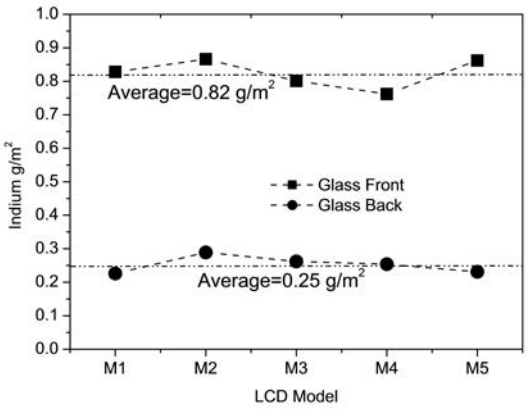


Figure 3.6. Indium content per square meter of GF and GB.

3.2 Hydrometallurgy: As-is Samples

The control samples C2-M2-GF and C2-M2-GB were tested for indium recovery by dissolving them as is in AR. A range of time intervals (30 minutes and 2, 6, 12, 24 and 48 hours) and AR concentrations (1%, 2%, 2.5%, 5%, 10% and 15%) was used. The dissolution of indium in AR was performed using two techniques: with a shaker and with US waves. The shaker technique was carried out at 70°C, with mixing at 200 rpm; the US technique included cavitation and a resulting gradual increase in acid temperature to 90°C. Figure 3.7 shows the effect of shaker dissolution of indium in AR of different concentrations over a range of time intervals for the GF sample. As the indium coating was found to be uniform on the glass surface, it is possible to extrapolate the ICP-OES values to calculate the number of mg of indium per kg of GF or m^2 of GF.

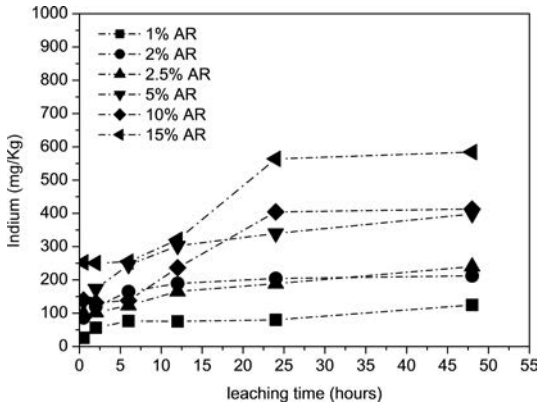


Figure 3.7. C2-M2-GF glass leached in 1–15% AR for different time periods from 30 minutes to 48 hours in a shaker at 70°C and 200 rpm agitation was analysed using ICP-OES. The data show the influence of time and AR concentration on indium leaching.

It can be observed in Figure 3.7 that 15% AR dissolved the highest amount of indium, reaching a plateau at 24 hours and continuing to 48 hours; 10% and 5% AR showed a similar trend to 15% AR but with a lower dissolution rate and the remaining AR concentrations showed lower dissolution rates even after 48 hours of leaching.

The GF as-is samples were also tested for indium leaching in AR under the influence of US waves. During this analysis the temperature profiles of AR at the different concentrations used were recorded with US waves up to 15 minutes. It was found that the acid temperature increased rapidly and after 15 minutes of sonication reached a maximum temperature of 90°C (Figure 3.8). No change in temperature profile with acid alone (without US) was observed.

The following AR concentrations and time intervals were used for indium dissolution under the influence of US: 1%, 2%, 2.5%, 5% and 10% and 30 seconds, 1 minute, 3 minutes, 5 minutes, 10 minutes and 15 minutes. The ICP-OES results show that 2%, 2.5%, 5% and 10% AR have a similar indium dissolution rate after 10 minutes and 15 minutes of sonication, whereas 1% AR had a lower indium dissolution rate (Figure 3.9).

As-is samples of GB were also leached in a shaker using different concentrations of AR ranging from 1% to

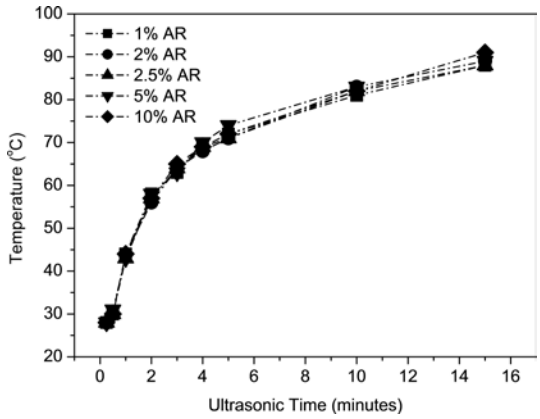


Figure 3.8. Temperature profile of different concentrations of AR under US sonication.

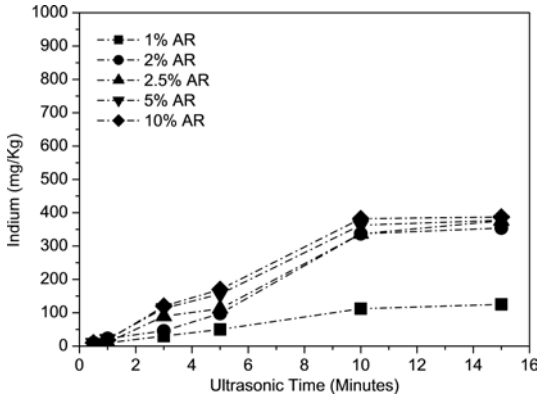


Figure 3.9. C2-M2-GF glass leached in 1–10% AR for different time periods from 30 minutes to 15 minutes under the influence of US waves. The data show the influence of time and AR concentration on indium leaching.

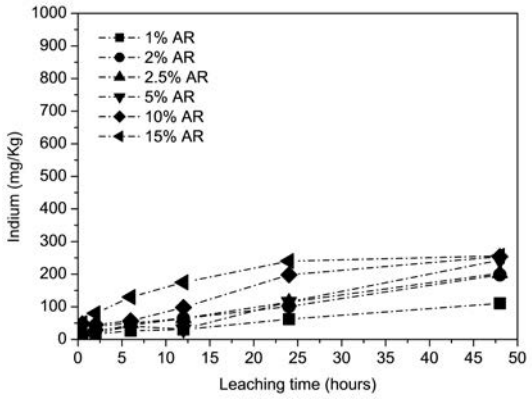


Figure 3.10. C2-M2-GB glass leached in 1–10% AR for different time periods from 30 minutes to 48 hours in a shaker at 70°C and 200 rpm agitation was analysed using ICP-OES. The data show the influence of time and AR concentration on indium leaching.

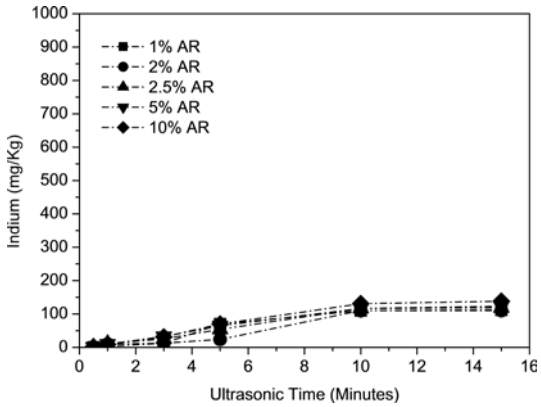


Figure 3.11. C2-M2-GB glass leached in 1–10% AR for different time periods from 30 minutes to 15 minutes under the influence of US waves. The data show the influence of time and AR concentration on indium leaching.

15% for different time periods ranging from 30 minutes to 48 hours at 70°C and 200 rpm agitation. The results show that indium dissolution from GB is lower than that from GF. AR of 15% and 10% was found to achieve the highest possible dissolution after 24 hours of leaching whereas for other AR concentrations the leaching time required to reach the highest possible dissolution rate was 48 hours (Figure 3.10).

The dissolution of indium from GB under the influence of US waves is shown in Figure 3.11. All of the concentrations of AR showed a similar trend and indium dissolution values.

3.3 Hydrometallurgy: Bulk Ground Samples

The bulk grinding of the GF and GB samples was performed on C2-M3 samples. These samples were ground to powder for approximately 5 minutes using the mortar and pestle technique, as discussed in Chapter 2. The powder obtained was filtered through a series of sieves to separate the powder into 53 µm particles, 125 mm particles, 250 mm particles (particles remaining after filtration) and 500 µm particles (powder obtained after bulk grinding without filtration

Figure 3.12. C2-M3-GF SEM image showing GF particles filtered through a 53 µm filter after bulk grinding.

Figure 3.13. C2-M3-GF SEM image showing GF particles filtered through a 125 µm filter after bulk grinding.

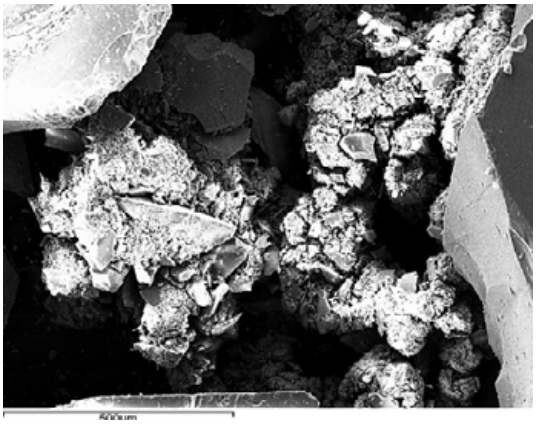


Figure 3.14. C2-M3-GF SEM image showing GF particles remaining after filtration through 53 µm and 125 µm filters. The approximate sample size is 250 µm.

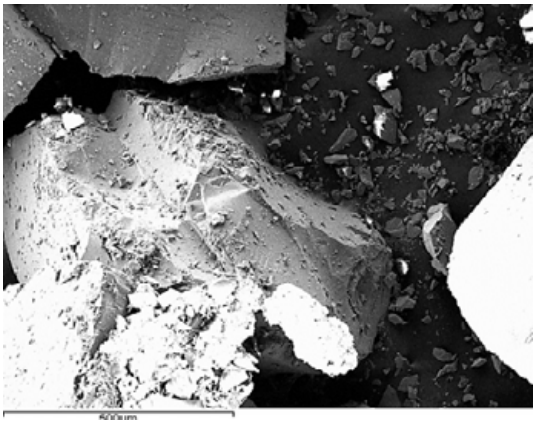


Figure 3.15. C2-M3-GF SEM image showing GF particles obtained after grinding but without filtration. The approximate particle size is 500 µm.

performed). Figures 3.12–3.15 display SEM images of the four GF samples.

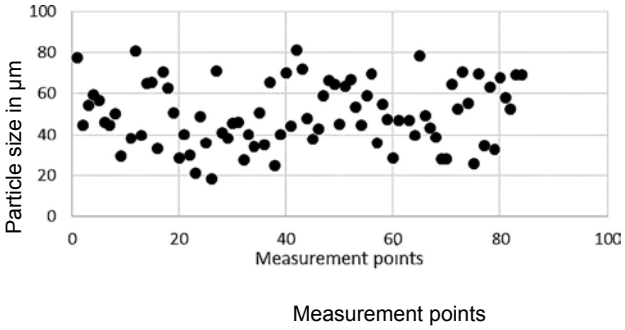
In Figures 3.12 and 3.13 it can be seen that the particle size is uniformly distributed, whereas, in Figures 3.14 and 3.15, among the particles of size 250 and 500 µm there are finer particles embedded in the matrix of a brighter material, which is considered to be due to the presence of the colour dye in the pixels. The four different samples were analysed using ImageJ software to determine the average particle size. The data obtained after filtration through a 53 µm filter are shown in Figure 3.16. The average particle sizes for all four samples are provided in Table 3.3.

The filtered GB powders were also observed using SEM for an initial particle size analysis, as shown in Figures 3.17–3.20. The results were similar to those for the GF powders. In the 250 µm and 500 µm powders, the agglomerates of fine particles in the bright matrix were not found, in contrast to the GF powders, supporting the suggestion that the bright matrix material in the GF powders was due to the colour dye.

The four GB powder samples were analysed using ImageJ software to find the average particles size. The data obtained after filtration through a 53 µm filter are shown in Figure 3.21.

The average particle sizes for all four samples are provided in Table 3.4.

The EDX point analysis was carried out on the different particle sizes from the GF. Figure 3.22 shows the < 53 µm particle size point analysis, with



Measurement points

Figure 3.16. Particle size analysis of the bulk ground GF sample after filtration through a 53 µm filter.

Table 3.3. Particle size analysis of the bulk ground GF samples shown in Figures 3.12–3.15

Sample	Average particle size (µm)
53 µm sieve	49.8
125 µm sieve	125
250 µm sieve	230
Pre-filtered sample	As bulk ground (500)

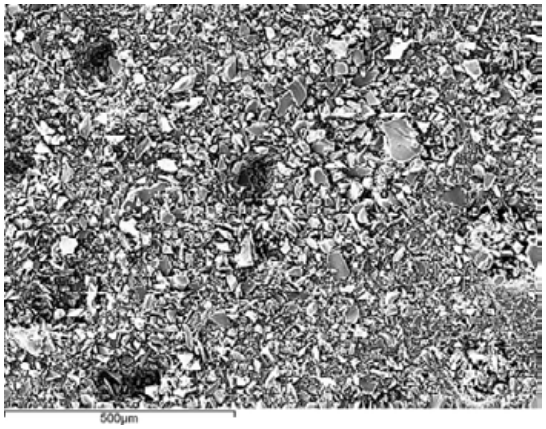


Figure 3.17. C2-M3-GB SEM image showing GB particles filtered through a 53 µm filter after bulk grinding.

Figure 3.18. C2-M3-GB SEM image showing GB particles filtered through a 125 µm filter after bulk grinding.

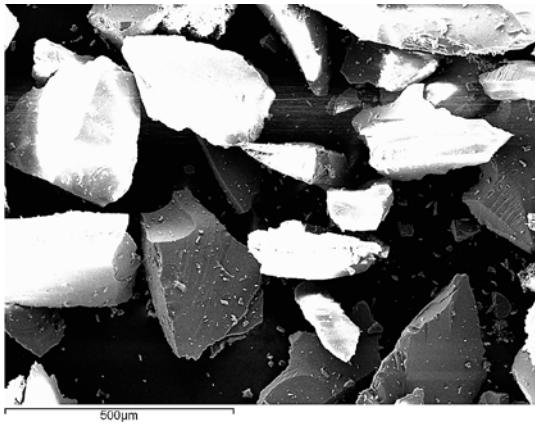


Figure 3.19. C2-M3-GB SEM image showing GB particles remaining after filtration through 53 µm and 125 µm filters. The approximate sample size is 250 µm.

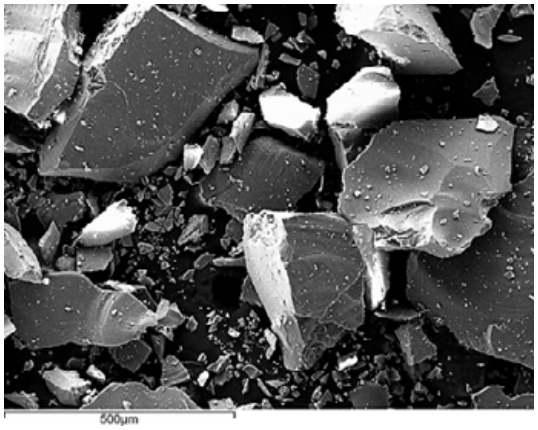


Figure 3.20. C2-M3-GB SEM image showing GB particles obtained after grinding but without filtration. The approximate particle size is 500 µm.

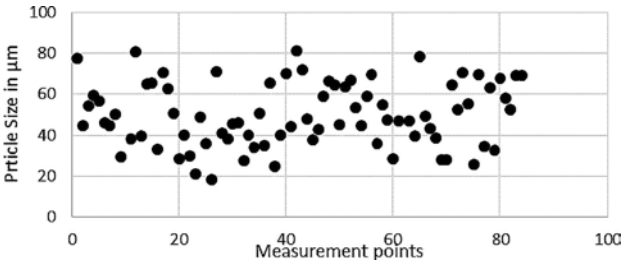


Figure 3.21. Particle size analysis of the GB bulk ground sample after filtration through a 53 µm filter.

Table 3.4. Particle size analysis of the bulk ground GB powders shown in Figures 3.17–3.20

Sample names	Average particle size (µm)
53 µm sieve	34.3
125 µm sieve	137
250 µm sieve	210
Pre-filtered sample	As bulk ground (500)

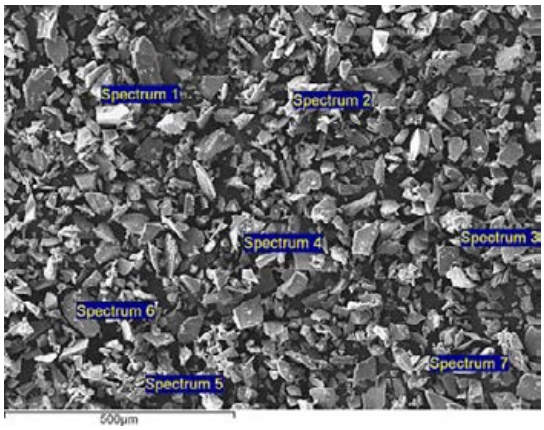


Figure 3.22. C2-M3-GF EDX point analysis of the < 53 µm particle size sample. Spectra 1–6, shown on the microstructure, are the points used for analysis.

Table 3.5. EDX mapping point analysis data in wt% from Figure 3.22

Spectrum	Magnesium	Aluminium	Silicon	Calcium	Indium	Tin
1	5.42	17.06	67.15	9.36	0.55	0.46
2	2.45	14.07	57.52	26.11	0.50	
3	4.33	14.21	51.60	23.32	4.68	1.86
4	4.57	16.39	68.03	9.49	0.32	1.20
5	3.83	16.08	65.25	14.25	0.39	0.20
6	3.86	16.42	67.40	13.03		0.02
7	2.72	13.63	50.88	30.42	0.34	2.01

seven spectrum points, and Table 3.5 shows the point analysis data. In almost all of the spectra, indium and tin were detected, along with magnesium, aluminium, silicon and calcium. These elements could have come from the colour dye and the glass.

The EDX point analysis was also carried out on the different particle sizes from the GB. Figure 3.23 shows the < 53 µm particle size point analysis, with six spectrum points.

Table 3.6 shows the corresponding data analysis. Almost all of the spectra showed the presence of indium and tin, as well as magnesium, aluminium, silicon and calcium. These additional elements could have come from the glass and the electrode connectors on the GB.

The bulk ground GF filtered samples (53 µm, 125 µm, 250 µm and 500 µm) were tested for leaching of indium in 10% AR for 24 hours in a shaker at 70°C and 200 rpm agitation. The analysis showed that the 53 µm particles leached a higher concentration of indium than the 125 and 250 µm particles; the 500 µm particles, which are unfiltered, leached a higher concentration of indium than the 125 and 250 µm particles but a lower concentration than the 53 µm particles (Figure 3.24).

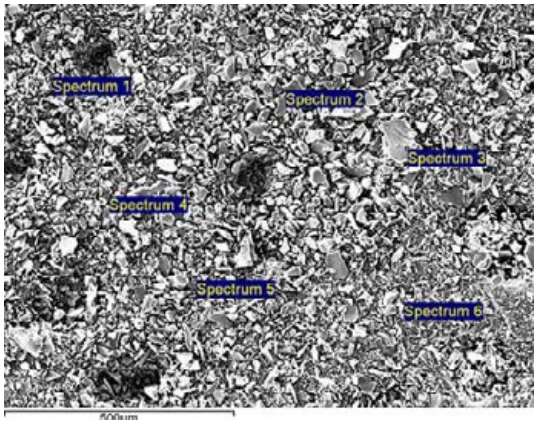


Figure 3.23. C2-M3-GB EDX point analysis of the < 53 µm particle size sample. Spectra 1–6, shown on the microstructure, are the points used for analysis.

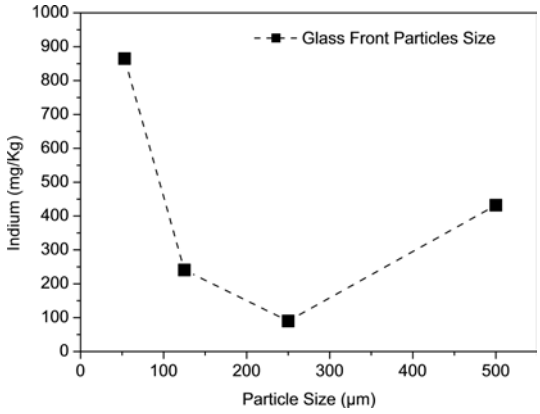


Figure 3.24. C2-M3-GF bulk ground powder filtered to four different particle sizes (53, 125, 250 and 500 µm) and tested for indium dissolution. The samples were dissolved in 10% AR for 24 hours in a shaker at 70°C and 200 rpm agitation.

Table 3.6. EDX mapping point analysis data in wt% from Figure 3.23

Spectrum	Magnesium	Aluminium	Silicon	Calcium	Indium	Tin
1	1.28	16.66	68.41	12.77	0.22	0.66
2	1.18	16.25	67.01	14.91	0.33	0.32
3	1.40	16.80	68.82	12.20	0.05	0.73
4	1.19	15.84	67.84	14.87	0.66	
5	1.45	17.84	66.95	13.06	0.27	0.43
6	1.46	16.96	68.29	12.57	2.42	

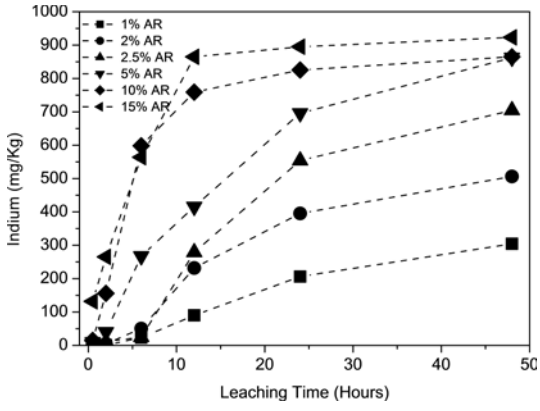


Figure 3.25. C2-M3-GF bulk ground powder filtered through a 53 µm filter was leached in 1–15% AR for different time periods from 30 minutes to 48 hours in a shaker at 70°C and 200 rpm agitation, followed by analysis by ICP-OES. The data show the influence of time and AR concentration on indium leaching.

This indicates that the 53 µm particles would be more useful for further study.

The 53 µm GF sample was leached in 1%, 2%, 2.5%, 5%, 10% and 15% AR for time periods of 30 minutes and 2, 6, 12, 24 and 48 hours, followed by measurement of the dissolved indium concentration using ICP-OES (see Chapter 2 for methodology). The results show that 15% and 10% AR have a similar indium dissolution pattern after 12 hours of leaching whereas 5% AR had a similar indium value to that for 15% and 10% AR after 48 hours of leaching (Figure 3.25). The trends also reveal that the rates of dissolution for the lower concentrations of AR were still gradually increasing at 48 hours, indicating that these AR concentrations will need a longer dissolution time, i.e. greater than 48 hours in this case.

The 53 µm GF sample was selected for further dissolution under the influence of US waves. The sample selected was leached in 1%, 2%, 2.5%, 5% and 10% AR for 30 seconds and 1, 3, 6, 12 and

15 minutes, followed by measurement of the dissolved indium concentration using ICP-OES (see Chapter 2 for methodology). The results show that 10% and 5% AR dissolved similar indium concentrations at 12 minutes and above (Figure 3.26). The indium concentration dissolved by 2.5%, 2% and 1% AR increased gradually with time but was always low.

The bulk ground GB filtered samples (53, 125, 250 and 500 µm) were also tested for leaching of indium in 10% AR for 24 hours in a shaker at 70°C and 200 rpm agitation. The analysis showed that the 53 µm particles leached a higher concentration of indium than the 125 and 250 µm particles; the 500 µm particles, which are unfiltered, leached a higher concentration of indium than the 125 and 250 µm particles but a lower concentration than the 53 µm particles (Figure 3.27). This indicates that the 53 µm particles are more useful for further studies.

The 53 µm GB sample was leached in 1%, 2%, 2.5%, 5%, 10% and 15% AR for time periods of 30 minutes and 2, 6, 12, 24 and 48 hours, followed by measurement of the dissolved indium concentration using ICP-OES (see Chapter 2 for methodology). The results show that 15% AR leaches the highest indium concentration, followed by 10%, 5%, 2.5%, 2% and 1% AR. For all AR concentrations, rapid indium dissolution was observed up to 12 hours, which was followed by a gradually increasing plateau. For low AR concentrations the results indicate a need for longer dissolution times to match the indium concentrations achieved by 15% AR (Figure 3.28).

The 53 µm GB sample was selected for further dissolution under the influence of US waves. The sample was leached in 1%, 2%, 2.5%, 5% and 10% AR for 30 seconds and 1, 3, 6, 12 and 15 minutes. The results show that 10% and 5% AR have similar indium dissolution rates throughout, whereas 2.5%, 2% and 1% AR show an increasing trend indicating a need for a higher sonication time to achieve the maximum indium dissolution rate (Figure 3.29).

3.4 Hydrometallurgy: Surface Ground Samples

The LCD screen GF (Figure 3.30a) was subjected to surface grinding with a wire brush, wire wool, silicon carbide (SiC) paper and a scraper. The resulting surfaces are shown in Figure 3.30b.

The scraper technique was found to have limitations, as it resulted in glass cracks and SiC paper was found to scratch the glass surface, which can potentially contaminate the powder. Table 3.7 shows the different techniques used for surface grinding of the GF, the time taken to remove a fixed area, the yield and the efficiency of the process.

The samples produced using the heavy steel scraper and the low-speed drill and metal wool were selected for further testing as the removal efficiency was 100%.

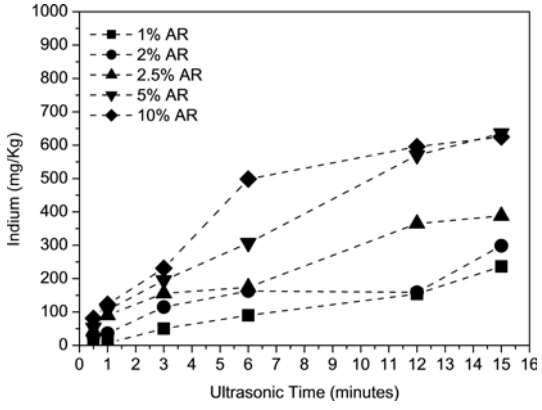


Figure 3.26. C2-M3-GF bulk ground powder filtered through a 53 µm filter was leached in 1–10% AR for different time periods from 30 seconds to 15 minutes under the influence of US waves, followed by analysis using ICP-OES. The data show the influence of time and AR concentration on indium leaching.

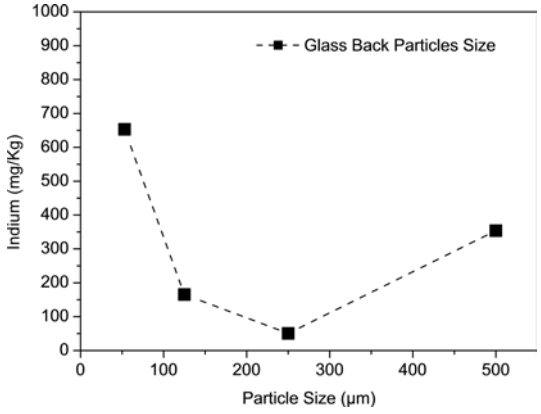


Figure 3.27. C2-M3-GB GB bulk ground powder filtered to four different particle sizes (53, 125, 250 and 500 µm) and tested for indium dissolution. The samples were dissolved in 10% AR in a shaker for 24 hours at 70°C and 200 rpm agitation.

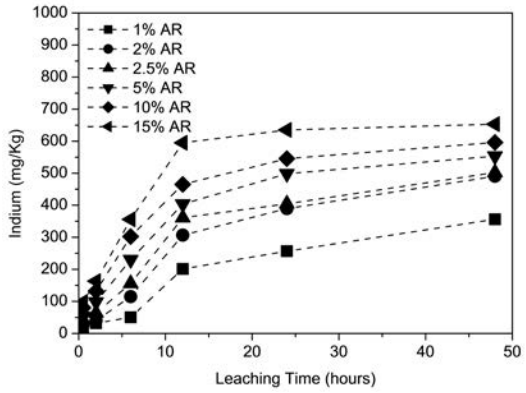


Figure 3.28. C2-M3-GB bulk ground powder filtered through a 53 µm filter was leached in 1–15% AR for different time periods from 30 minutes to 48 hours in a shaker at 70°C and 200 rpm agitation, followed by analysis using ICP-OES. The data show the influence of time and AR concentration on indium leaching.

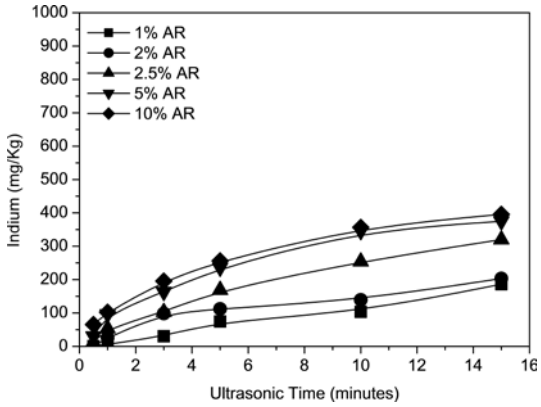
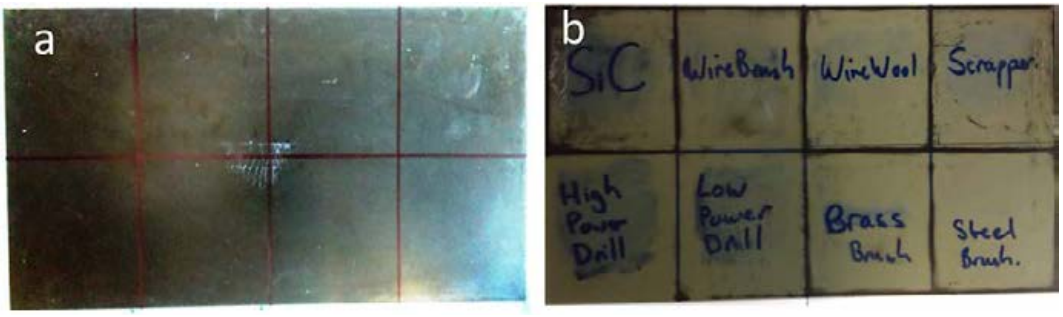


Figure 3.29. C2-M3-GB bulk ground powder filtered through a 53 µm filter was leached in 1–10% AR for different time periods from 30 seconds to 15 minutes under the influence of US waves. The data show the influence of time and AR concentration on indium leaching.



Before **After**

Figure 3.30. (a) The LCD screen GF segmented into equal areas to test the efficiency of different grinding techniques on ITO + colour dye removal from the glass surface. (b) The surface of the LCD screen GF after removal of ITO + colour dye using the different techniques indicated in the figure and also listed in Table 3.7.

Table 3.7. Different surface grinding techniques tested for ITO + colour dye removal from the GF

Extractor	ITO residue collected per area		
	Removal time (s)	Yield (g)	Efficiency (%)
Small wire brush (brass)	911.5	0.0253	20.9
Wire wool (steel)	579.0	0.0913	75.3
Small wire brush (steel)	554.8	0.0738	60.8
Grinding paper (SiC 320 grit)	455.2	0.0619	51.0
Low-speed drill + metal wool (618.8 rpm)	409.0	0.1213	100.0
Large wire brush (steel)	404.0	0.0898	74.0
Heavy scraper (steel)	383.0	0.1213	100.0
High-speed drill + metal wool (2924 rpm)	119.0	0.1213	100.0

The high-speed drill and metal wool technique showed 100% efficiency but the metal wool disintegrated because of the speed; hence, this removal technique was not considered further.

The powder produced by heavy metal scraper removal of ITO + colour dye from the GF was studied under SEM and EDX. Under SEM this powder was found to have long fibre-like structures ~500 µm in length that were bundled together. One observation that is critical for the scraper method is that the removal of ITO + colour dye surrounding the glass cracks was difficult. Such an attempt could potentially chip the glass at the crack boundaries. The ITO powder removed by the scraper was used for EDX point analysis. Six spectra were measured at random places (Figure 3.31) and the analysis data are reported in Table 3.8.

The EDX point scans showed the presence of indium of up to ~89 wt% in the points selected.

Next, the samples produced by surface grinding using metal wool to remove ITO + colour dye from the GF were examined. Under SEM this powder was found to have flakes that are ~100 µm in size (Figure 3.32). One observation that is critical for the metal wool technique is that the removal of ITO + colour dye surrounding the glass cracks was efficient and did not chip the glass. The metal wool-removed ITO powder was used for EDX point analysis. Six spectra were measured at random places and the analysis data are reported in Table 3.9.

The metal wool and scraper-removed ITO + colour dye was leached in 10% AR for different time intervals. The dissolution of indium was carried out in a shaker at 200 rpm. The analysis data are presented in Figure 3.33. The scraper was found to leach a lower concentration of indium in AR than metal wool. Metal wool was therefore selected for further analysis.

Figure 3.34 shows the metal wool-removed ITO + colour dye dissolution in AR of different concentrations and for different time intervals. AR at 15% and 10% show similar indium dissolution rates; 5% AR has a lower indium dissolution rate than 10%

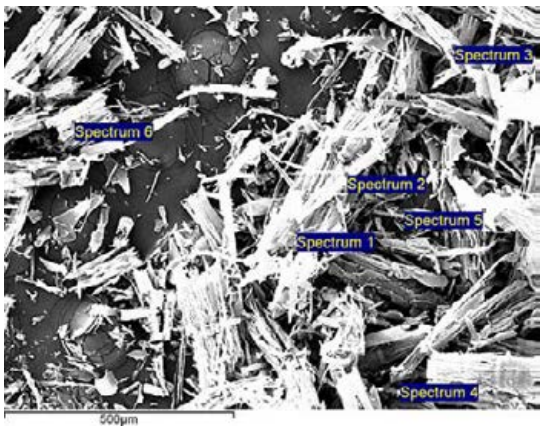


Figure 3.31. C2-M1-GF microstructure used to perform EDX point analysis. Spectra 1–6, shown on the microstructure, are the points used for analysis.

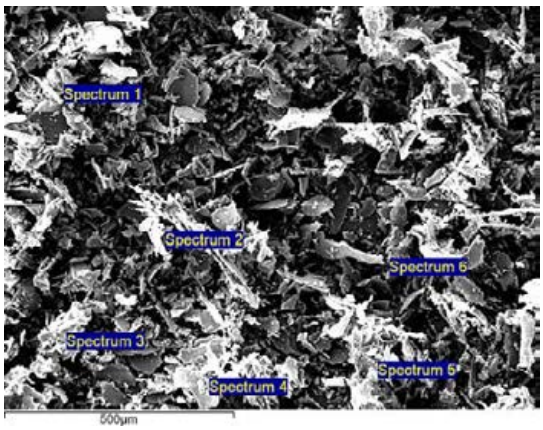


Figure 3.32. C2-M1-GF microstructure used to perform EDX point analysis. Spectra 1–6, shown on the microstructure, are the points used for analysis.

Table 3.8. EDX mapping point analysis data in wt% from Figure 3.31

Spectrum	Magnesium	Aluminium	Silicon	Calcium	Indium	Tin
1	0.14	0.65	0.99	0.15	89.27	8.80
2	0.68	2.24			89.82	7.97
3	0.10	2.11	0.87		90.61	6.85
4	0.32	1.70	0.10		89.19	9.01
5		1.75			89.52	9.54
6		1.47	0.31		89.81	8.90

Table 3.9. EDX mapping point analysis data in wt% from Figure 3.32

Spectrum	Magnesium	Aluminium	Silicon	Calcium	Indium	Tin
1	0.14	0.65	0.99	0.15	89.27	8.80
2	0.68	2.24			89.82	7.97
3	0.10	2.11	0.87		90.61	6.85
4	0.32	1.70	0.10		89.19	9.01
5		1.75			89.52	9.54
6		1.47	0.31		89.81	8.90

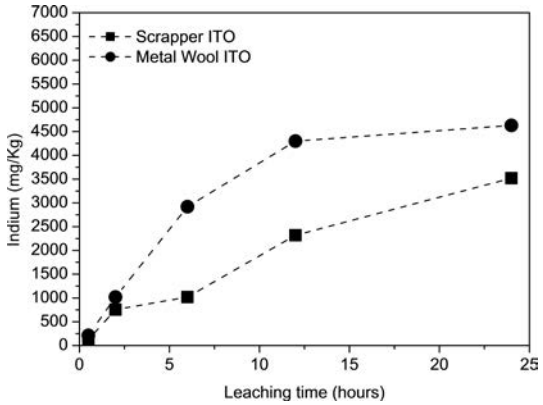


Figure 3.33. C2-M1-GF colour dye + ITO leached in 10% AR for different time periods from 30 minutes to 24 hours in a shaker at 70°C and 200 rpm agitation, followed by analysis using ICP-OES. The data show the influence of time and 10% AR on indium leaching.

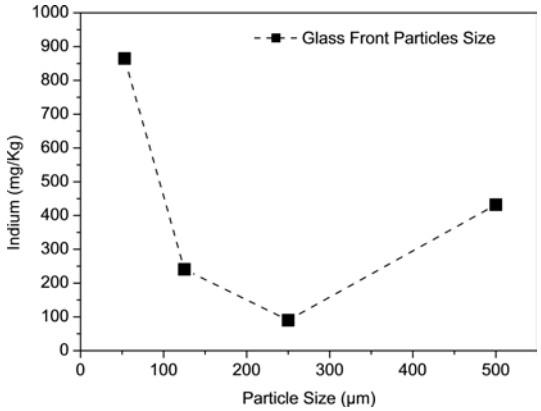


Figure 3.34. C2-M1-GF and C2-M5-GF colour dye + ITO leached in 1–15% AR for different time periods from 30 minutes to 48 hours in a shaker at 70°C and 200 rpm agitation, followed by analysis using ICP-OES. The data show the influence of time and AR concentration on indium leaching.

and 15% AR at 24 hours but a similar rate at 48 hours. This indicates that the high AR concentrations can dissolve indium more quickly. Given a prolonged dissolution time, the low AR concentrations can reach the maximum indium dissolution rate.

Powders from the GB using surface removal techniques were not analysed as the trials to generate ITO from the GB using metal wool and scraper techniques were unsuccessful.

The scraper-removed ITO and metal wool-removed ITO were dissolved in 10% AR for 15 minutes under the influence of US waves. The scraper and metal wool-removed ITO showed similar rates of indium dissolution after 5 minutes of US application (Figure 3.35). With US application of between 5 and 15 minutes, the metal wool-removed ITO showed greater dissolution of indium. Further understanding was sought by subjecting this powder to different AR concentrations and time under US influence. AR at concentrations of 10% and 5% under the influence of US waves showed similar higher indium dissolution rates whereas 2.5%, 2% and 1% AR showed lower dissolution rates but with an increasing trend with time. This thus indicates that 10% and 5% AR need less time to dissolve indium whereas 2.5, 2 and 1% AR need more time (Figure 3.36).

3.5 Hydrometallurgy: Laser-processed Samples

The laser technique was used to remove ITO from the LCDs. The C2-M4-S1-GF and C2-M4-S1-GB samples were cut into dimensions of 10 cm × 10 cm and tested for ITO removal using spot laser and scan laser at 50 W power. The same technique and parameters were also used on the GFs and GBs of light-emitting diode (LED) displays. In the case of LEDs, the samples were tested with liquid crystals and without liquid crystals.

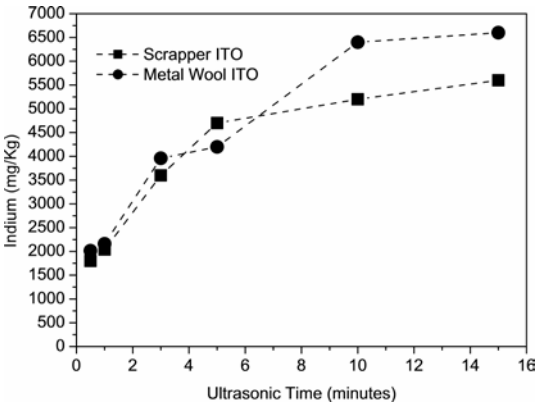


Figure 3.35. C2-M1-GF colour dye + ITO leached in 10% AR for different time periods from 30 seconds to 15 minutes under the influence of US waves. The data show the influence of time and AR concentration on indium leaching.

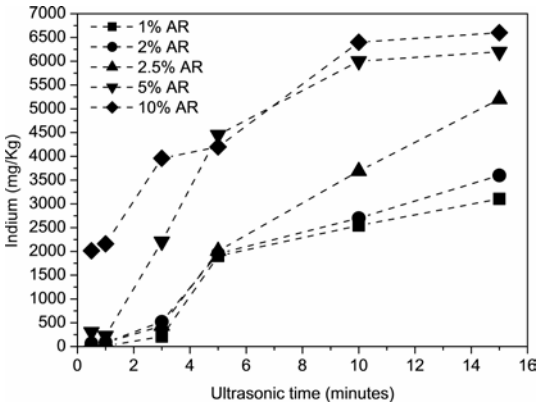


Figure 3.36. C2-M1-GF and C2-M5-GF colour dye + ITO leached in 1–10% AR of for different time periods from 30 seconds to 15 minutes under the influence of US waves. The data show the influence of time and AR concentration on indium leaching.

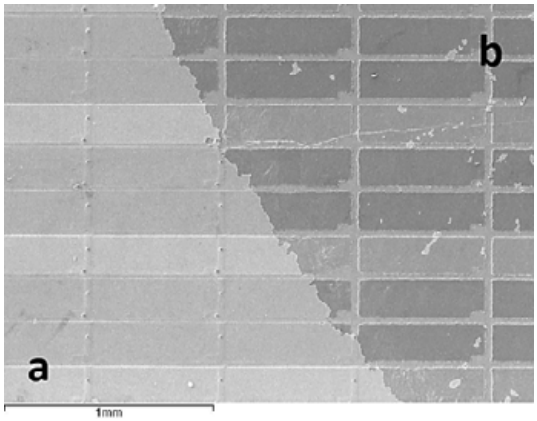


Figure 3.37. GF-C2-M4-S1: low-magnification SEM image of a GF sample ablated with a 50 W spot laser, showing the curvature of the spot: (a) the area that was unaffected by the laser and (b) the area ablated with the laser.

Figure 3.37 shows non-ablated (a) and laser-ablated (b) regions of the GF of an LCD display from which the liquid crystals were previously removed. The laser-ablated region shows clear pixels whereas the non-ablated region shows a grey cover over pixels. This cover could be an ITO layer. In order to test the influence of the laser on the screen, EDX mapping was performed, as shown in Figure 3.38.

The EDX mapping shows the presence of aluminium, silicon, calcium, indium and tin. In the maps, brighter regions indicate the presence of these elements and darker regions represent the absence of any elements. Therefore, the EDX mapping shows that the region that was ablated with the laser has lost most of the indium whereas the region that was not ablated still contains indium. This is the same for tin, thus confirming that the laser successfully removed the ITO layer. As the region rich in ITO is lost as a result of laser ablation, the elements that were once covered under the ITO are found to be brighter, indicating that colour dyes are rich in aluminium, silicon and calcium. It is interesting to find pixels and connectors that are unaffected in the laser-ablated region, indicating that the laser has selectively removed ITO.

The laser ablation technique was also tested on the LED GF in the presence of liquid crystals. When laser scans were made on samples with liquid crystals, a black smoke came off the surface, indicating the

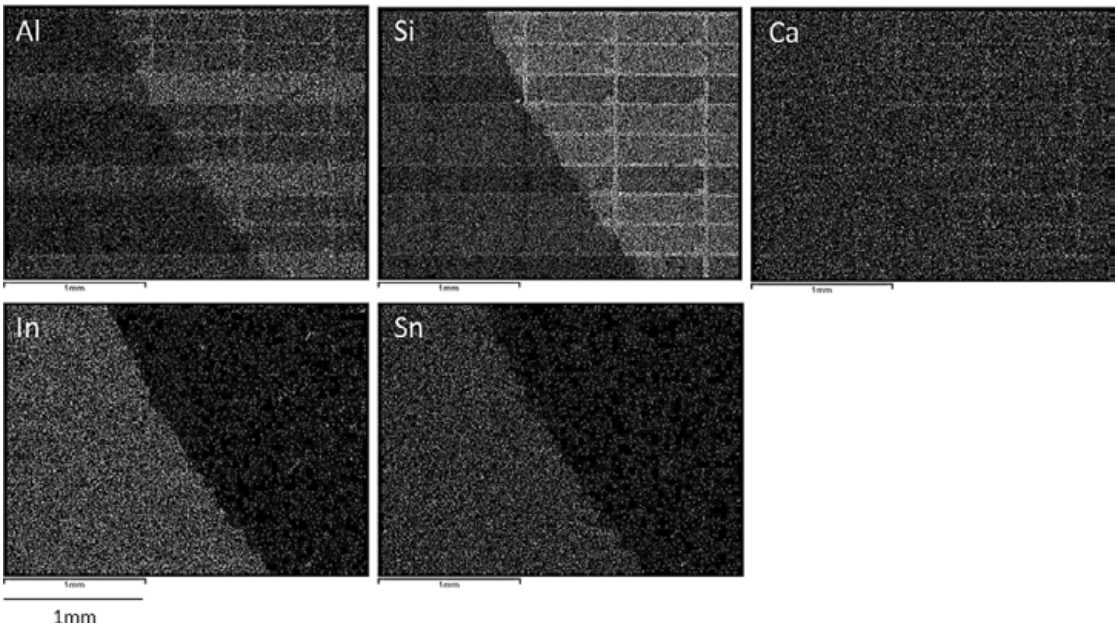


Figure 3.38. EDX mapping of the area shown in Figure 3.37. The darker regions in the maps represent the absence of any element and the brighter regions show the presence of elements. Mapping revealed the difference in aluminium, silicon, calcium, indium and tin distribution in region a and region b.

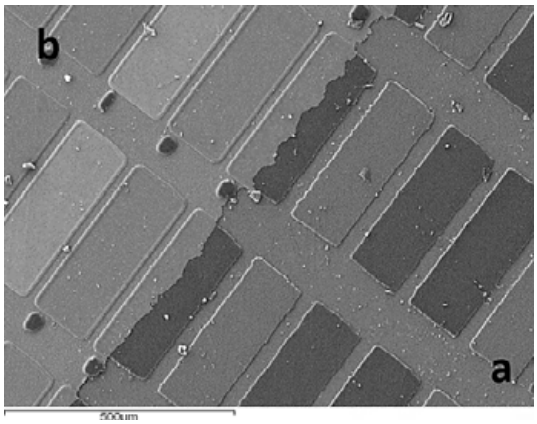


Figure 3.39. LED GF with liquid crystals: low-magnification SEM image of a GF sample ablated with a 50 W scan laser: (a) the area that was unaffected by the laser and (b) the area ablated with the laser.

evaporation of liquid crystals; this smoke was absent in the samples without liquid crystals. Figure 3.39 shows the results from the LED GF with liquid crystals. The microstructure is similar to that shown in Figure 3.37. The EDX mapping revealed a clear boundary between the laser-ablated region (b) and the non-ablated region (a). This boundary clearly separates the region with and the region without ITO (Figure 3.40). The laser ablation has selectively removed ITO from the LED glass.

The presence of liquid crystals had no influence on the removal of indium from the LED GF; there was a similar boundary between ablated and non-ablated regions in terms of microstructure and EDX mapping of other samples.

The laser ablation technique was also tested on the LED GB in the presence of liquid crystals and in the absence of liquid crystals. Figure 3.41 shows the results from the LED GB with liquid crystals. The microstructure is similar to that revealed for the LCD GB. The EDX mapping revealed a clear boundary between the laser-ablated region (b) and the non-ablated region (a). In the region that was ablated the ITO was removed and the electrodes in the GB were destroyed (Figure 3.42).

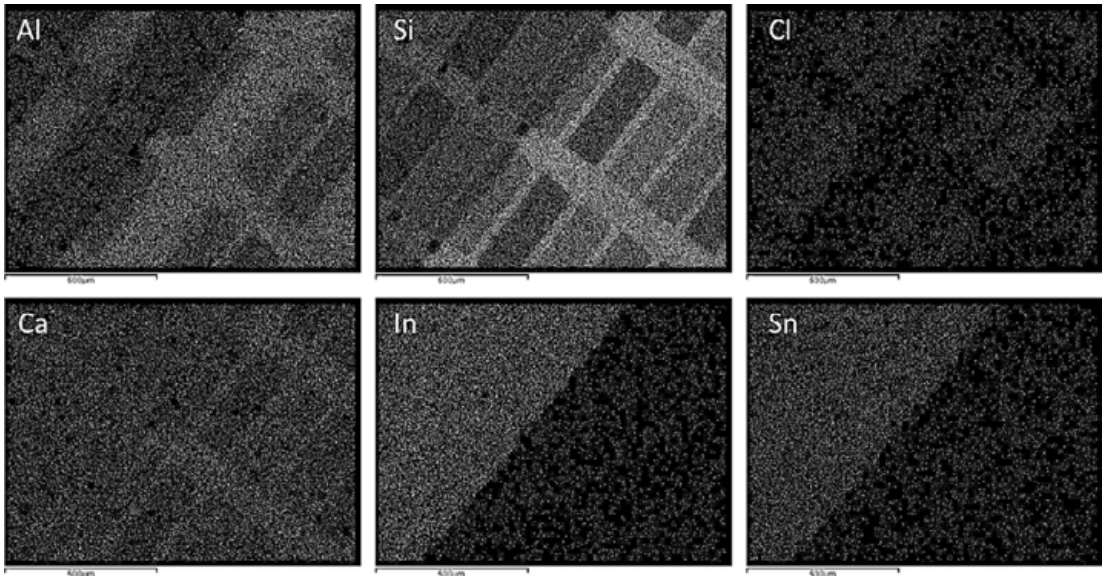


Figure 3.40. EDX mapping of the area shown in Figure 3.39. The darker regions in the maps represent the absence of any element and the brighter regions show the presence of elements. Mapping revealed the difference in aluminium, silicon, calcium, indium and tin distribution in region a and region b.

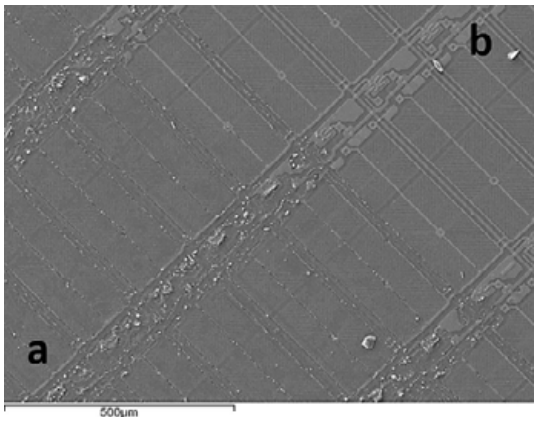


Figure 3.41. LED GB with liquid crystals: low-magnification SEM image of a GB sample ablated with a 50 W scan laser: (a) the area that was unaffected by the laser and (b) the area ablated with the laser.

The presence of liquid crystals had no influence on the removal of indium from the GB. A similar boundary between ablated and non-ablated regions in terms of microstructure and EDX mapping was found for both types of sample.

3.5.1 Capture of ITO from the laser ablation process

During ablation of the LED GF and the GB with the laser, the generated ITO, which produces vapour, was captured by absorption on to different media such as filter paper, glass, cotton wool and water. This was carried out both with and without liquid crystals to assess the effect of the liquid crystals on the quality and quantity of the recovered ITO. All of the media with the absorbed ITO, apart from water, were analysed for indium using ICP-OES (Figures 3.43 and 3.44). The water media samples were not measured with ICP-OES because the method used to collect the water samples from laser ablation resulted in greater losses of ITO.

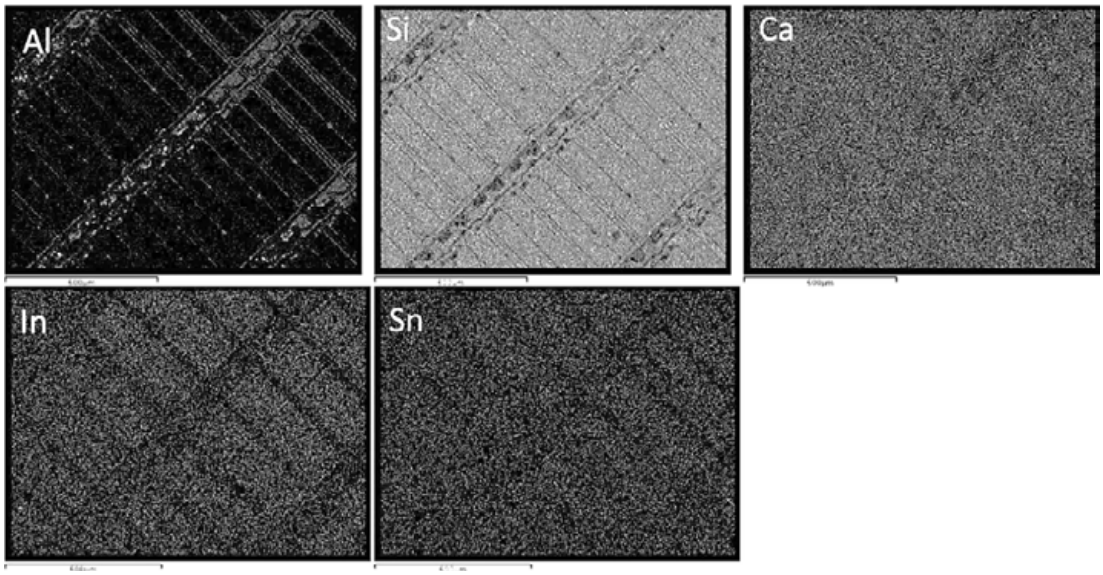


Figure 3.42. EDX mapping of the area shown in Figure 3.41. The EDX mapping revealed the presence of aluminium, silicon, chlorine, calcium, indium and tin.

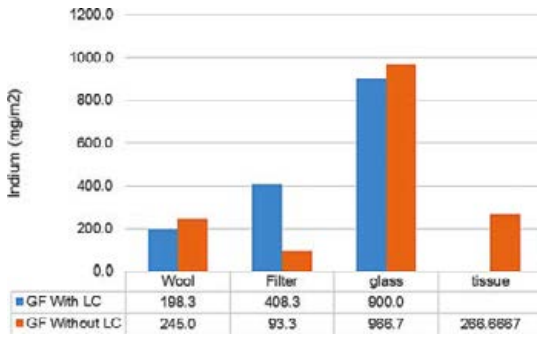


Figure 3.43. LED GF with and without liquid crystals: ICP-OES analysis of ITO collected onto cotton wool, filter paper, a glass slide and tissue paper during laser ablation.

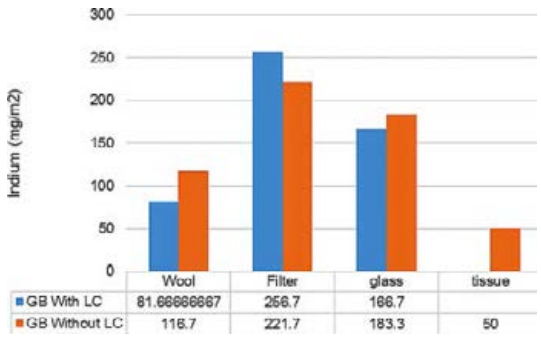


Figure 3.44. LED GB with and without liquid crystals: ICP-OES analysis of ITO collected onto cotton wool, filter paper, a glass slide and tissue paper during laser ablation.

4 Hydrometallurgical Discussion and Conclusion

4.1 As-is Samples

As-is GF and GB samples were leached in different concentrations of AR for different time periods. The maximum time period used for leaching was 48 hours and the maximum acid concentration was 15%. Leaching performed on GF samples with 15% AR for 30 minutes showed the highest indium dissolution rate. AR at a concentration of 15% showed consistent indium leaching values from 24 hours to 48 hours. A similar study by Forte (2014) found a maximum indium dissolution rate at 24 hours. AR at a concentration of 5% showed similar levels of indium leaching as 15% AR at 6 hours and 12 hours, but at 24 hours and 48 hours it reached a plateau, which matched the trend seen with 10% AR. Leaching of GF samples with 2.5% and 2% AR showed similar results, indicating that this slight change in the acid concentration has no influence on as-is leaching of samples. The 1% AR samples showed the lowest level of indium leaching; there was a gradual increase in leaching even up to 48 hours.

As-is GB samples were leached using a similar method to that used for GF samples. The highest rate of indium leaching was seen with 15% AR. Between 24 and 48 hours of leaching, the 15% AR samples reached a plateau, indicating that no leaching occurred after 24 hours. A similar pattern was observed for 10% AR, with a plateau seen from 24 hours to 48 hours. Interestingly, 5% AR showed a similar indium leaching rate as that for 15% and 10% AR at 48 hours of leaching. AR at concentrations of 2% and 2.5% showed an increasing trend, even after 48 hours of leaching. These results indicate that lower AR concentrations can achieve the highest indium recovery rates with longer durations of leaching. The same indium recovery rate can be achieved more quickly if higher AR concentrations are used (Li *et al*., 2002). These results also revealed that the indium content in the GB samples is almost half that in the GF samples.

As-is GF samples were leached in an ultrasonic sonicator in different concentrations of AR (1–10%) for different time periods (from 30 seconds to 15 minutes). Compared with leaching in an incubator at 70°C, the US dissolution of indium is a dynamic mechanism. When US waves were applied the temperature of the solution increased up to 90°C in 15 minutes. The increase in temperature occurs because of collapsing cavities that are generated during sonication. When collapsed these bubbles generate high temperatures and pressures near the surfaces, resulting in faster ITO dissolution (Li *et al*., 2011; Zhang *et al*., 2017). Hence, a greater indium dissolution rate was recorded within 15 minutes of sonication, which was possible with 10% AR after incubating for 24 hours at 70°C and 200 rpm mixing. This indicates the efficiency of the ultrasonic process.

As discussed, in the case of GF samples, the highest indium dissolution rate was seen with 10% AR, with the same trend and similar values seen for 5%, 2% and 2.5% AR. AR at a concentration of 1% showed the lowest indium dissolution rate. This indicates that, as well as the pressure and temperature exerted by the US waves, acid concentration plays a major role in the dissolution of ITO. Similar results were found for GB samples. Indium leached in GB was more than half that of the indium leached from GF, supporting our finding that GB has less indium content. A similar study also found an improved indium leaching rate in acid with the help of US waves (Souada *et al*., 2018). These findings are similar to findings from other studies in which sonication improved the recovery of metal from waste (Álvarez Sánchez *et al*., 2008; Huang *et al*., 2011; Wang *et al*., 2011; Li *et al*., 2014).

There were some limitations of the sonication process because of technical reasons. AR at a concentration of 15% was not tested and testing for longer than 15 minutes was not performed. The ultrasonic sonicator was made of titanium and it was thought that stronger acids could damage the titanium probe and internal parts of the equipment. In addition, the temperature of the acid solution reached 90°C at 15 minutes of sonication, which was found to vaporise the acid. Such acid vaporisation could potentially damage the sonicator by corrosion. Hence, these parameters were avoided.

4.2 Bulk Ground Samples

The mortar and pestle bulk ground GF and GB filtered samples were analysed for particle size. The average particle size after passing the samples through a < 53 µm sieve was greater for GF samples (49.8 µm) than for GB samples (34.3 µm). This can be attributed to the presence of a ~2 µm thick colour dye in the GF samples, which acted as a binding agent between the finer particles. When crushing the dye, it could have acted as a shock absorber to reduce the ability of the glass to break into finer particles. The colour dye is absent from the GB samples; hence, taking advantage of the glass brittleness, a finer particle size was achieved. The effect of the dye becomes negligible with increasing particle size and any deviations can only be an effect of crushing in the mortar.

An analysis was performed of indium dissolution for different GF particle sizes using 10% AR and 24 hours in a shaker at 70°C and 200 rpm agitation. These parameters were selected from the knowledge gained from the as-is samples. It was found that the 53 µm particle size sample showed the highest level of indium dissolution, at ~850 mg/kg. Abrasion of the surface of the glass particles during crushing could have liberated ITO from larger particles and concentrated it in the particles of size 53 µm; therefore, more indium is dissolved compared with particles of size 125 and 250 µm. This could prove that, because of increased colour dye content in the finer particles, a resistance to crushing could have occurred, which resulted in particles size of ~ 49.8 µm for GF samples.

A detailed study was performed of the 53 µm GF samples as they contain the majority of the ITO fraction. These samples were tested for the influence of time and acid concentration on the dissolution rate of indium. AR at a concentration of 15% showed the highest indium dissolution rate at 12 hours, after which the rate plateaued up to 48 hours. AR at a concentration of 10% followed a similar trend as 15% AR but showed a slightly lower dissolution rate. It showed an almost similar dissolution rate as 15% AR within 6 hours of leaching. AR at a concentration of 5% reached a similar indium dissolution level to that of 10% AR after 24 hours. Lower concentrations of 1%, 2% and 2.5% AR showed an increasing trend of indium dissolution up to 48 hours, indicating that leaching for even greater time periods will allow these concentrations to achieve indium dissolution rates similar to those for 10% and 15% AR.

An analysis was performed of indium dissolution for different GB particle sizes using 10% AR and 24 hours in a shaker at 70°C and 200 rpm agitation. It was found that the 53 µm GB particles showed the highest level of indium dissolution, indicating that ITO from the larger particles was removed as a result of abrasion and concentrated as finer particles (as for the GF samples). The 53 µm particles size sample was analysed further to understand its dissolution over different time periods and using different AR concentrations. Analysis proved again that 15% AR produces the highest level of indium dissolution within 12 hours, which is maintained up to 48 hours. The 10% AR sample followed the same trend as the 15% AR sample but with an increase in indium dissolution up to 48 hours, indicating that a longer dissolution time period is necessary to meet the 15% AR dissolution rate. This was the same for 5%, 2.5% and 2% AR. All of these AR concentrations showed a lower indium dissolution rate and an increasing trend, indicating the need for a longer dissolution time beyond 48 hours to reach the maximum indium dissolution value. In the literature it has been suggested that particle size does not have any influence on indium recovery (Fontana *et al*., 2015); however, in this study it was found that particles size does have an influence on metal recovery, which may be related to the varying composition of the different-sized filtered particles.

An analysis of the effect of US waves on leaching was performed on the 53 µm samples using different concentrations of AR and different time periods. In this study US leaching was performed over time periods up to 15 minutes and using AR concentrations of 10% or less. AR at a concentration of 10% showed the highest level of indium dissolution. A gradual increase in indium dissolution was recorded up to 15 minutes and a plateau was not achieved in this case. Interestingly, 5% AR achieved a similar indium dissolution rate as that of 10% AR at 12 minutes and 15 minutes of sonication, indicating that lower acid concentrations can achieve higher indium dissolution rates within 15 minutes of sonication. In addition, in this study an increasing trend in indium dissolution was observed for all AR concentrations, with 10% and 5% AR showing the highest dissolution rates.

The slight increasing trend in indium dissolution after 15 minutes of sonication indicates that not all of the indium is dissolved in AR at 15 minutes and that a longer sonication time is required. This might be because the powder particles have been moved to dead spots in the beaker, i.e. places in the beaker where the ultrasonic cavitation effect is minimal and hence less mixing occurs. Therefore, a slow and gradual increase in indium dissolution after reaching a plateau may be seen.

4.3 Surface Ground Samples

Different surface grinding techniques were employed to remove the ITO layer from the GF surfaces. Of all the techniques tested, the metal wool and scraper removal techniques were considered for further testing. The scraper and metal wool techniques were successful only for GF samples, as described in Chapter 3. The ITO on the GB samples is less than 15 nm thick, as per the indium measured. The scraper tip and metal wool are not precision made and their tip sizes are in micrometers; hence, the scraper and metal wool slide over the GB surfaces and cannot penetrate the ITO layer to remove it. In the case of metal wool it is possible to abrase the GB surface using either ethanol or acetone to create a grip between the metal wool and the glass surface, which resulted in scratches on the glass, thus indicating that metal wool can partly remove ITO from GB surfaces. However, this was not a viable solution as not enough material was obtained for further analysis; hence, the scraper and metal wool techniques were employed only to remove ITO from GF samples.

The morphology of scraper-removed ITO and metal wool-removed ITO powder was examined under SEM and using EDX. The metal wool-removed ITO showed a flaky structure whereas scraper-removed ITO formed long fibres that were in bundles. The metal wool-removed ITO and scraper-removed ITO were tested for leaching over different time scales up to 24 hours using 10% AR in a shaker at 70°C and 200 rpm agitation. The metal wool-removed ITO had the highest dissolution rate, reaching a plateau within 12 hours, which continued up to 24 hours. Scraper-removed ITO leaching studies always showed lower indium levels than metal wool-removed ITO leaching studies, with a gradual increase up to 24 hours of leaching.

When dissolving samples in AR, it is possible that the colour dye on the pixels could play a role in delaying the leaching of ITO in the pixels; hence, less ITO dissolution and an increasing trend in ITO dissolution up to 24 hours were recorded for the scraper-removed ITO, whereas the metal wool-removed sample reached a plateau within 12 hours of leaching under the same conditions, potentially indicating the importance of morphology for the rate of dissolution of ITO.

A detailed study of the metal wool-removed ITO was performed by varying the leaching time and acid concentrations used for leaching. With an increase in the leaching time an increase in indium dissolution was recorded for all AR concentrations used. In these studies, 15% AR showed the highest rate of indium dissolution at every dissolution point recorded. A plateau was formed, indicating that most of the indium from the ITO was dissolved from 12 hours to 48 hours. Interestingly, a plateau was recorded for 10% AR from 12 hours and the indium dissolution rate was similar to that for 15% AR from 12 hours to 48 hours. AR at a concentration of 5% showed an increasing trend in dissolution and reached a similar indium dissolution rate as that recorded for 15% and 10% AR at 48 hours. AR at concentrations of 1%, 2% and 2.5% all showed an increasing trend in indium dissolution up to 48 hours, indicating that a longer leaching time is required for lower AR concentrations.

The powders produced from the glass surfaces by the metal wool and scraper techniques were also digested with the assistance of US. The results again showed higher indium dissolution rates for metal-removed ITO. One interesting aspect is that scraper-removed ITO and metal wool-removed ITO showed similar trends in indium dissolution up to 5 minutes of US leaching. Sonication started with the AR at room temperature; hence, in the initial stages temperature has a minimal effect on indium dissolution. The ultrasonic pressure and acid concentrations are the main factors that could influence the rate of dissolution.

For the metal wool samples,10% AR showed the highest indium dissolution rate, followed by 5% AR. Both of these samples showed a plateau between 10 minutes and 15 minutes of sonication. In contrast, 1%, 2% and 2.5% AR showed an increasing trend in indium dissolution, indicating that a longer sonication time is required for lower AR concentrations to reach a plateau.

4.4 Laser Samples

The spot or scan laser ablation process was performed on GF samples, with the total time of the spot laser or scan laser tests being less than 1 second. After spot laser ablation a change in the colour of the area ablated was observed. A similar removal test was performed on GB samples. The ablated and non-ablated areas were analysed under SEM and EDX to understand the influence of the laser on the ITO. The EDX mapping of the samples showed that the ablated area has no indium or tin, whereas in the non-ablated area indium and tin are present.

The laser ablation technique was also tested on LED display GF samples in the presence of liquid crystals and in the absence of liquid crystals. This is an indirect test method in which the laser is not directly applied to the ITO from the ITO side of the glass panel, but instead is applied from the other side and passes through all of the layers of the GF to focus on the ITO. When laser scans were carried out on samples with liquid crystals, a black smoke came off the surface, indicating the evaporation of liquid crystals; this smoke was absent from the samples without liquid crystals. The EDX mapping revealed a clear boundary between the ablated region and the non-ablated region. The laser ablation was proved to selectively remove ITO.

The laser ablation technique was also tested on LED display GB samples in the presence of liquid crystals and in the absence of liquid crystals. The results were similar to those seen for the GF samples. Ablation was found to destroy the electrodes in GB samples, as revealed by the aluminium EDX map; in this case the laser removed the ITO and the electrodes. The results showed that there was a similar boundary between ablated and non-ablated regions in terms of microstructure and EDX mapping.

While the GF samples were ablated with the laser the generated ITO was captured onto different media such as filter paper, glass, cotton wool and water. The ITO captured onto a glass surface was found to be particulate, with no major difference found between the GB and the GF samples. However, in the samples without liquid crystals, white spheres were seen, which were found to be indium from EDX analysis. In the samples with liquid crystals, such white spheres were not observed.

The capture of ITO onto water media from GF samples in the presence of liquid crystals and without liquid crystals was investigated. The problem faced in testing these samples was to obtain proper samples for SEM analysis. The ITO particles captured onto water were floating and they were not stable enough to place in the sample holder used in the SEM analysis. The EDX mapping of these samples showed that the particles are rich in aluminium, silicon and calcium, as well as indium and tin.

The ITO captured onto glass, filter paper and cotton wool, with and without liquid crystals, was subjected to ICP-OES measurements. The water samples were not measured with ICP-OES because the method used to collect the water samples resulted in a greater loss of ITO to surface tension of the container wall; hence, the ICP-OES results for the water samples would not represent the true level of indium collected.

The materials containing the captured ITO were dissolved in 10% AR in a shaker at 70°C and 200 rpm agitation for 10 hours, after which ICP-OES was performed. The ICP-OES measurements confirmed the presence of indium in the captured materials, indicating that the ITO was successfully captured. The glass surface captured the highest level of indium for GF samples and the filter paper captured the highest level of indium for GB samples.

4.5 Hydrometallurgical Study Conclusions

The as-is GF samples required 24 hours in a shaker and 15% AR to achieve the highest possible indium dissolution level of ~600 mg/kg. A slightly lower indium dissolution level of ~400 mg/kg was recorded when the acid concentration was reduced to 10%. When indium in the as-is samples was dissolved using US waves and 10% AR, an indium dissolution value of ~400 mg/kg was recorded after 10 minutes of sonication. In a shaker, two parameters can influence indium dissolution, time and temperature, but in the case of US waves another parameter, pressure, is involved, thus reducing the total dissolution time from 24 hours to 10 minutes.

The filtered bulk ground powder showed a higher indium dissolution rate for the 53 µm particle size for both GF and GB samples. Two conclusions can be

drawn from these observations, which could have a combined effect on indium dissolution:

- Indium could have dissolved more quickly from finer particles than from coarser particles because of the greater surface area.
- The higher dissolution rate seen for the smaller particles could be the result of the grinding process: when larger particles were abrased with finer particles, indium is removed from the larger surface areas and is found to concentrate in the finer particles; hence, filtration concentrated indium in the 53 µm particles. A trend of decreasing indium concentration with increasing particle size was observed for both GF and GB samples. For instance, in GF samples, ~900 mg/kg of indium was recorded for the 53 µm particles, 250 mg/kg for the 125 µm particles and 50 mg/kg for the 250 µm particles, thus indicating that the powder filtration process can separate indium from the bulk of the glass and concentrates in finer volumes.

In surface ground samples the ITO and colour dye were obtained by different treatments. After the removal of the ITO and colour dye the glass was transparent and could be recycled. It was found that the method of surface grinding is important as each method resulted in a powder with a different morphology. In this study the scraper method resulted in a powder containing long fibre-like structures bundled together whereas the metal wool technique resulted in flake-like structures. When these two powders were dissolved in AR, the flake-like structures obtained by abrasion with metal wool were found to leach indium more quickly than the fibre bundles obtained by abrasion with the scraper. This proves the importance of the powder structure in indium dissolution.

The ITO samples produced by metal wool grinding were dissolved in AR and it was found that the maximum indium dissolution level obtained was ~4500 mg/kg. The same samples dissolved under ultrasonication resulted in an indium dissolution level of ~6500 mg/kg, thus indicating that ultrasonic dissolution is better for surface ground powder.

In laser ablation, it is possible to selectively remove ITO from the surface of GF and GB samples. The laser was found to be transparent to the polariser, glass and colour dye; however, it breaks ITO into finer particles by liberating them from the surface of the GF and GB samples. Of the capturing medias tested, glass, filter paper and water were found to be effective. Such interface-captured ITO was found to be rich in indium.

5 Investigation of Scale-up of the Process

5.1 Main Objective

The main objective was to investigate the potential of automating any emerging process for indium recovery.

The specific objectives were to:

- develop process steps and flow diagrams for the process;
- evaluate automation of the potential process options;
- model the most viable selected process.

The potential processes that have been hydrometallurgically tested are as follows:

- as-is sample hydrometallurgy techniques;
- bulk grinding techniques;
- surface grinding techniques;
- laser ablation techniques.

The process flow for each process is presented in the following section. Process maps show what steps are required, with material inputs and outputs. Tables summarise the pros and cons of each process with regard to automation. The most viable processes for automation are shown graphically in section 5.3.

5.2 Process Steps, Flow Diagrams and Ease of Automation Assessment

5.2.1 Automation of the as-is hydrometallurgy technique

Dissolve ITO directly from the liquid crystal glass panel as delivered from a conveyor system

Process principle

In this process, LCD glass panels are delivered in bulk in containers (stillages) from different processors that have removed the liquid crystal glass panels from the LCDs in remote locations. Ideally, the containers would all have the same form for ease of automation of extraction of the liquid crystal glass panels. It is possible to automate this part of the process, but automation is likely to be complex and expensive because of the random nature of the liquid crystal glass panels as currently delivered. Pre-sorting of the liquid crystal glass panels adds labour and requires specialised containers. Manual loading is recommended if this process is adopted.

Process details

For efficient handling of the liquid crystal glass panels, the polariser film must be attached. The glass layers are usually broken and will not remain in single manageable pieces if the film is removed. However, the presence of the polariser film may pose a contamination risk to the chemical processes required to dissolve the ITO.

Liquid crystal is first washed off the individual glass layers. The layers need to be separated for this and subsequent processing. It is proposed that an acid-resistant rack would be used to contain the glass layers and keep them separated in the tanks.

The volume of acid required is likely to be at least the same as the volume of the glass layers being processed. The glass layers may be lowered into a pre-filled acid bath or a dry tank, to which the acid is added. After the acid has become saturated with ITO and/or lost effectiveness, it is drained and transported for ITO recovery.

The time required for dissolution and the throughput required will determine the number of sheets to be processed at any one time.

Figure 5.1 details the flow diagram for this process and Table 5.1 summaries the advantages and disadvantages of the process.

Ease of automation conclusion

The issues of the organic polariser film contaminating the acid bath, the expense of the large volume of acid required and the volume of potentially hazardous waste makes this option expensive and unattractive.

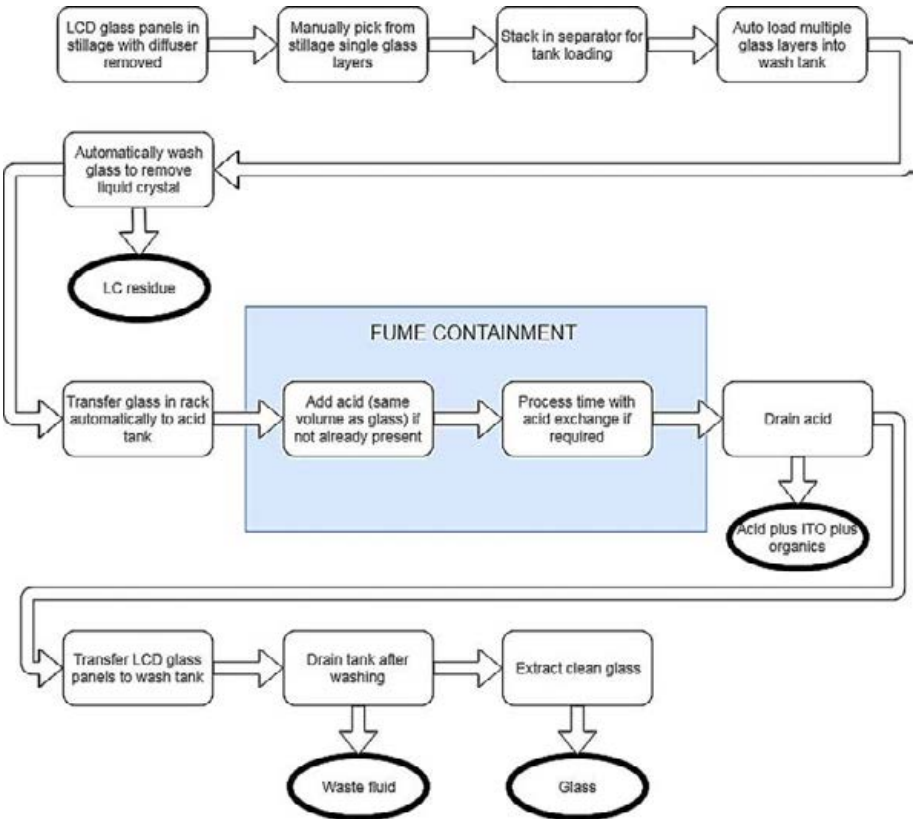


Figure 5.1. Dissolution directly from glass layers. LC, liquid crystal.

Table 5.1. As-is technique ITO removal from LCD panels: pros and cons

Pros	Cons
• LCD glass panels are potentially feedstock as opposed to being a waste product • Simple for recycler • Potentially local processing is possible for the panel separation process from the LCD	• Uses a significant volume of acid per panel (probably a minimum of the volume of glass per screen and potentially more) • Volume of dissolution tank (for batch processing or multiple smaller tanks for continuous processing) is likely to be very significant • Liquid crystal removal (washing) is required • Organic polariser film is likely to reduce the effectiveness of the acid for ITO removal • Wet chemistry process may not suit standard recycler • Dissolution time is significant without US • Ultrasonic generators need to vibrate the whole volume being processed to obtain the benefit of US • Ultrasonic vibration to accelerate the process causes evaporation of acid, which necessitates a closed container for dissolution • Produces a hazardous waste output • Acid not readily cost-effectively recycled, which potentially results in the production of a significant amount of hazardous waste • The consumables cost probably exceeds the recovered metals value depending on the throughput • Broken glass layers will make handling (loading and unloading) impractical if the polariser sheet is removed • Shipping costs of liquid crystal glass panels to a central location

5.2.2 *Automation of the bulk grinding powder hydrometallurgy technique*

Bulk processing panels by mortar and pestle or ball milling to generate a powder and sieving to extract the ITO-containing fraction

Process principle

In this process liquid crystal glass panels are collected from remote suppliers and ground in bulk. The liquid crystal glass panels are tipped into, for example, a ball mill and ground to a powder. The powder is sieved to separate fractions based on particle size. The smallest sized fraction, sub-53 µm sieved powder, has been shown to contain the highest proportion of ITO, as previously detailed in Chapter 4.

Process details

This process is tolerant of random and broken liquid crystal glass panel delivery. Each holding device or stillage of liquid crystal glass panels from the LCD extraction process is simply tipped into an appropriately sized ball mill or similar grinding machine for crushing. The output of this is a powder of mixed particle size. Sieving this delivers particulates of known sizes.

Removal of the polariser film may be carried out prior to the grinding step by dissolving it. This adds a consumable material and a further chemical process. Manual removal of this film is impractical because of the fragmented nature of the panels. Sizing of the equipment is dependent on the throughput required and the subsequent hydrometallurgical process time.

Figure 5.2 details the flow diagrams for this process and Table 5.2 summarises the advantages and disadvantages of the process.

Ease of automation conclusion

The lower yield of ITO from this process suggests that it is unlikely to be cost-effective; however, if the yield can be improved by an order of magnitude, this method is the simplest to automate.

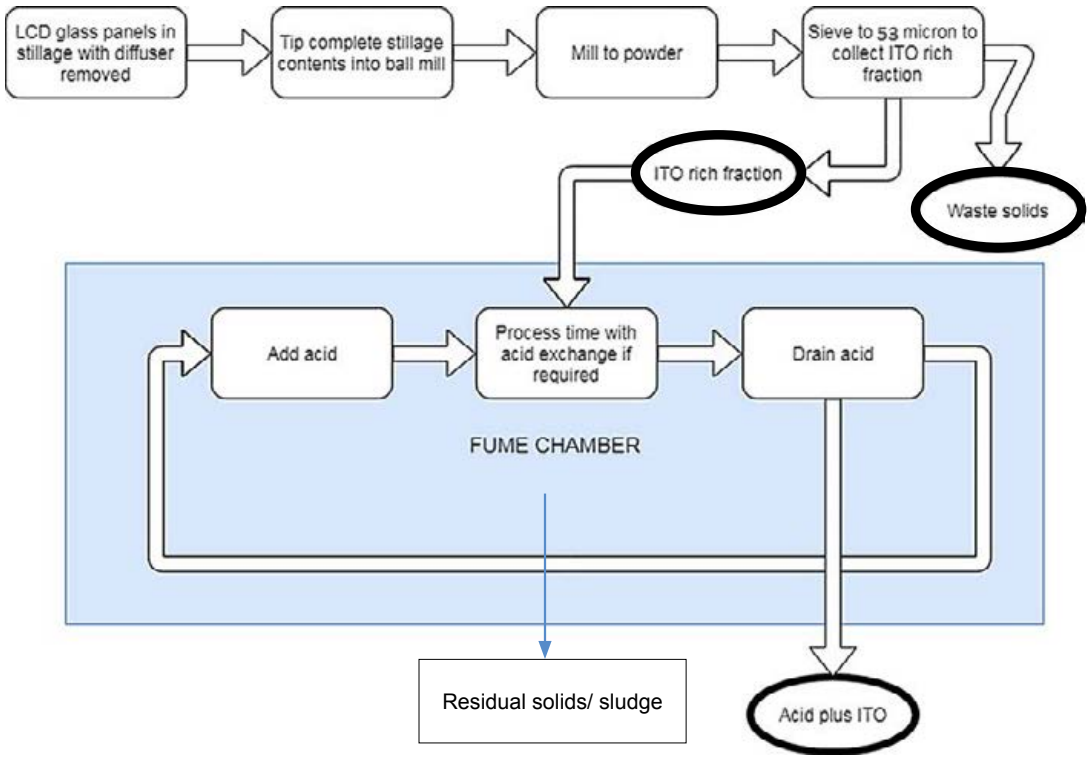


Figure 5.2. Ball milling of an entire LCD glass panel.

Table 5.2. Bulk grinding of LCD glass panels: pros and cons

Pros	Cons
• Bulk processing is applicable • Potentially fast enough for economic operation • Simpler automation than handling individual panels • Scalable • The 53 µm particles obtained by sieving will mainly consist of ITO • LCD panels are potentially feedstock as opposed to being a waste product	• High capital cost for high throughput equipment • Sieved material may be too impure for economic recovery • Produces a hazardous waste output • Shipping costs of liquid crystal glass panels to a central location • Polariser film is organic and may affect the hydrometallurgical ITO extraction process • Removal of polariser film prior to milling will add cost and an additional waste stream • Poor yield

5.2.3 Automation of the surface grinding powder hydrometallurgy technique

Surface grinding the ITO to a powder from the individual liquid crystal glass layers

Process principle

This process takes individual glass layers and scours or abrades the ITO mechanically from the surface of the glass. An operator loads the glass layers individually onto a conveyor, oriented with the ITO layer facing the abrasion device. The process then operates automatically, collecting the powder and delivering cleaned glass with the polariser film still attached.

Process details

Liquid crystal glass panels are assumed to be delivered in bulk in containers (stillages) from different processors, who have cut the liquid crystal glass panels from the LCDs in a remote location. It is possible to automate this part of the process, but automation is likely to be complex and expensive because of the random nature of the liquid crystal glass panels as currently delivered. Pre-sorting of the liquid crystal glass panels at the previous location adds labour and may require specialised containers, similar to the as-is technique.

Automating picking of the liquid crystal glass panels from random drops in stillages is complex and so manual picking and loading of the machine is recommended for the initial machine. If the liquid crystal glass panels can be supplied on a conveyor, this will reduce the labour content.

Washing the liquid crystal from each panel is carried out automatically as part of the process prior to abrading. At this step each glass layer is rolled to flatten broken fragments to improve the effectiveness of the abrading step.

Metal wool has been shown to be effective on the GF layer. More work is required to determine the effectiveness of ITO removal from the GB layer by this method. It is possible that two different methods of removal will be required for maximum effectiveness.

Alternative methods include grinding with an abrasive grit on a flap wheel or sanding belt. Potential issues from this are contamination of the ITO with grit, typically aluminium oxide, and glass. Collection of ITO from this grinding process is also likely to be challenging. The amount of ITO on the GB layer is less than that on the GF layer and so it may be not cost-effective to attempt removal of ITO using an automated process.

Figure 5.3 details the flow diagram for this process and Table 5.3 summarises the advantages and disadvantages of the process.

Ease of automation conclusion

This process has good potential for automation. The areas requiring further testing include optimisation of grinding media and collection and determination of ITO yield by this method. Scouring using steel wire wool contaminates the ITO with iron and researching alternatives is required. Scouring does not work on the GB layer and so there is no recovery possible using scouring.

Loading the machine with randomly supplied liquid crystal glass panels in stillages would be expensive

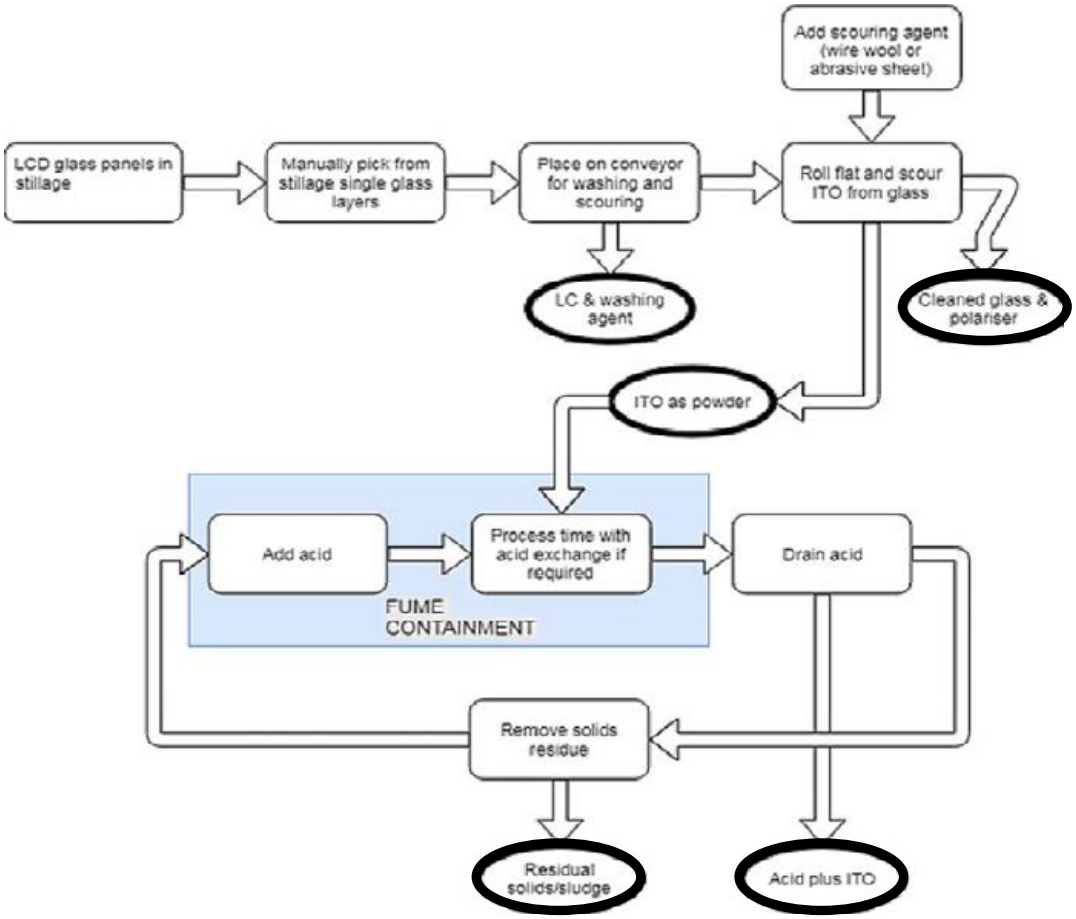


Figure 5.3. Surface grinding of LCD panels to remove ITO.

Table 5.3. Surface grinding of LCD panels: pros and cons

Pros	Cons
• Small volume of material to ship post processing • Faster dissolution • Less acid required than for the as-is technique • LCD panels are potentially feedstock as opposed to being a waste product • Potentially local processing • Use existing labour to load scouring/grinding recovery system • Potential for glass recovery (cleaned glass, mainly broken) • Potential to test the process with liquid crystals present	• Broken liquid crystal glass panels may be an issue for automatic handling (loading and unloading) • Difficult to grind broken liquid crystal glass panels • Wire wool is a consumable • Wire wool potentially contaminates and/or contains useful ITO • Abrasive may quickly become clogged • Changing wire wool or grinding medium is likely to be a manual process. Automating this step is unlikely to be cost-effective • Grinding from GB layer appears more difficult and may not be practicable • Liquid crystal removal (washing) is currently required • Shipping costs of LCD screens to a central location • Produces a hazardous waste output

to automate (high capital cost) and this may be less reliable than manual loading. Loading predictably oriented liquid crystal glass panels should be readily automated.

5.2.4 Automation of the laser processing technique

Laser removal of ITO from individual panels

Process principle

This process takes individual LCD glass panels and removes the ITO from the surface of the glass layers by heating with a focused laser. The particles of ITO thus removed are collected on a filter medium, with the potential for vacuum assistance. A manometer measuring the pressure drop across the filter medium determines when the filter is saturated. The filter is then indexed onto a new area to continue the process. It is envisaged that the filter will be a paper web fed from a roll and wound onto a second roll. When finished, this paper roll will contain the removed ITO. An alternative collection method is a conventional vacuum dust collector with re-usable filter. The process operates automatically, collecting the powder and delivering cleaned glass with the polariser film still attached.

The saturated roll is sent to the indium processor for indium recovery.

Process details

The LCD glass panels are assumed to be delivered in bulk in containers from different processors, who have cut the liquid crystal glass panels from the LCDs in a remote location. An operator loads the glass layers individually onto a conveyor, oriented with the ITO layer facing the filter paper. Ideally, the containers would all have the same form for ease of automation of extraction of the liquid crystal glass panels. Automation is likely to be complex and expensive because of the random nature of the liquid crystal glass panels as currently delivered. Pre-sorting of the liquid crystal glass panels by the previous processors adds labour and may require specialised containers.

Automating picking of the liquid crystal glass panels from random drops in stillages is complex and so manual picking and loading of the machine is recommended for the first machine. If the liquid crystal glass panels can be supplied on a conveyor, this will reduce the labour content.

Burning the liquid crystal itself produces smoke and so washing the liquid crystal off before the laser process may be necessary. Washing the liquid crystal from each panel is carried out automatically as part of the process. At this step the glass layer is rolled. This flattens broken fragments to enable focusing of the laser on the ITO layer.

The cleaned glass is delivered automatically from the output end of the conveyor. The filter paper containing the recovered ITO will be removed once the roll is finished and sent to the indium recovery site. A new filter roll is installed at this time. The frequency of changes depends on the rate of saturation, the filter pore size and the length of the web.

Figure 5.4 details the flow diagram for this process and Table 5.4 summarises the advantages and disadvantages of the process.

Ease of automation conclusion

This option has good potential for automation. The areas requiring further testing are ITO media collection and determination of yield. In addition, the capital cost of a laser of sufficient power for the speed required for commercial viability has to be determined.

Loading the machine with randomly supplied liquid crystal glass panels in stillages would be expensive to automate (high capital cost); however, loading predictably oriented liquid crystal glass panels should be readily automated.

5.3 Automation Solution Using the Laser or Surface Grinding Technique for ITO Removal

5.3.1 Overview

Laser removal of ITO and grinding of ITO from the surface of individual glass layers have been selected as the most viable options for automation. The

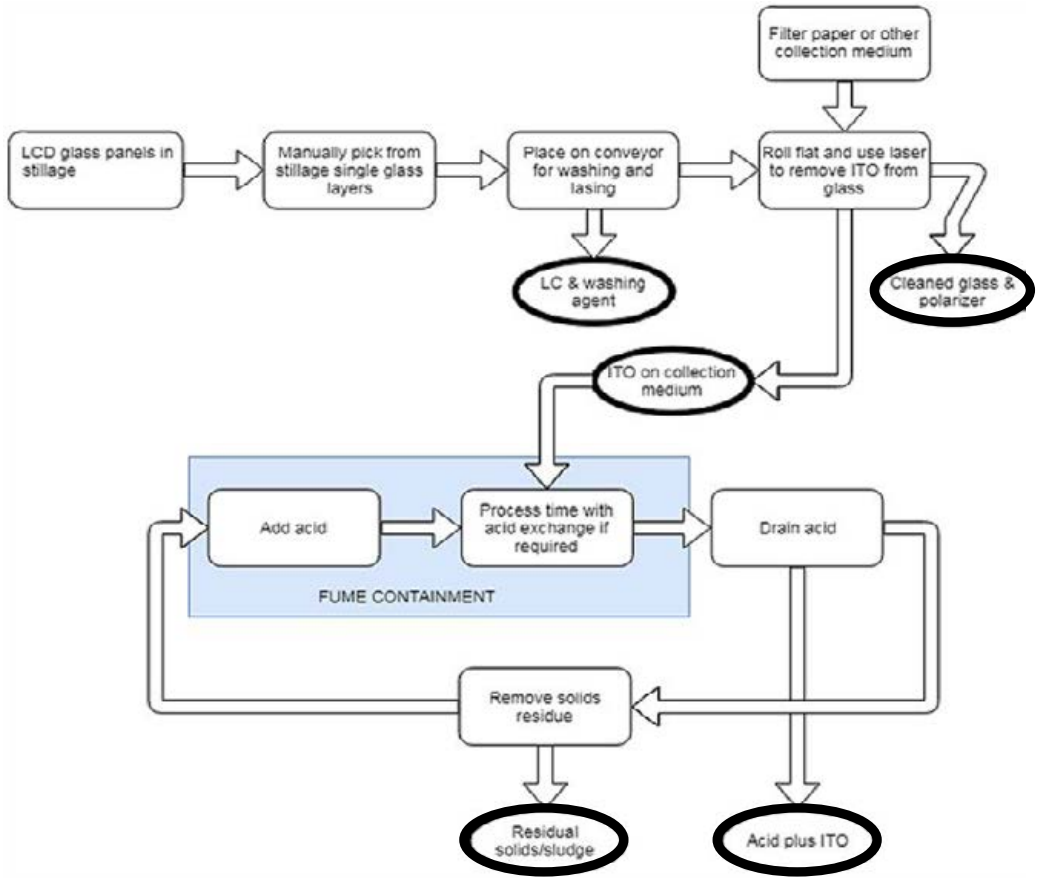


Figure 5.4. Laser removal of ITO.

Table 5.4. Laser removal of ITO: pros and cons

Pros	Cons
• Potential to use waste LCDs as a feedstock • Potential for glass recovery (cleaned glass, mainly broken) • Small volume of material to ship post processing • Faster dissolution • Potentially local processing	• Capital cost and complexity of liquid crystal glass panel handling at the start of the process • Random and broken liquid crystal glass panels supplied in stillages. Requires sorting either manually or with automation • Broken liquid crystal glass panels may be an issue for automatic handling (loading and unloading) • Shipping costs of LCD screens to a central location and return of custom stillages • Collection medium is a consumable • Collection medium may act as a bath contaminant • Liquid crystal removal (washing) is currently required • Produces a hazardous waste

equipment concept described below can be adapted to test both processes.

The LCD glass panel removed from the LCD is delivered to an operator work station. This can be either from a bulk container or directly from the removal process. The diffuser layers will already be removed.

Figure 5.5 shows equipment that is configured to take liquid crystal glass panels and/or liquid crystal glass layers from a conveyor, which may be loaded from stillages. All steps are carried out concurrently on glass layers as supplied by the operator. The slowest step determines the throughput of the machine.

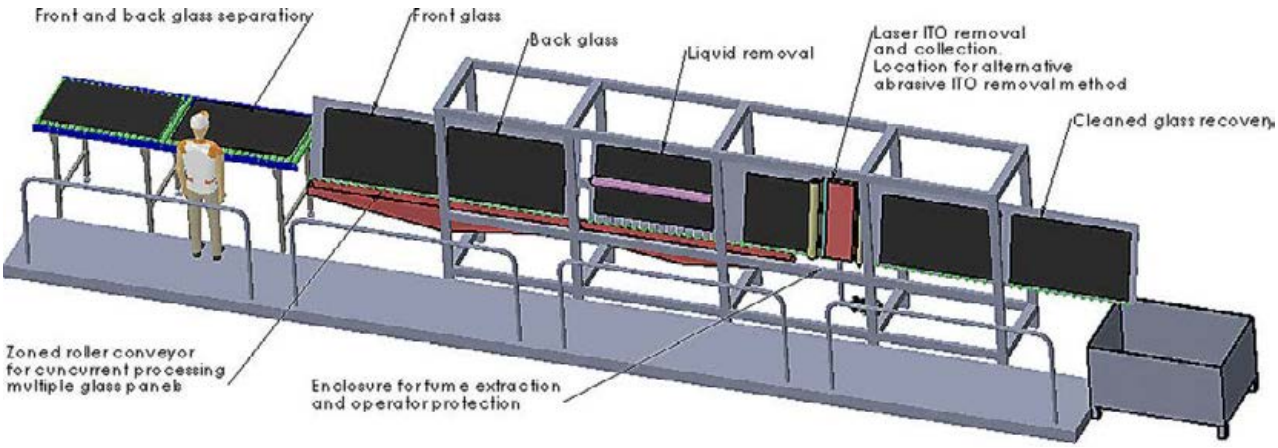


Figure 5.5. Overall view of the ITO recovery system using a laser or abrasion.

5.3.2 Operation steps

Loading the machine

The LCD panels are delivered in stillages and placed in front of the operator on a roller conveyor. The screen is indexed to the next station on a signal from the operator.

Glass layer separation

The glass layer is loaded onto the zoned roller conveyor in a near vertical plane, with the GB layer first (on top) and then the GF layer. The polariser film is oriented away from the operator (Figure 5.6). This leaves the ITO-coated surface available for cleaning at the next step. The conveyor indexes on command from the operator, after which the zoned conveyor operates automatically. Each glass layer is less than 1 kg and so is easily managed by an operator.

Liquid removal

In this stage, it is proposed to use a roller squeegee and/or washing and/or wiping process to remove traces of liquid crystal into a collection trough below the conveyor for disposal (Figure 5.7). A roller is likely to be required to flatten the glass layer prior to lasing or scouring/grinding. It is generally cracked by the time it reaches this stage and is held together by the polariser film.

The glass layer being cleaned is lifted from the conveyor to allow access to the total area. The conveyor indexes to the next step automatically on completion of cleaning.

ITO removal and recovery by laser or scouring/ grinding

In Figures 5.8 and 5.9, laser removal is assumed. The option of scouring or grinding is also possible at this stage.

In the laser ITO removal process, a laser scans vertically in sections, removing the ITO (see Figure 5.8). The removed ITO is collected on filter media (supplied on a roll) or other collection media using a vacuum to draw the ITO onto it (see Figure 5.9). ITO may also collect below the filter media under the effect of gravity. If this occurs, it may be worth installing a collection method for this residue. This gravity effect may be suitable for use of a scouring ITO removal method. Alternatively, it may be possible to deposit the ITO onto a glass surface for collection. How this will be achieved has not yet been determined. Proof of principle prototypes are required to test all methods.

After each laser sweep, the glass layer is indexed to allow the next sweep until it is cleaned. This is an automatic process. A manometer will determine when to index or measure the collection medium. It is envisaged that the collection medium will become effectively saturated with ITO. Vacuum pump speed control may be required to maximise collection efficiency.

The vacuum collection method can also be used to contain any fumes generated by the laser removal process.

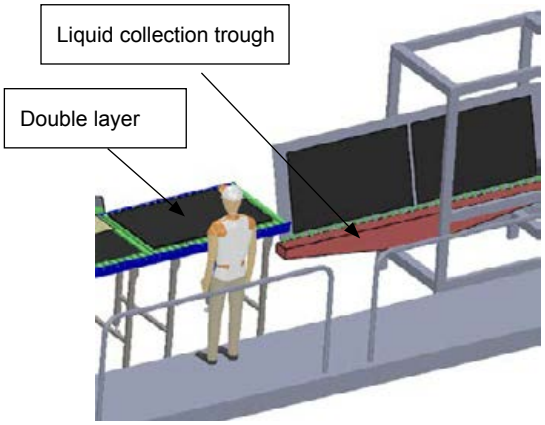


Figure 5.6. Glass layer separation.

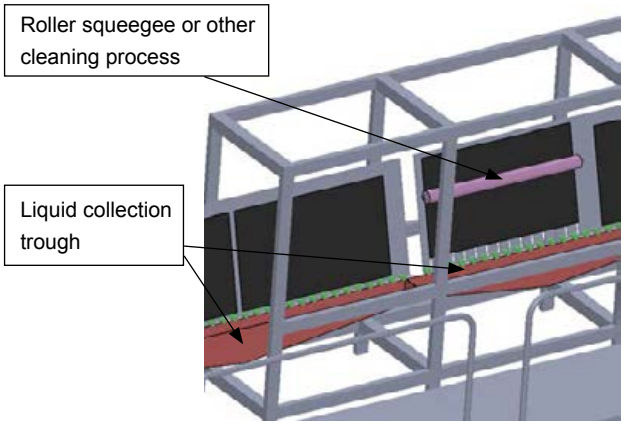


Figure 5.7. Liquid removal

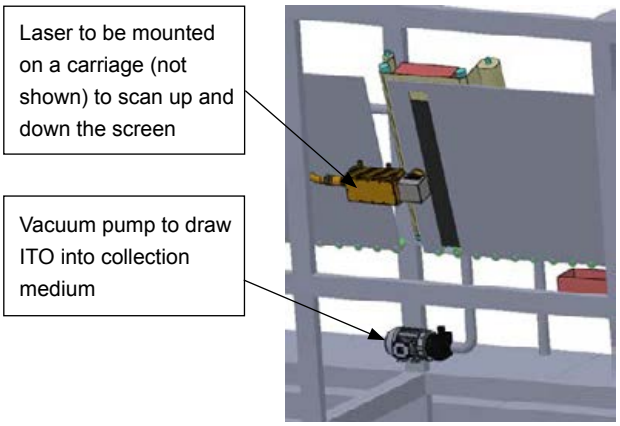


Figure 5.8. Laser for removing ITO.

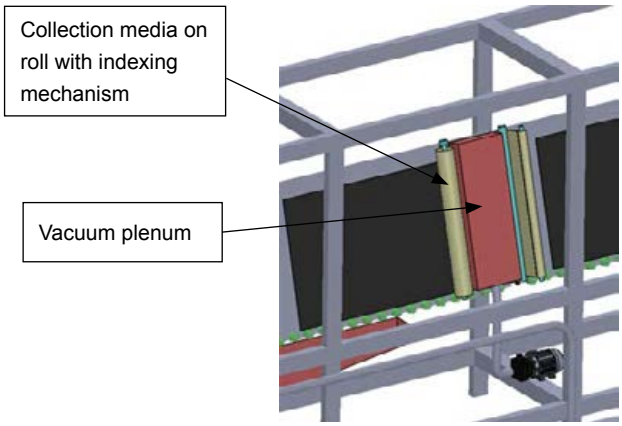


Figure 5.9. Collection of ITO

Once the roll of collection medium is filled to capacity with ITO, it is removed manually by the operator and packed for shipping to the indium processing site.

It may be possible to use a thin glass film to collect the ITO as the ITO adheres to a glass film after laser removal. This is a desirable method for the subsequent process of indium extraction.

An alternative collection method may be to use a commercially available or custom-designed dust collector with re-usable filtration media. This may have the advantage of requiring less input materials.

In the laser process, fumes are likely to be generated. This section of the equipment will be enclosed enough to allow for fume capture.

If a grinding/scouring process is employed, the glass layer will be held stationary while the grinding device (a cylindrical spinning abrasion tool) traverses downwards, similar to the roller squeegee at the previous step. A vacuum applied to the back of the polariser film will retain the glass layer integrity during the grinding/scouring step.

Automation of the surface grinding process would require design of a system for the collection of ITO.

Cleaned glass delivery

In this stage, the cleaned glass is driven automatically into an appropriate stillage for disposal (Figure 5.10). It is still attached to the polariser film, which is necessary for the steps prior to this to keep the glass together, as it is generally cracked and broken at the start of the process. Figure 5.10 illustrates the final cleaned glass fraction exiting the system into a container for storage or further processing.

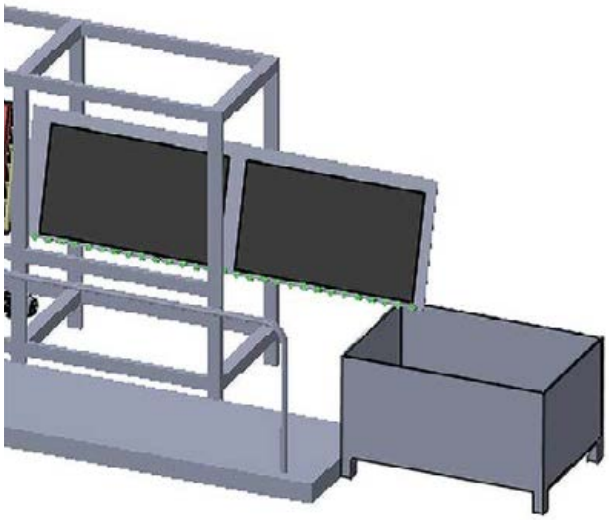


Figure 5.10. Cleaned glass delivery.

Table 5.5. Cost estimates for the laser or abrasion ITO removal processing machine

Item	Value	Units
Flat panel displays processed	240	Individual panels per hour (120 pairs per hour)
Running cost (energy)	€0.004	Per liquid crystal glass panel pair (€0.5 per hour)
Running cost (materials)	€0.01	Per liquid crystal glass panel pair

Table 5.5 details a cost estimate for the operation of an automated machine for the laser and/or abrasion process.

The main assumptions and key conclusions are summarised below:

- It should be possible to process 240 LCD panels per hour at a minimum. The laser process lends itself well to the potential for even higher throughputs. The laser is capable of processing a pair of panels in less than 10 seconds, potentially allowing up to 720 individual panels or 360 panel pairs to be processed per hour.
- The process limitation is likely to be determined by the capital cost required for multiple ITO removal stations.
- Materials costs are estimated based on what is required per screen processed so these will not change with throughput. Materials consumed are collection media and abrasives.
- Energy costs are assumed to be fixed and so these will reduce per screen if the rate is increased.
- Fixed overhead and depreciation costs are not included.
- Labour costs are not included as these will be influenced by the infeed set-up and whether or not this step would be automated. Manual separating and loading of panels, with an estimate of two people needed to run the machine, would add significantly to the operational cost of the process.
- It is believed that full automation with a high volume of feed stock would allow the process to be economically viable.

As the amount of ITO on each panel is very small, the commercial viability requires a high throughput process, considering the current indium prices. This is examined in the next chapter.

6 Economic Assessment and Further Work

6.1 The Amount and Price of Indium

The amount of indium within an LCD can range from 30 to 800 mg/kg of glass panel. The control samples of LCD glass panel used in these studies exhibited a maximum indium content of 550 mg/kg for the GF and 350 mg for the GB (using the as-is technique for a 4 cm^2 sample). For an average 32-inch LCD display (typical viewing area 2800 cm^2), the glass panel weight (both GF and GB) is approximately 1 kg. This means that there can be anywhere from 30 to 800 mg per device, depending on the make and model (and size) of the LCD panel.

The current price (in US dollars) of indium ranges from $190/kg to $300/kg. Using the higher price of indium of $300/kg, Table 6.1 shows the average value of indium within a device for both high and low indium concentrations per device.

From Table 6.1, it can be seen that 1250 LCD displays (at the higher indium concentration) would need to be recycled to generate 1 kg of indium with a maximum value of $300. However, the price of indium has varied greatly over time. At the time that this research project was proposed in 2015, the value of indium was closer to $800/kg. Figure 6.1 illustrates the variation in the price of indium over time.

It can be seen that the indium price varies significantly with the prevailing market conditions. The presentation by Indium Corporation concluded that the drive for recycling is the lack of availability of the metal. This is a critical point regarding indium, which is used as a transparent electrode not only in LCD displays but also in LED displays, organic light-emitting diode (OLED) displays and touch screens. Indium is a critical metal for the information and communication technology sector and the ability to recycle and capture indium from current products provides a safety net regarding market supply fluctuations. However, the challenge of recycling the metal cost-effectively, especially when the metal value is at a low ebb, is a critical aspect of meeting this challenge.

6.1.1 Economical assessment of the as-is technique

The research on the as-is samples, which represents the traditional approach to metal recycling via straightforward hydrometallurgy, showed that long cycling times are needed (25 hours to achieve maximal recovery), as well as concentrated acids in high ratios to the amount of indium (20 ml of acid for a 4 cm^2 sample) being recovered.

The acid volume and the long cycle time, as well as the generation of an acid by-product, results in high costs and low attractiveness of this traditional method. For example, scaling the volume of acid used per sample to that required for a full television, approximately 14 litres of acid at a cost of approximately €45 would be required to maintain the ratios used in the laboratory. However, aspects such as how quick the acid would saturate and the bath design needed to maximise the amount of display glass that could be submerged at once are critical. More research regarding the acid saturation points and lifespan should be undertaken. In addition, the fact that the traditional method has not been taken up by the market for recycling of indium further supports the unattractiveness of this approach. The use of ultrasonics has the potential to significantly reduce the cycle time (to approx. 15 minutes); however, ultrasonic effects on the volume and concentration of acid needed and the reusability of the acid still remains a critical challenge that needs further investigation.

Table 6.1. Indium content per device

Indium per device (mg)	Indium per device (kg)	Value ($) per device (at $300/kg)
800 (max.)	0.0008	0.24
30 (min.)	0.00003	0.009

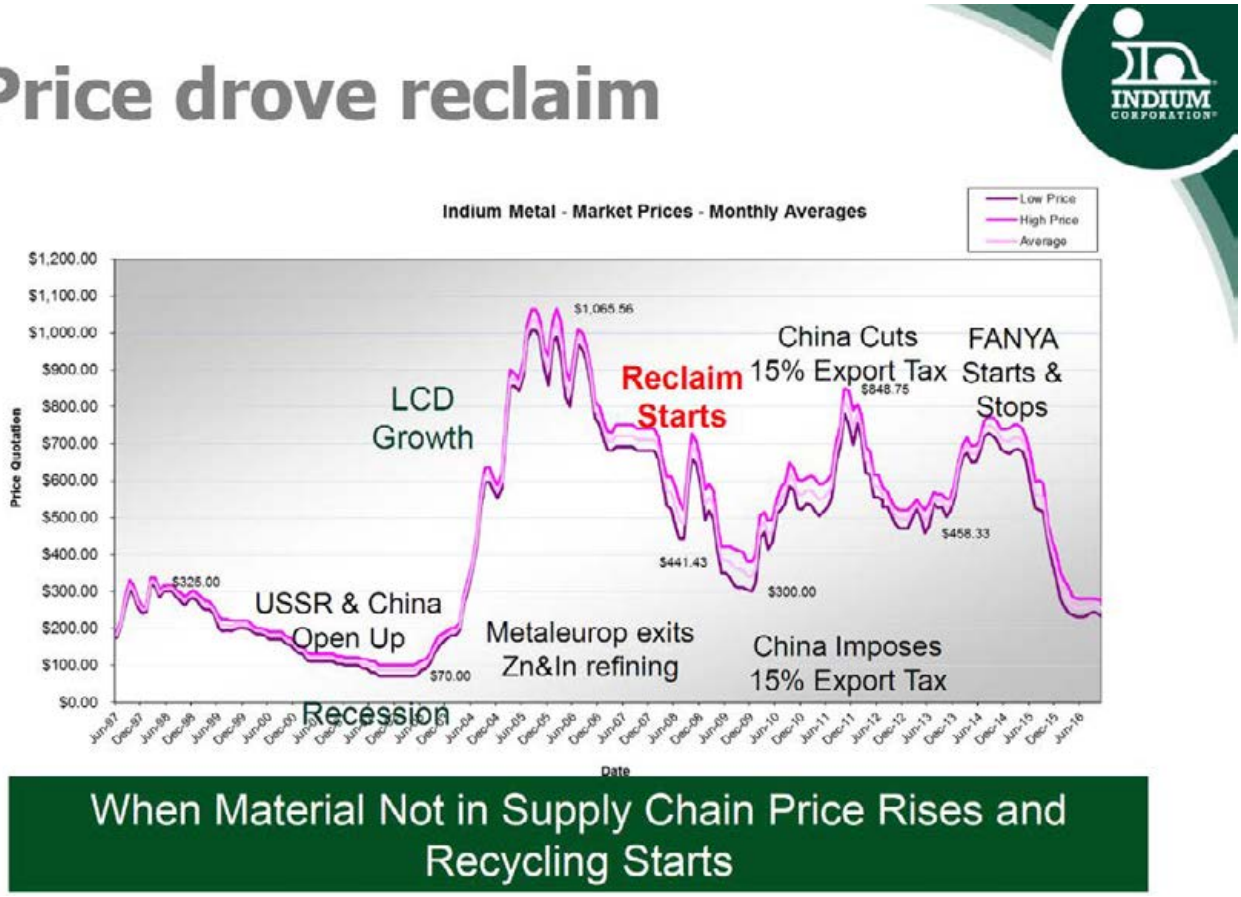


Figure 6.1. Variation in the price of indium over time. Source: "ITO history and recycling of critical metal Indium" presentation at IERC Congress, Salzburg, 18 January 2018, by Donna Vareha Walsh, Indium Corporation.

The use of automation to manipulate glass panels into an ultrasonic bath would not alleviate the cost of the acid required, the lifespan of the acid or the acid disposal costs. The polariser coatings on the panel may act as a bath contaminant and may require removal in advance. The cycle time is dependent on the required dwell time in the bath, which becomes a limiting factor in terms of the throughput of an automated process for this technique. In addition, the cost of automating such a process (€0.5 million minimum) would put a capital expenditure (capex) burden on top of the economic challenges already faced by the operational expenditure (opex) of the process. Therefore, having such a facility available at each local recycling plant would be prohibitive, meaning that a centralised facility would be required where LCD glass panels would need to be delivered, adding a significant cost of transportation per tonne of glass panel (including the difficulty involved in handling the glass panels). It is clear that the traditional hydrometallurgy process has significant drawbacks for indium recovery; however, it is useful as a baseline technique for comparison of the performance of alternative techniques.

6.1.2 Alternative techniques

The challenges involved in using traditional hydrometallurgical techniques directly on the glass panels led to an approach in which efficient physical separation of the ITO is the first step (which could potentially be performed locally at a recycling facility), followed by recovery of indium from ITO (which could potentially be performed centrally). Bulk grinding, surface grinding and laser ablation were investigated to physically separate the ITO from the glass panels. The focus here was to liberate the ITO in a high throughput process, free from contaminants, generating a fraction that could readily and cost-effectively be sent to a centralised specialist facility for recovery. The economics of the recovery step will depend heavily on the characteristics of the ITO fraction generated from the physical separation step.

6.1.3 Economic assessment of the bulk grinding technique

The bulk grinding technique produced three powder fractions, with one fraction containing the majority of the ITO (53 µm particle size). In this case, a 100 cm^2 (15 square inches) sample of glass panel weighing 17,000 mg generated approximately 1050 mg of ITO powder for the GF and 1430 mg of ITO powder for the GB. These powders had approximately 850 mg and 650 mg of indium/kg of powder. Scaling these values for the GF panel of a full 32-inch display (435.5 square inch glass panel) would yield 31,535 mg (0.031 kg) of ITO powder. This amount of ITO powder would contain approximately 26 mg of indium.

The lower yields of indium could be for a number of reasons, including loss of indium to the other sieved fractions after the bulk grinding process, that ITO powder (morphology, particle size or characteristic) does not lend itself well to digestion at the hydrometallurgical recovery step and the presence of other materials from dyes, glass and organic coatings, which were also ground during the grinding process. Further research should be carried out to investigate the potential of different sieving or segregation techniques and the effect of using different grinding media on the powder morphology.

The ease of automation of this technique, whereby a simple milling process could be installed cheaply at recycling facilities and powder transported efficiently to a centralised recovery plant, makes this an attractive process; however, the low yield currently renders this technique unattractive.

6.1.4 Economic assessment of the surface grinding technique

The surface grinding technique generated a powder from the GF with a yield of 4500 mg of indium/kg powder. The high yield per kilogram of powder is attractive for the downstream metallurgy process, with a more concentrated indium powder being more efficient for recovery. A 100 cm^2 (15 square inches) glass panel sample generated 60 mg of powder. Scaling this up for a 32-inch display (or viewing area 435.5 square inches) generates 1742 mg (0.0017 kg) of powder. The high concentration of indium in the powder (although a low amount of powder) results in the surface grinding technique being attractive from the downstream recovery aspect. However, the process was not effective for the GB of the glass panel, which is a major drawback. In addition, particles of the abrasive material acting as a contaminant are an issue and research regarding subsequent hydrometallurgy regarding bath-friendly grinding media would be required.

Automation of the process must deal with the glass panels being intact, meaning that there will be significant capital equipment costs; however, the fact that removal of the polariser is not required is a benefit. Although the high yield of the GF suggests that there is potential for this technique, further research would be required to increase the yield from the GB and optimise the selection of the grinding media for the following recovery step. In addition, the speed of the grinding step is a limiting factor in terms of process throughput. This will be dependent on the size and type of grinding media used to remove ITO from an entire panel. The length of time needed to manually surface grind one panel in the laboratory was approximately 1 minute (45 seconds to 1 minute depending on the rpm used); although this could be automated, it may still take a significant length of time. Additional grinding heads can be used on an automated set-up; however, this would add significantly to the cost.

6.1.5 Economic assessment of the laser ablation technique

The ITO separated by the laser technique from the GF showed the highest indium values when deposited and recovered on a glass medium, generating up to 900 mg/m^2 of glass panel. Scaling this up for a 32-inch display of viewing area 450.5 square inches, it can be seen that 261.5 mg of indium would be recovered for the GF. The laser results illustrate that the laser technique has the capability of recovering indium. It is thought that any indium losses are the result of not being able to capture all of the ITO vapour onto the media. It was found during testing that ITO coated the inside of the capture unit as well as the glass media.

Automation of the process could compensate for this by using vacuum-assisted vapour extraction directed or funneled towards the capture media. The process lends itself well to automation with regard to the laser set-up. Further research would be required regarding the capture unit design and optimisation of the capture

media to be used, especially considering downstream hydrometallurgy recovery. Cotton, tissue and filter paper would disintegrate in an acid bath and build up as a contaminant. However, there is the potential for a bath design that could simply remove the sludge. In addition, using a solid capture medium such as a thin sheet of glass, the indium could be deposited indefinitely from multiple samples. Such deposition offers many benefits, including increasing the concentration of indium on the medium and therefore reducing the ratio of media to indium being introduced into the recovery bath.

Automation of this process has the capability to yield the highest throughput of all of the techniques as the laser processing time is measured in seconds. Further fine-tuning of the laser parameters and laser speed is suggested as an area for further research. In Chapter 5, it was proposed that 120 glass panels or 240 individual glass sheets (i.e. GF and GB) could be processed per hour. With the assumption that 800 mg of indium is available per panel, this could liberate 0.096 kg/hour of ITO powder, with a value of $28.8, assuming an indium value of $300/kg. In the event that the metal markets fluctuate to previous values ($800/kg), the hourly throughput of the laser technique could potentially yield a fraction with an indium value of $76.8 per hour.

A high process throughput is critical to achieving the economics required for the indium recovery process. The laser technique is believed to have the greatest potential for a high throughput process that could be utilised to locally capture ITO on a medium that can then be processed at a centralised facility using hydrometallurgy. In addition, the option to utilise different capture media offers many downstream advantages for the hydrometallurgy recovery step. From this study, it is believed that the laser processing technique has strong potential regarding large-scale indium recovery.

7 Summary and Conclusions

Below is a summary of the key findings of the research and the conclusions drawn:

- Analysis of the indium concentrations on the LCD panels showed large variations, from 38 to 800 mg/kg of glass. The GF generally contained more indium than the GB.
- A series of pre-treatments were investigated to physically remove and separate the indium-containing ITO from the LCDs. These included acid digestion in as-is condition, bulk grinding of the panel and sieving to produce different particle sizes, surface grinding and finally laser ablation of the ITO.
- From these, the best-performing pre-treatments were considered to be the surface grinding and the laser ablation processes as both removed ITO directly with minimal contaminants being collected.
- The surface grinding process yielded a powder that predominantly consisted of ITO and this has an advantage of introducing fewer contaminants to the acid bath than the bulk grinding technique at the hydrometallurgical step. However, the chosen grinding media used may also act as a bath contaminant.
- Although the laser process has the potential to be a very high throughput process, the ITO has to be captured on an absorption medium, which subsequently must be submerged into an acid bath and will act as an undesirable constituent. However, the ability to deposit or capture the ITO on a bath-friendly medium (i.e. a medium that would not act as a bath contaminant) offers huge potential for this process.
- The potential to automate both the laser and the surface grinding techniques was considered in detail. Similar process set-ups would be required by both. The advantage of automating these processes is that the polariser film of the panel does not require removal. This has a significant impact on the time and quality of the process compared with the other options available (“as-is” digestion and bulk grinding).
- The laser technique has the potential to achieve very high process throughputs; a minimum of approximately 240 individual panels or 120 panel pairs can be processed per hour.
- The amount of indium that can be collected with one laser is approximately 90 g/hour. This could be increased by using multiple lasers to scale the process up for fully automated closed-loop indium recycling from LCDs.

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Abbreviations

AR	Aqua regia
CCFL	Cold cathode fluorescent lamp
CRM	Critical raw material
EDX	Energy-dispersive X-ray spectroscopy
GB	Glass back
GF	Glass front
ICP-OES	Inductively coupled plasma optical emission spectrometry
ITO	Indium tin oxide
LCD	Liquid crystal display
Le	Leaching
LED	Light-emitting diode
MW	Metal wool
SEM	Scanning electron microscopy
SiC	Silicon carbide
US	Ultrasound

AN GHNÍOMHAIREACHT UM CHAOMHNÚ COMHSHAOIL
Tá an Ghníomhaireacht um Chaomhnú Comhshaoil (GCC) freagrach as an gcomhshaol a chaomhnú agus a fheabhsú mar shócmhainn luachmhar do mhuintir na hÉireann. Táimid tiomanta do dhaoine agus don chomhshaol a chosaint ó éifeachtaí díobhálacha na radaíochta agus an truaillithe.

Is féidir obair na Gníomhaireachta a roinnt ina trí phríomhréimse:

Rialú: *Déanaimid córais éifeachtacha rialaithe agus comhlíonta comhshaoil a chur i bhfeidhm chun torthaí maithe comhshaoil a sholáthar agus chun díriú orthu siúd nach gcloíonn leis na córais sin.*

Eolas: *Soláthraímid sonraí, faisnéis agus measúnú comhshaoil atá ar ardchaighdeán, spriocdhírithe agus tráthúil chun bonn eolais a chur faoin gcinnteoireacht ar gach leibhéal.*

Tacaíocht: *Bímid ag saothrú i gcomhar le grúpaí eile chun tacú le comhshaol atá glan, táirgiúil agus cosanta go maith, agus le hiompar a chuirfidh le comhshaol inbhuanaithe.*

Ár bhFreagrachtaí

Ceadúnú
Déanaimid na gníomhaíochtaí seo a leanas a rialú ionas nach ndéanann siad dochar do shláinte an phobail ná don chomhshaol:
- saoráidí dramhaíola *(m.sh. láithreáin líonta talún, loisceoirí, stáisiúin aistrithe dramhaíola)*;
- gníomhaíochtaí tionsclaíocha ar scála mór *(m.sh. déantúsaíocht cógaisíochta, déantúsaíocht stroighne, stáisiúin chumhachta)*;
- an diantalmhaíocht *(m.sh. muca, éanlaith)*;
- úsáid shrianta agus scaoileadh rialaithe Orgánach Géinmhodhnaithe *(OGM)*;
- foinsí radaíochta ianúcháin *(m.sh. trealamh x-gha agus radaiteiripe, foinsí tionsclaíocha)*;
- áiseanna móra stórála peitril;
- scardadh dramhuisce;
- gníomhaíochtaí dumpála ar farraige.

Forfheidhmiú Náisiúnta i leith Cúrsaí Comhshaoil
- Clár náisiúnta iniúchtaí agus cigireachtaí a dhéanamh gach bliain ar shaoráidí a bhfuil ceadúnas ón nGníomhaireacht acu.
- Maoirseacht a dhéanamh ar fhreagrachtaí cosanta comhshaoil na n-údarás áitiúil.
- Caighdeán an uisce óil, arna sholáthar ag soláthraithe uisce phoiblí, a mhaoirsiú.
- Obair le húdaráis áitiúla agus le gníomhaireachtaí eile chun dul i ngleic le coireanna comhshaoil trí chomhordú a dhéanamh ar líonra forfheidhmiúcháin náisiúnta, trí dhíriú ar chiontóirí, agus trí mhaoirsiú a dhéanamh ar leasúchán.
- Cur i bhfeidhm rialachán ar nós na Rialachán um Dhramhthrealamh Leictreach agus Leictreonach (DTLL), um Shrian ar Shubstaintí Guaiseacha agus na Rialachán um rialú ar shubstaintí a ídíonn an ciseal ózóin.
- An dlí a chur orthu siúd a bhriseann dlí an chomhshaoil agus a dhéanann dochar don chomhshaol.

Bainistíocht Uisce
- Monatóireacht agus tuairisciú a dhéanamh ar cháilíocht aibhneacha, lochanna, uiscí idirchriosacha agus cósta na hÉireann, agus screamhuiscí; leibhéil uisce agus sruthanna aibhneacha a thomhas.
- Comhordú náisiúnta agus maoirsiú a dhéanamh ar an gCreat-Treoir Uisce.
- Monatóireacht agus tuairisciú a dhéanamh ar Cháilíocht an Uisce Snámha.

Monatóireacht, Anailís agus Tuairisciú ar an gComhshaol
- Monatóireacht a dhéanamh ar cháilíocht an aeir agus Treoir an AE maidir le hAer Glan don Eoraip (CAFÉ) a chur chun feidhme.
- Tuairisciú neamhspleách le cabhrú le cinnteoireacht an rialtais náisiúnta agus na n-údarás áitiúil *(m.sh. tuairisciú tréimhsiúil ar staid Chomhshaol na hÉireann agus Tuarascálacha ar Tháscairí)*.

Rialú Astaíochtaí na nGás Ceaptha Teasa in Éirinn
- Fardail agus réamh-mheastacháin na hÉireann maidir le gáis cheaptha teasa a ullmhú.
- An Treoir maidir le Trádáil Astaíochtaí a chur chun feidhme i gcomhair breis agus 100 de na táirgeoirí dé-ocsaíde carbóin is mó in Éirinn.

Taighde agus Forbairt Comhshaoil
- Taighde comhshaoil a chistiú chun brúnna a shainaithint, bonn eolais a chur faoi bheartais, agus réitigh a sholáthar i réimsí na haeráide, an uisce agus na hinbhuanaitheachta.

Measúnacht Straitéiseach Timpeallachta
- Measúnacht a dhéanamh ar thionchar pleananna agus clár beartaithe ar an gcomhshaol in Éirinn *(m.sh. mórphleananna forbartha)*.

Cosaint Raideolaíoch
- Monatóireacht a dhéanamh ar leibhéil radaíochta, measúnacht a dhéanamh ar nochtadh mhuintir na hÉireann don radaíocht ianúcháin.
- Cabhrú le pleananna náisiúnta a fhorbairt le haghaidh éigeandálaí ag eascairt as taismí núicléacha.
- Monatóireacht a dhéanamh ar fhorbairtí thar lear a bhaineann le saoráidí núicléacha agus leis an tsábháilteacht raideolaíochta.
- Sainseirbhísí cosanta ar an radaíocht a sholáthar, nó maoirsiú a dhéanamh ar sholáthar na seirbhísí sin.

Treoir, Faisnéis Inrochtana agus Oideachas
- Comhairle agus treoir a chur ar fáil d'earnáil na tionsclaíochta agus don phobal maidir le hábhair a bhaineann le caomhnú an chomhshaoil agus leis an gcosaint raideolaíoch.
- Faisnéis thráthúil ar an gcomhshaol ar a bhfuil fáil éasca a chur ar fáil chun rannpháirtíocht an phobail a spreagadh sa chinnteoireacht i ndáil leis an gcomhshaol *(m.sh. Timpeall an Tí, léarscáileanna radóin)*.
- Comhairle a chur ar fáil don Rialtas maidir le hábhair a bhaineann leis an tsábháilteacht raideolaíoch agus le cúrsaí práinnfhreagartha.
- Plean Náisiúnta Bainistíochta Dramhaíola Guaisí a fhorbairt chun dramhaíl ghuaiseach a chosc agus a bhainistiú.

Múscailt Feasachta agus Athrú Iompraíochta
- Feasacht chomhshaoil níos fearr a ghiniúint agus dul i bhfeidhm ar athrú iompraíochta dearfach trí thacú le gnóthais, le pobail agus le teaghlaigh a bheith níos éifeachtúla ar acmhainní.
- Tástáil le haghaidh radóin a chur chun cinn i dtithe agus in ionaid oibre, agus gníomhartha leasúcháin a spreagadh nuair is gá.

Bainistíocht agus struchtúr na Gníomhaireachta um Chaomhnú Comhshaoil
Tá an ghníomhaíocht á bainistiú ag Bord lánaimseartha, ar a bhfuil Ard-Stiúrthóir agus cúigear Stiúrthóirí. Déantar an obair ar fud cúig cinn d'Oifigí:
- An Oifig um Inmharthanacht Comhshaoil
- An Oifig Forfheidhmithe i leith cúrsaí Comhshaoil
- An Oifig um Fianaise is Measúnú
- Oifig um Chosaint Radaíochta agus Monatóireachta Comhshaoil
- An Oifig Cumarsáide agus Seirbhísí Corparáideacha

Tá Coiste Comhairleach ag an nGníomhaireacht le cabhrú léi. Tá dáréag comhaltaí air agus tagann siad le chéile go rialta le plé a dhéanamh ar ábhair imní agus le comhairle a chur ar an mBord.

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Investigation of Indium Recovery from End-of-life LCDs

Authors: Chinnam Rama Krishna, John Mulcahy and Lisa O'Donoghue

Identifying Pressures

The LCD Val project was focused on the potential for recycling and recovery of indium from waste liquid crystal displays (LCDs). Once the liquid crystal glass panel that contains indium is removed from LCDs at recycling facilities, the only available solution is to incinerate or export it. The LCD Val project focused on researching potential options for the recycling of this component to recover indium and generate a clean glass fraction, thereby turning waste into a resource.

Informing Policy

The data and information generated from the project include verification of the quantities of indium present in different LCDs arising in Ireland, testing of techniques to recover indium that are compatible with Irish recycling infrastructure, economic assessment of these techniques and selection of a best available technique tailored to Ireland's waste sector, value chain and economy. These results provide valuable data regarding Ireland's recycling challenges and opportunities and this information is available to inform policy.

Developing Solutions

The LCD Val project involved testing of hydrometallurgical treatment processes for the recovery of indium. Subsequently, a unique methodological approach was undertaken in which a series of pre-treatment processes were developed to up-concentrate indium within the waste fraction prior to the hydrometallurgical step, thereby reducing the cost and operational aspects of the overall process. A series of surface grinding, bulk grinding and laser processing techniques were tested and the laser/surface grinding technique was selected as the best-performing technique, with strong industrial applicability. A scale-up of the process for potential Irish deployment was modelled and presented in the key findings.

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