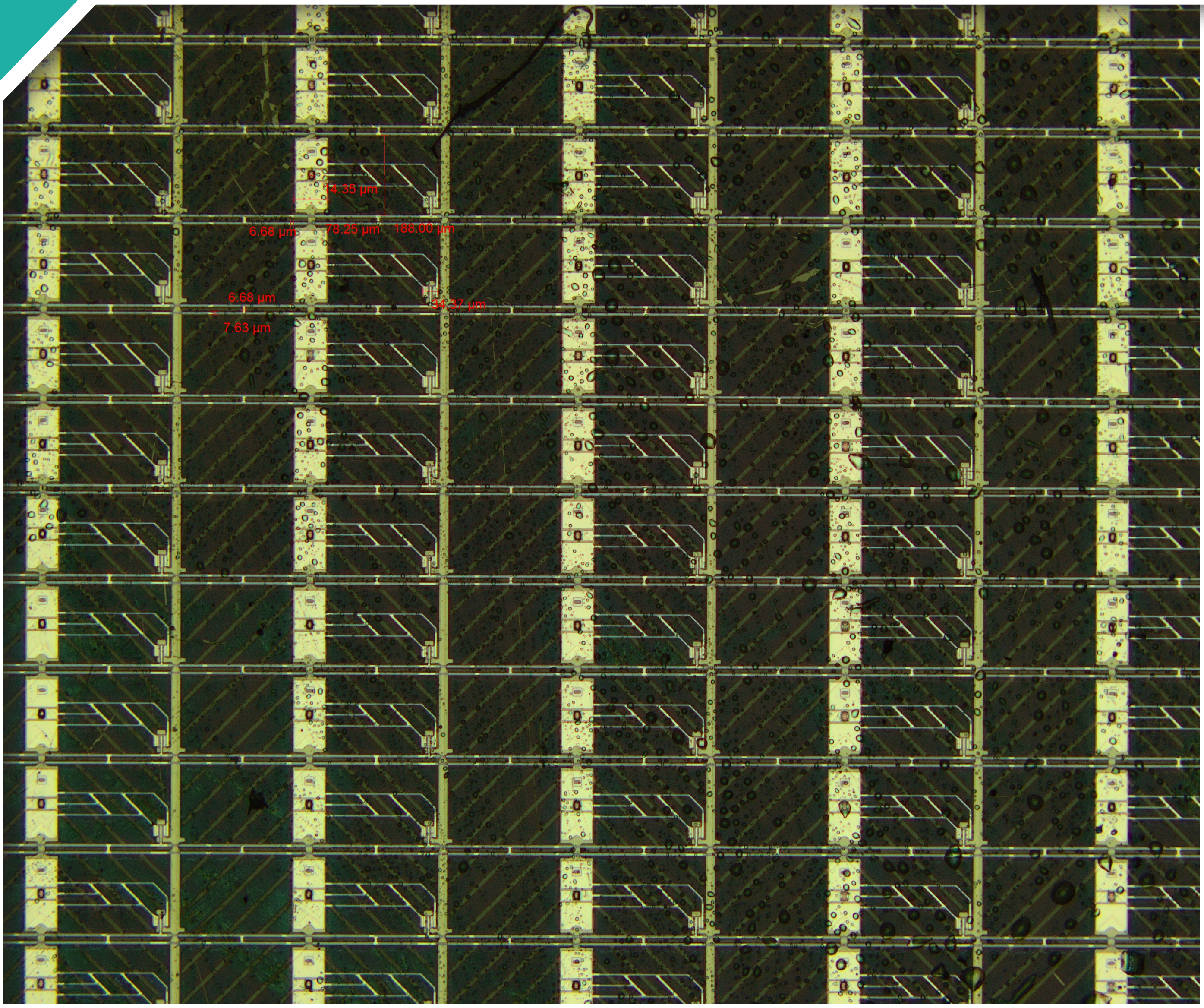
Report No. 201

epa Research
Climate - Water - Sustainability
Identifying pressures • Informing policy • Developing solutions

Investigation of Liquid Crystal Displays as a Source of Indium

Authors: Lisa O'Donoghue and Paul Moroney



Roinn Cumarsáide, Gníomhaithe
ar son na hAeráide & Comhshaoil
Department of Communications,
Climate Action & Environment

www.epa.ie

ENVIRONMENTAL PROTECTION AGENCY
The Environmental Protection Agency (EPA) is responsible for protecting and improving the environment as a valuable asset for the people of Ireland. We are committed to protecting people and the environment from the harmful effects of radiation and pollution.

The work of the EPA can be divided into three main areas:

Regulation: *We implement effective regulation and environmental compliance systems to deliver good environmental outcomes and target those who don't comply.*

Knowledge: *We provide high quality, targeted and timely environmental data, information and assessment to inform decision making at all levels.*

Advocacy: *We work with others to advocate for a clean, productive and well protected environment and for sustainable environmental behaviour.*

Our Responsibilities

Licensing

We regulate the following activities so that they do not endanger human health or harm the environment:

- waste facilities *(e.g. landfills, incinerators, waste transfer stations)*;
- large scale industrial activities (*e.g. pharmaceutical, cement manufacturing, power plants)*;
- intensive agriculture *(e.g. pigs, poultry)*;
- the contained use and controlled release of Genetically Modified Organisms *(GMOs)*;
- sources of ionising radiation *(e.g. x-ray and radiotherapy equipment, industrial sources)*;
- large petrol storage facilities;
- waste water discharges;
- dumping at sea activities.

National Environmental Enforcement

- Conducting an annual programme of audits and inspections of EPA licensed facilities.
- Overseeing local authorities' environmental protection responsibilities.
- Supervising the supply of drinking water by public water suppliers.
- Working with local authorities and other agencies to tackle environmental crime by co-ordinating a national enforcement network, targeting offenders and overseeing remediation.
- Enforcing Regulations such as Waste Electrical and Electronic Equipment (WEEE), Restriction of Hazardous Substances (RoHS) and substances that deplete the ozone layer.
- Prosecuting those who flout environmental law and damage the environment.

Water Management

- Monitoring and reporting on the quality of rivers, lakes, transitional and coastal waters of Ireland and groundwaters; measuring water levels and river flows.
- National coordination and oversight of the Water Framework Directive.
- Monitoring and reporting on Bathing Water Quality.

Monitoring, Analysing and Reporting on the Environment

- Monitoring air quality and implementing the EU Clean Air for Europe (CAFÉ) Directive.
- Independent reporting to inform decision making by national and local government *(e.g. periodic reporting on the State of Ireland's Environment and Indicator Reports)*.

Regulating Ireland's Greenhouse Gas Emissions

- Preparing Ireland's greenhouse gas inventories and projections.
- Implementing the Emissions Trading Directive, for over 100 of the largest producers of carbon dioxide in Ireland.

Environmental Research and Development

- Funding environmental research to identify pressures, inform policy and provide solutions in the areas of climate, water and sustainability.

Strategic Environmental Assessment

- Assessing the impact of proposed plans and programmes on the Irish environment *(e.g. major development plans)*.

Radiological Protection

- Monitoring radiation levels, assessing exposure of people in Ireland to ionising radiation.
- Assisting in developing national plans for emergencies arising from nuclear accidents.
- Monitoring developments abroad relating to nuclear installations and radiological safety.
- Providing, or overseeing the provision of, specialist radiation protection services.

Guidance, Accessible Information and Education

- Providing advice and guidance to industry and the public on environmental and radiological protection topics.
- Providing timely and easily accessible environmental information to encourage public participation in environmental decision-making *(e.g. My Local Environment, Radon Maps)*.
- Advising Government on matters relating to radiological safety and emergency response.
- Developing a National Hazardous Waste Management Plan to prevent and manage hazardous waste.

Awareness Raising and Behavioural Change

- Generating greater environmental awareness and influencing positive behavioural change by supporting businesses, communities and householders to become more resource efficient.
- Promoting radon testing in homes and workplaces and encouraging remediation where necessary.

Management and structure of the EPA

The EPA is managed by a full time Board, consisting of a Director General and five Directors. The work is carried out across five Offices:

- Office of Environmental Sustainability
- Office of Environmental Enforcement
- Office of Evidence and Assessment
- Office of Radiation Protection and Environmental Monitoring
- Office of Communications and Corporate Services

The EPA is assisted by an Advisory Committee of twelve members who meet regularly to discuss issues of concern and provide advice to the Board.

EPA RESEARCH PROGRAMME 2014–2020

Investigation of Liquid Crystal Displays as a Source of Indium

[2008-WRM-MS-5 (Extension)]

EPA Research Report

Prepared for the Environmental Protection Agency

by

University of Limerick

Authors:

Lisa O’Donoghue and Paul Moroney

Contributors’ lab research and industry surveys:

Paul Moroney, John Mulcahy, William Harrison and Ava Maloney

ENVIRONMENTAL PROTECTION AGENCY
An Ghníomhaireacht um Chaomhnú Comhshaoil
PO Box 3000, Johnstown Castle, Co. Wexford, Ireland

Telephone: +353 53 916 0600 Fax: +353 53 916 0699
Email: info@epa.ie Website: www.epa.ie

ACKNOWLEDGEMENTS

This report is published as part of the EPA Research Programme 2014–2020. The programme is financed by the Irish Government. It is administered on behalf of the Department of Communications, Climate Action and Environment (DCCAE) by the EPA, which has the statutory function of co-ordinating and promoting environmental research.

The authors would like to thank the members of the project steering committee for their support, including Dr Brian Donlon and Martin Doyle, EPA; Darren Byrne, DCCAE; Kurt Kyck, KMK Metals; John Cuddy, Irish Lamps; and Leo Donovan, WEEE Ireland.

DISCLAIMER

Although every effort has been made to ensure the accuracy of the material contained in this publication, complete accuracy cannot be guaranteed. The Environmental Protection Agency, the authors and the steering committee members do not accept any responsibility whatsoever for loss or damage occasioned, or claimed to have been occasioned, in part or in full, as a consequence of any person acting, or refraining from acting, as a result of a matter contained in this publication. All or part of this publication may be reproduced without further permission, providing the source is acknowledged.

The EPA Research Programme addresses the need for research in Ireland to inform policymakers and other stakeholders on a range of questions in relation to environmental protection. These reports are intended as contributions to the necessary debate on the protection of the environment.

COVER IMAGE

A surface optical image of the internal surface of a liquid crystal panel with the indium containing ITO layer

EPA RESEARCH PROGRAMME 2014–2020
Published by the Environmental Protection Agency, Ireland

ISBN: 978-1-84095-681-8 August 2017

Price: Free Online version

Project Partners

Lisa O'Donoghue
University of Limerick
Design and Manufacturing Technology
Department
Plassey
Limerick
Ireland
Tel.: +353 61 202910
Email: lisa.odonoghue@ul.ie

Paul Moroney
University of Limerick
Design and Manufacturing Technology
Department
Plassey
Limerick
Ireland

William Harrison
University of Limerick
Design and Manufacturing Technology
Department
Plassey
Limerick
Ireland

Contents

List of Figures

List of Tables

Executive Summary

The objectives of the research were to investigate and explore the potential for indium recovery from liquid crystal displays (LCDs) and specifically to:

- investigate the presence and amount of indium contained in the liquid crystal panels of LCDs;
- review potential methodologies and best available techniques for the recovery of indium from LCDs;
- review the requirement for indium recovery from an industrial perspective;
- review the requirement for LCD recycling from a policy perspective.

To investigate the presence and amount of indium contained in the liquid crystal panels of LCDs, samples of liquid crystal panels from 10 different LCD TVs were selected and prepared for examination by various microscopic and spectroscopic techniques. The findings from the 10 samples tested revealed that indium content varied significantly from 38 to 292 mg indium per kg glass substrate where the liquid crystal panel was made up of two of these substrates (namely glass front and glass back substrates). Generally, it was found that the glass front substrate contained higher concentrations of indium than the glass back substrate, correlating with a uniform electrode layer and an intermittent electrode layer, respectively. Optical analysis revealed different surface structures of the electrode coatings where transparent electrodes exhibited either rectangular cell or V-cell structures. It was found that rectangular cell structures generally contained higher indium concentrations and were the most common structure in the samples examined. Other elements were regularly present including aluminium, silicon, molybdenum and titanium. Understanding the trend in the variation of the LCDs and their indium content allows the recycler to make informed decisions regarding a metal recovery process. The potential to correlate the cell structure (using a simple optical microscope or X-ray) with the indium level could be a fast and cheap process-control method to separate out panels with high indium concentrations.

Regarding the potential methodologies and best available techniques for the recovery of indium from LCDs, a detailed review of literature, industry articles and patents was undertaken. While some new plants in France, Spain and Asia have been reported to be undertaking indium recovery, information regarding processing is not available. Hydrometallurgy is a well-known and traditional route used to recover metal, with the predominant techniques for indium recovery focused on solvent extraction and ion exchange. These technologies are usually operated at scale but require optimisation regarding the recovery of indium from LCDs to deal with low indium concentrations, the presence of other metals and best pre-processing steps. Novel techniques have also been reported, such as the lift-off method, the sub-critical water process, biotreaments and mechanochemical treatments. However, these are mainly at the laboratory-scale testing phase and are not reported as being deployed commercially, highlighting a gap between laboratory and commercial scale-up of the recovery process options reported. The review has highlighted both the interest in indium from a research and industrial perspective and also that finding an economical process to recover indium at scale is still required. The influence of the business case on the technology is a significant facet of rolling out a large-scale recovery plant, and this aspect requires future development.

Regarding the requirement for indium recovery from an industrial perspective, a detailed survey of relevant industrial organisations was undertaken including producers, recyclers and associations across the EU. All organisations believed that indium recovery operations would have an impact on their businesses; however, whether this would be a positive or negative impact would be determined by the costs and benefits of the particular indium recovery methodology being used. The key parameters that would influence indium recovery were predominantly cost of recovery and purity of indium obtained, as well as having an adequate value chain in which manufacturers would be willing to purchase the recycled indium. When asked about current industrial drivers for indium recovery, all participant organisations indicated that cost of recovery was key and that the price of the indium from the recovery process must compare well with the price of indium from traditional sources on the market. Other driving forces were ease of recycling and the quantity of indium that the process could produce. Regarding the upscaling challenges for indium recovery, from a technical perspective, feedback focused on purity of indium and the low levels of indium in the LCDs. When asked about the future requirement for indium in products, most participant organisations generally agreed that there is a future requirement, as indium is used in a large number of products now and will continue to be used for the foreseeable future.

Any process development or technology scale-up for indium recovery should focus on the key parameters highlighted in this report: technical aspects (purity and low-concentration indium recovery) and commercial aspects (cost of recovery and acceptance of recovered indium as a product for the end user, i.e. the manufacturer). Therefore, regarding the technical aspects, processes should be designed utilising a knowledge bank to record the presence of other elements and indium concentration expected per batch. In addition, using pre-processing steps ensures a minimum indium concentration per batch of panels to be processed. Regarding the commercial aspects, control samples to compare and contrast recovery indium with traditionally sourced indium are important, as well as engaging the end purchasers regarding quantities of indium required, characterisations of the product and any other factors that they would consider critical when potentially using recovered indium as a supply.

Regarding the requirement for LCD recycling from a policy perspective, both primary and secondary research were undertaken to understand the relevant policies and standards affecting LCD recycling operations specifically within the Irish context. The WEEELABEX (WEEE Label of excellence) standard is an important industry standard and is developing threshold values for mercury after the recycling process for flat panel displays. The standard requires that consideration should be given to the indium tin oxide fraction of the liquid crystal panel; however, this does not require mandatory recovery. These thresholds are currently under review and it is expected that the standard will be completed along with its translation into a European Committee for Electrotechnical Standardization (CENELEC) standard before the end of 2016. Regarding both LCD recycling and indium recovery, it has been clear that engagement of multiple stakeholders along the value chain is required, and not just by the recycler, in order to achieve change or a step forward in terms of processing capability. The drivers for implementation of technologies usually lie with the end user, in this case a complex chain from recyclers and smelters to manufacturers (and potential metal traders in between). The recommendation regarding policy is the continued engagement of Irish industry and stakeholders with European policy development as well as increasing engagement with connections and network opportunities such as the European Innovation Partners (WEEE2020) and Raw Materials Knowledge and Innovation Community, all of which incorporate a focus on electronic recycling.

1 Introduction

1.1 Background

Liquid crystal displays (LCDs) are used in applications ranging from small displays on machines up to 100-inch TV screens. Global LCD sales were estimated at over 214 million units in 2015 alone (Statista, 2015). Given that many LCDs have a short lifespan (average 6–8 years), a large number of LCDs are made redundant each year and require appropriate end-of-life recycling. In Ireland, 4202 tonnes of TVs and monitors were collected in 2014 with LCD units representing approximately 16,000 units (WEEE Ireland, 2014).

The European Directive for Waste Electrical and Electronic Equipment [(WEEE) Directive 2012/19/EU], which all EU Member States are required to implement, stipulates that components containing mercury (cold cathode fluorescent tubes used to illuminate an LCD screen) and liquid crystals (liquid crystal panel used to produce the image) must be removed from waste LCDs. The majority of recyclers use a manual disassembly process to remove the cold cathode fluorescent lighting (CCFL) tubes and liquid crystal panels. The liquid crystal panels, once separated from the LCDs, are either stockpiled at recycling facilities or sent for disposal by incineration.

1.2 Liquid Crystal Panels

The liquid crystal panel is a subsection of the LCD display consisting of two glass substrates with liquid crystals in between. One of the glass substrates incorporates a regular alternating pattern of red, green and blue picture elements (pixels) formed using dyed colour filters. Light from the rear of the LCD is passed through to the pixels illuminating specific pixels in a pattern to form the image on the display. Liquid crystals are used to control the passage of light to the pixels and hence control the image formation. Liquid crystals perform these functions by rotating their position at the molecular level; in one orientation, they allow light to pass while in an alternate orientation, they block the light. The orientation of the liquid crystals can be controlled and manipulated by the application of electricity to the liquid crystals. An electrode is used to apply the electricity to the liquid crystals and is made of a transparent conducting material, usually indium tin oxide (ITO), which is deposited on the inner surface of the two glass substrates parallel to the liquid crystals.

In some cases, a thin passivation layer (silica or polymer) intended to prevent diffusion of ions into the liquid crystal mixture may be deposited on top of the electrodes (a passivation layer may also be present on the glass substrate under the ITO). This is usually followed by an alignment layer, which will be in direct contact with the liquid crystal mixture in order to induce a homogeneous orientation in the required plane geometry. The two glass substrates are then assembled and glued together leaving a hole so that the evacuated cell can be filled with a liquid crystal under positive pressure, cleaned and then sealed (Kelly, 2000).

1.3 Future Requirements

As can be seen from the above, the transparent electrode is a key component within LCD displays and is also used extensively in next generation technologies including LCD-light-emitting diode (LCD-LED) displays and organic light-emitting diodes (OLEDs). LCD-LEDs use light-emitting diodes to provide the backlighting that replaces the CCFLs (mercury containing). In contrast, OLEDs use an emissive electroluminescent layer of organic compound that emits light in response to an electric current. It is clear that display technologies of the future have a large requirement for transparent electrodes.

ITO is one of the most widely used transparent conducting oxides because of two key properties, namely its electrical conductivity and optical transparency, as well as the ease with which it can be deposited as a thin film. Alternative electrode materials include aluminium-doped zinc oxide (AZO), gallium-doped zinc oxide (GZO), indium-doped zinc oxide (IZO) and indium-doped cadmium oxide (ICO).

Indium is produced mainly from residues generated during zinc ore processing (George, 2003). China is the leading producer of indium (390 tonnes in 2012), followed by Canada, Japan and South Korea with 70 tonnes each (George, 2003). Indium consumption in ITO accounts for over 70% of its total consumption and the greatest demand on ITO is reported as being LCDs (He *et al*., 2014). Currently, the price is around 540 USD/kg (Strategic Metal Report, 2014). It has been reported that there are fewer than 14 years of indium supplies left, based on current rates of extraction, demonstrating the need for recycling (George, 2003). In addition, avoiding risks to supply of resources within Europe for the information and communications technology (ICT) sector is of strategic European importance. Therefore, the waste liquid crystal panels have the potential for urban mining as a source of indium that can be used to secure Europe's future resource requirements.

1.4 Objectives

The objectives of the research were to investigate and explore the potential for indium recovery from LCDs. The specific objectives were to:

- investigate the presence and amount of indium contained in the liquid crystal panels of LCDs;
- review potential methodologies and best available techniques for the recovery of indium from LCDs;
- review the requirement for indium recovery from an industrial perspective;
- review the requirement for LCD recycling from a policy perspective.

2 Analysis of Liquid Crystal Panels

2.1 Objective

The objective of this research is to investigate the presence, structure and composition of the transparent electrode within liquid crystal panels of 10 different LCD displays. ITO, which is the most common transparent electrode, is a solid solution of indium(III) oxide (In_2O_3 – 90–95%) and tin(IV) oxide (SnO_2 – 5–10%) to be found in liquid crystal panels (He *et al.*, 2014). However, depending on the LCD manufacturer and the year of production, the indium content as well as the presence of alternative electrode materials within the liquid crystal panel can vary. Table 2.1 displays indium concentrations measured from different liquid crystal panels reported by different authors. It is clear that the amount of indium reported varies significantly from approximately 100 to 400 mg indium per kg of glass substrate.

There has been little investigation to date to understand the compositional and quantificational variations of transparent electrodes in LCDs; however, the key to achieving any industrially viable recovery process for indium will be centred around the actual amount of indium that is available for recovery. Therefore, understanding the possible liquid crystal panel composition and structural variations and their effect on subsequent viability of downstream recycling and recovery options will be of great significance. Of interest will be the indium location, thickness, pattern structure and presence of other materials in the liquid crystal panel, which may have an effect on the chosen recovery process for indium.

Table 2.1. List of reported indium quantity in liquid crystal panels of LCD displays

Author	Indium reported
Wang *et al.* (2013)	102 mg/kg of glass panel
Ruan *et al.* (2012)	0.37 mg/g of circuit board
Hasegawa *et al.* (2013)	380–410 mg/kg of glass panel
Yoshida *et al.* (2014)	321–395 mg/kg of glass panel
Lee *et al.* (2013)	260.7 mg/kg of glass panel

2.2 Experimental Procedure

Ten samples of liquid crystal panels were taken from different LCD displays, as detailed in Table 2.2. The individual samples were approximately 3 cm long × 3 cm wide.

Each sample was split to break the panel into the two glass substrates and the plastic filter sheets. The individual components were denoted as follows: glass front (GF) and glass back (GB) and plastic filter sheets (for which there could be up to three sheets per sample) as can be seen in Figure 2.1.

These samples were then analysed by an array of different microscopic techniques to determine the structure, location and thickness of transparent electrode and concentrations of indium present as described in the following sections.

2.2.1 Structure of liquid crystal panel internal surfaces

In order to examine the internal surface of the liquid crystal samples, the glass front and glass back of each sample were gently washed to remove any

Table 2.2. Details of the LCD displays from which samples of the liquid crystal panel components were sampled

Sample name	LCD make and model	Screen size (inches)
S1	Techhnika model 2610 207 CE	32
S2	Playsonic model 20 CLB	32
S3	Mirai model T27 004	26
S4	Finlux 32 FLD 850BC	32
S5	Technosoft LCD 2602	26
S6	Sony KDL-32P30	27
S7	Sony bravo KDL 32 4000	32
S8	Philips 26 PF 8946/12	26
S9	Philips 32 PF 5321/1A	32
S10	Normend NM 38A30	32

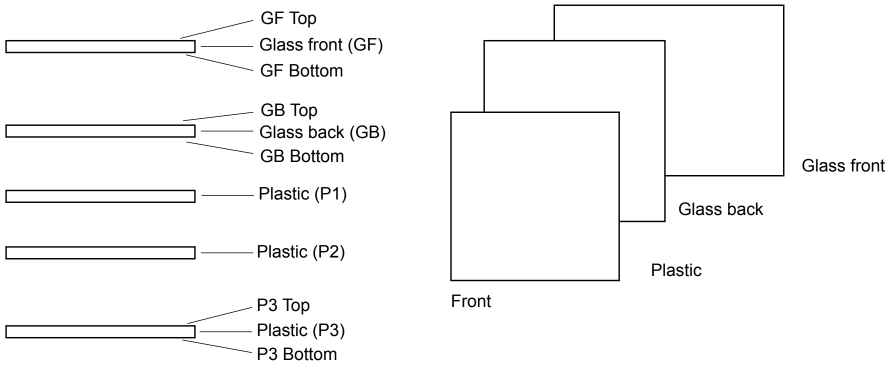


Figure 2.1. Schematic of the liquid crystal panel components and labelling terminology used.

liquid crystal residue. The samples were then placed under an optical microscope and the images recorded to reveal the patterning structure of the transparent electrode and thin film transistor.

2.2.2 Location of transparent electrode

In order to determine the location of the transparent electrode, the surface samples of the glass front and glass back of each liquid crystal panel substrate were examined under energy dispersive spectroscopy (EDS) analysis. The initial testing involved surveying the surface for indium and creating a visual map of the sample where lighter contrasting regions highlight the presence and location of indium on the surface of the sample. This technique was then augmented with EDS point analysis, where specific sites on the surface of the samples were analysed for weight per cent of indium present as well as recording the presence of other elements and metals. The point EDS data is accompanied by scanning electron microscope (SEM) micrographs specifying the analysis region for each particular sample.

2.2.3 Thickness of the transparent electrode

In order to determine the thickness of the transparent electrode layer, both the glass front and glass back of each of the liquid crystal panel substrates were cross-sectioned and mounted in cold epoxy resin as illustrated in Figure 2.2. The samples were ground on 200, 400, 800 and 1200 silicon carbide paper and then polished using 6 µm and 1 µm diamond polishing slurry. The samples were gold coated to increase conductivity and prevent charging of the sample, while undergoing SEM and EDS analysis.

2.2.4 Quantification of indium concentrations on the liquid crystal panel internal surfaces

In order to determine the concentration of indium, three of the samples exhibiting different structures (S1, S2 and S4) were selected for further external analysis by inductively coupled plasma–optical emission spectroscopy (ICP-OES). The glass front and glass back of each selected liquid crystal panel were ground down with the RETSCH ZM200 mill with 0.250 mm sieving. The resulting powder was leached with aqua regia [1 part HNO_3 (69%): 3 parts HCL (37%)] to dissolve the indium. The solutions were measured with the Agilent 5100 ICP-OES system (IDO Labs, Germany) with seaspray nebuliser. Calibration was performed with NIST-certified standard from Merck.

In addition to ICP-OES, the samples were also analysed in house under atomic absorption spectroscopy (AAS). Five square samples were cut from each of 10 LCD-thin-film transistor (TFT) panels and these samples were carefully trimmed using scissors until they weighed ~1.0 g. Each sample was individually digested in a 25 ml solution of fuming aqua regia at 60°C until the solution was completely evaporated. The remaining residue was dissolved with drops of concentrated HCL and then washed into a 50 ml volumetric flask and the five solutions made up to the mark using deionised water. Aliquots of each solution were drawn through a 0.2 µm Minisart syringe filter and placed in five 20 ml test tubes. A stock solution of pure indium was used to prepare standard solutions of indium in 50 ml volumetric flasks with four concentrations of 0.5, 5, 10 and 40 mg/l. Each standard and sample solution was aspired individually through a Spectra 2220 AAS using an air-acetylene

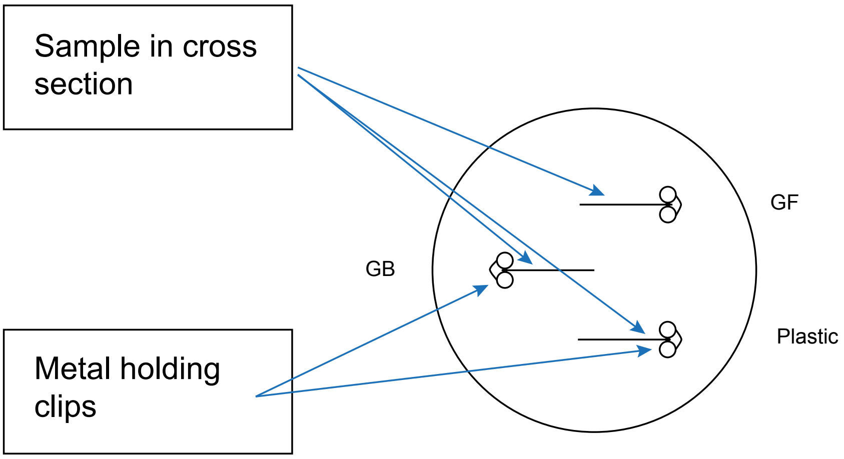


Figure 2.2. Schematic of the mounted cross-section samples and labelling terminology used. GF, glass front; GB, glass back.

flame and an indium cathode lamp (303.97 nm). The following AAS operating conditions were used for all repetitions (5 l/min air, 0.8 l/min C_2H_2 flow rate, burner slot 10 cm, 5 mA lamp current and 0.2 mm slit width). The nebuliser was rinsed carefully with deionised water between each run to clean it and the final indium concentration was quoted as mg of indium per kg of TFT glass.

2.2.5 Detailed characterisation of liquid crystal panel internal surfaces

In order to further characterise the internal surfaces of the liquid crystal panels, the glass front and glass back samples were analysed under X-ray diffraction (XRD) to reveal information regarding the composition and material phases present in the different samples. For these techniques, the surface samples were gently washed to remove liquid crystal residue.

Samples S1, S2 and S4 were selected for further study under focused ion beam (FIB) spectroscopy, which allows a mill of the surface sample to reveal the structure beneath the surface. For this technique, a platinum strip of approximately 10 µm is deposited on the internal surface of the liquid crystal panel in order to maintain the integrity of the surface to be milled. Milling with a gallium beam then removes material and creates a trench into the sample. The sample is then rotated and micrographs of the structure revealed within the trenched region are taken.

2.3 Experimental Results

2.3.1 Structure of liquid crystal panel internal surfaces

The glass front and glass back substrates of all 10 samples (S1–S10) were examined under the optical microscope on their internal surface (i.e. the surface containing the transparent electrode). Four main surface structures were evident and four samples to illustrate each structure were selected and are displayed in Figure 2.3. Sample S1 exhibited a rectangular cell structure, while S2 also exhibited a rectangular cell structure but with a substructure contained within. Sample S8 displayed a very different structure with a V-shaped cell with a substructure contained within, while S10 showed a plain V-cell structure. Samples S3, S4, S6, S7 and S9 all exhibited similar structures to S2 consisting of a rectangular cell with a subcell structure.

2.3.2 Location of transparent electrode

The EDS mapping results of the samples reveal the location of any indium present on the surface of the samples. The technique uses a brighter contrast to reveal the presence of indium, while darker regions on the micrograph reveal an absence of indium. By comparing this with the traditional SEM image of the sample in the same location, the dominant regions can pinpoint where indium is located.

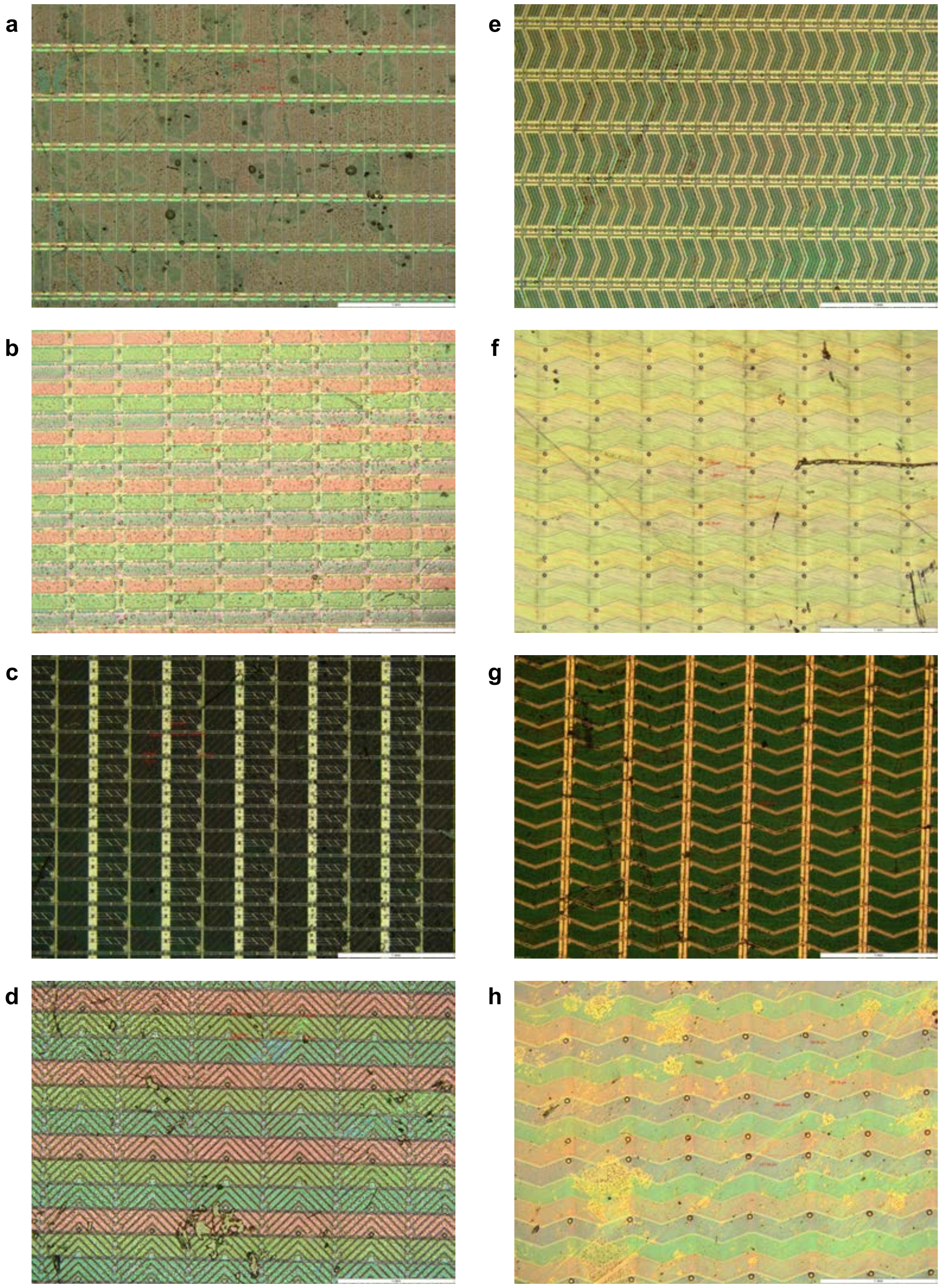


Figure 2.3. Optical surface images of samples of both glass substrates (i.e. glass back and glass front) for liquid crystal panel samples S1–S10. Scale bar: white bar at bottom of image = 1 mm. (a) S1 glass back internal surface; (b) S1 glass front internal surface; (c) S2 glass back internal surface; (d) S2 glass front internal surface; (e) S8 glass back internal surface; (f) S8 glass front internal surface; (g) S10 glass back internal surface; (h) S10 glass front internal surface.

The glass front and glass back substrates of all 10 samples (S1–S10) were examined under EDS on their internal surface (i.e. the surface containing the transparent electrode). Samples S1 and S2, illustrating the rectangular and subcell structures, are displayed in Figure 2.4, while the V-cell samples S8 and S10 did not reveal indium locations under EDS mapping. Samples S3, S4, S6, S7 and S9 all exhibited similar structures to S2.

In order to further investigate the presence and location of the transparent electrode, all 10 samples

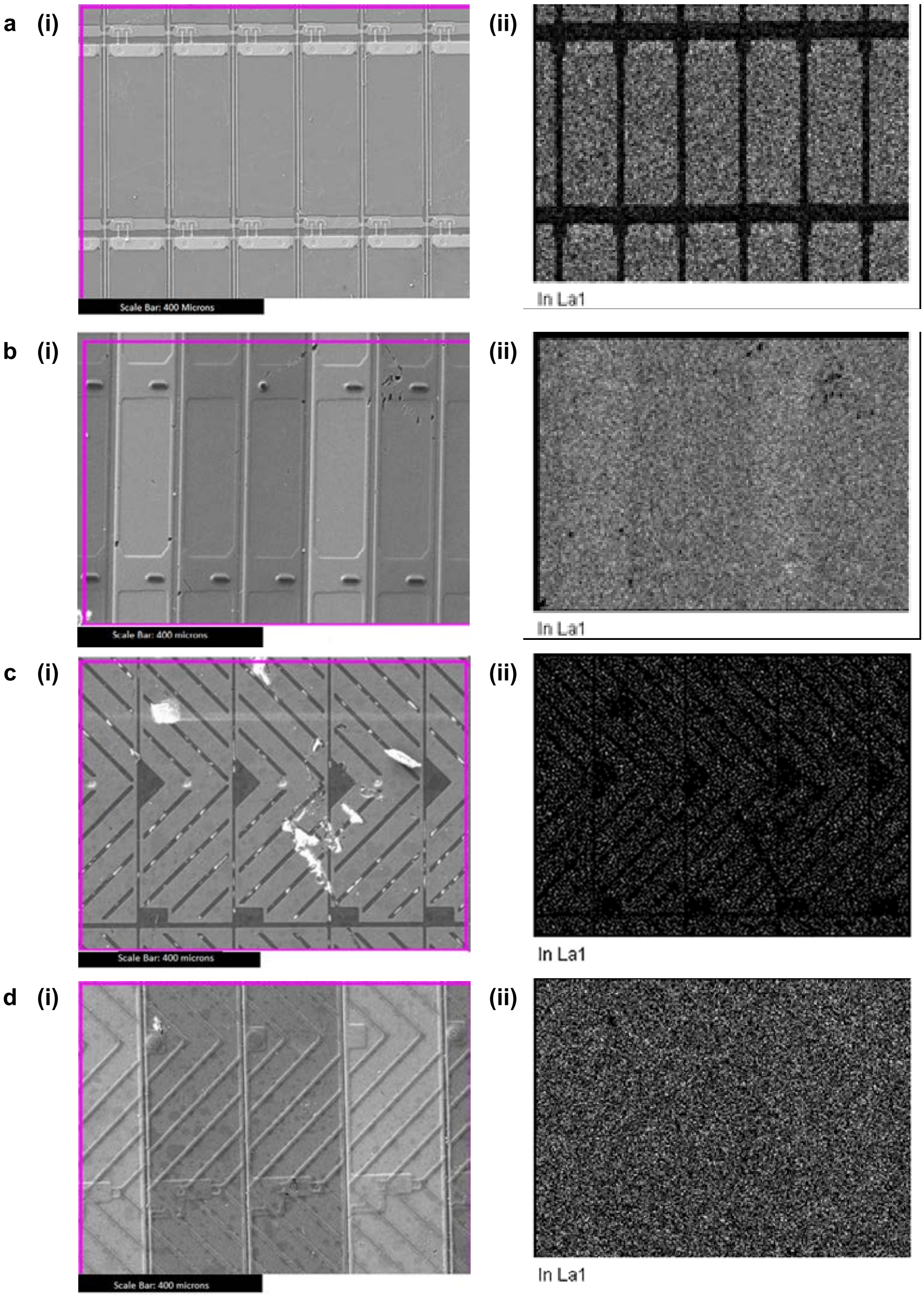
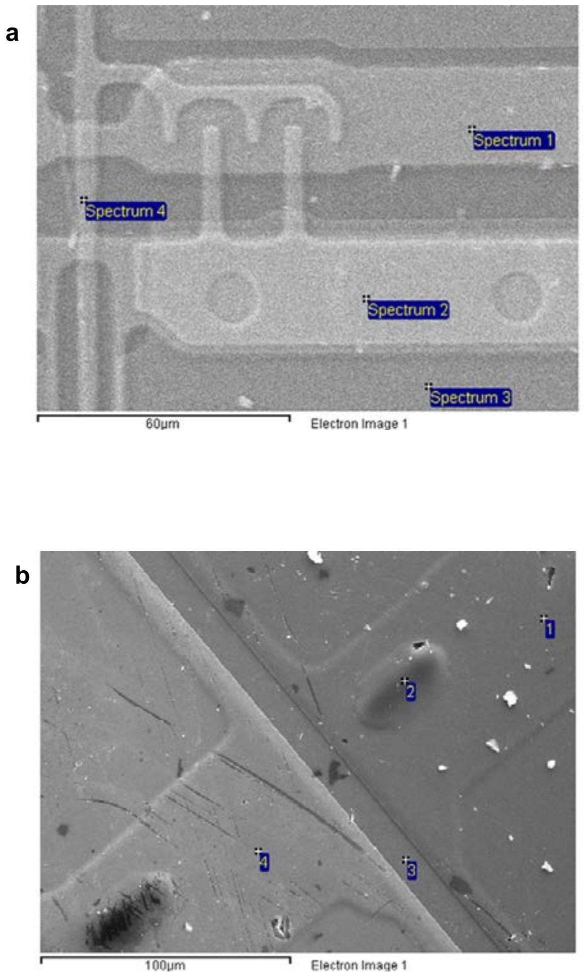


Figure 2.4. EDS maps of the internal surfaces of the liquid crystal panels. (a) Sample S1 glass back indium EDS map; (b) Sample S1 glass front indium EDS map; (c) Sample S2 glass back indium EDS map; (d) Sample S2 glass front indium EDS map.

were analysed under the point EDS, where up to five specific points were selected on the surface for weight per cent readings of the elements present at that location. These readings are accompanied by the SEM micrograph illustrating the exact location of the reading. The samples illustrating the four main surface structures (S1, S2, S8 and S10), as identified in section 2.3.1, were chosen and are displayed in Figures 2.5, 2.6, 2.7 and 2.8.

Samples S1 and S2 with the cell structures generally showed similar trends where relatively high levels of indium were detected on the front glass substrate with lower indium level on the back glass substrate. Sample S8 V-subcell structure did not exhibit the



Spectrum location (Weight % concentrations):

Spectrum 1: Al (21.71), Si (49.22), Ca (2.13), Mo (26.65), In (0.36), Sn (0).

Spectrum 2: Al (23.20), Si (32.27), Ca (0.69), Mo (33.59), In (8.78), Sn (1.48).

Spectrum 3: Al (8.13), Si (74.31), Ca (6.02), Mo (0), In (12.33), Sn (0.40).

Spectrum 4: Al (20.99), Si (49.61), Ca (2.03), Mo (26.65), In (0.14), Sn 0.58).

Spectrum location (Weight % concentrations):

Spectrum 1: Al (5.31), Si (22.21), Cl (1.06), Ca (2.9), Ni (0), Cu (1.62), Br (0), In (60.49), Sn (6.41), N (0).

Spectrum 2: Al (2.15), Si (10.06), Cl (1.09), Ca (0), Ni (0), Cu (1.14), Br (0), In (76.23), Sn (9.33), N (0).

Spectrum 3: Al (8.26), Si (35.14), Cl (0), Ca (4.56), Ni (0), Cu (0), Br (0), In (47.33), Sn (4.72), N (0).

Spectrum 4: Al (0), Si (9.51), Cl (1.77), Ca (1.22), Ni (1.67), Cu (1.11), Br (26.08), In (41.13), Sn (3.4), N (14.11).

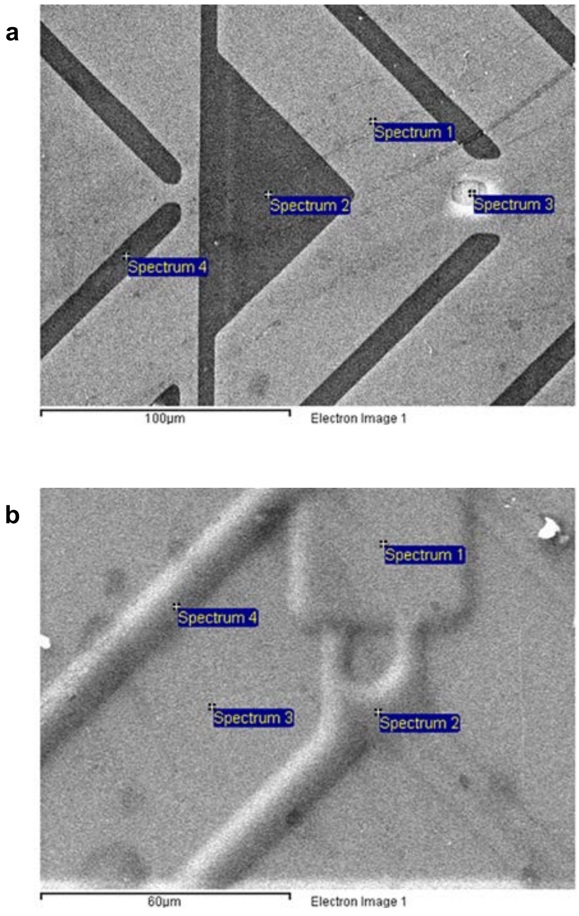
Figure 2.5. Location of the transparent electrode on the panel internal surfaces of sample S1. (a) S1 glass back internal surface SEM micrograph and corresponding surface EDS results; (b) S1 glass front internal surface SEM micrograph and corresponding surface EDS results.

presence of indium on the front glass substrate and had only low concentrations in a specific region on the back glass substrate. The back glass substrate also displayed readings for zinc and arsenic. The plain V-cell structured sample S10 continued the trend, showing the lack of presence of indium on the front glass substrate and having a low reading on the back glass substrate.

2.3.3 Thickness of the transparent electrode

In order to further investigate the thickness of the transparent electrode, all 10 samples were prepared for cross-sectional analysis and examined under the SEM. Figure 2.9 illustrates sample S1 taken in the region of the internal layers of the cross section to identify the transparent electrode both for the glass front and the glass back substrates.

Samples S1 and S2 with the cell structures generally showed similar trends where relatively high levels of indium were detected on the front glass substrate with lower indium level on the back glass substrate. Sample S8 V-subcell structure did not exhibit the presence of indium on the front glass substrate and only low concentrations in a specific region on the back glass substrate. The back glass substrate also displayed readings for zinc and arsenic (potential alternative electrode composition). The plain V-cell structured sample S10 continued the trend, showing the lack of presence of indium on the front glass substrate and a low reading on the back glass substrate.



Spectrum location (Weight % concentrations):

Spectrum 1: Al (20.66), Si (31.95), S (2.73), Ti (15.97), In (26.24), Sn (2.46).

Spectrum 2: Al (16.45), Si (54.81), S (3.93), Ti (23.52), In (0.26), Sn (1.03).

Spectrum 3: Al (12.97), Si (45.55), S (0), Ti (21.35), In (17.75), Sn (2.63).

Spectrum 4: Al (27.38), Si (45.13), S (3.42), Ti (23.23), In (0.32), Sn (0.52).

Spectrum Location (Weight % Concentrations):

Spectrum 1: Al (3.89), Si (5.89), S (1.27), Cl (12.81), Cu (1.36), Br (22.76), In (47.37), Sn (4.65).

Spectrum 2: Al (1.83), Si (4.06), S (3.34), Cl (11.11), Cu (0.98), Br (20.09), In (52.52), Sn (6.07).

Spectrum 3: Al (3.73), Si (9.78), S (1.21), Cl (12.61), Cu (1.13), Br (25.50), In (40.80), Sn (5.24).

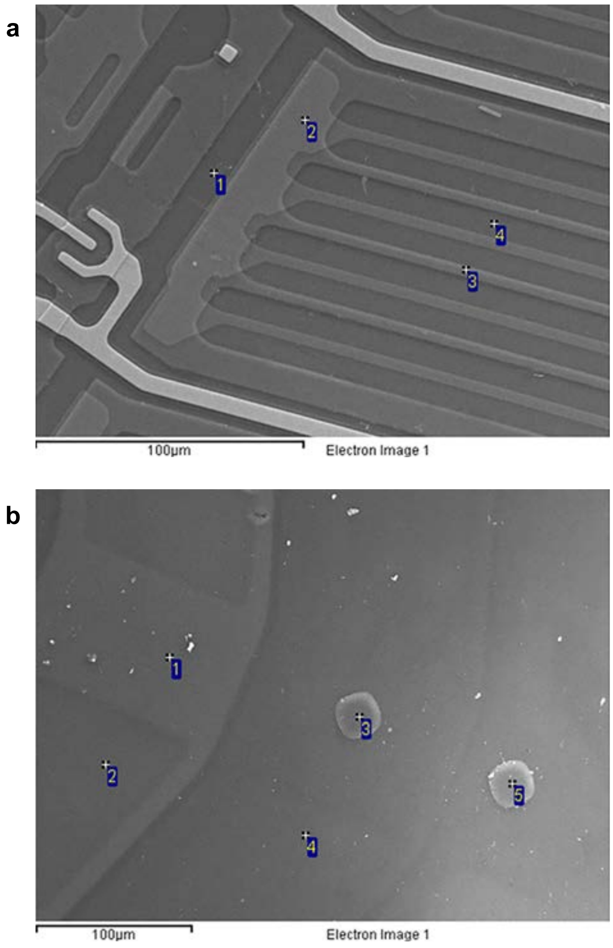
Spectrum 4: Al (2.67), Si (5.68), S (2.53), Cl (11.73), Cu (1.08), Br (19.44), In (51.33), Sn (5.54).

Figure 2.6. Location of the transparent electrode on the panel internal surfaces of sample S2. (a) S2 glass back internal surface SEM micrograph and corresponding surface EDS results; (b) S2 glass front internal surface SEM micrograph and corresponding surface EDS results.

2.3.4 Quantification of indium concentrations on the liquid crystal panel internal surfaces

In order to investigate the specific concentrations of indium present in the liquid crystal panels, three samples of the more common rectangular cell structure were selected for inductively coupled plasma–optical absorption spectroscopy (ICP-OAS), which was located at IDO Labs, Germany. Plain rectangular cell structured sample S1 revealed similar indium levels for the glass front and the glass back substrates ranging from 181.1 to 186.5 mg of indium per kg glass substrate. The subcell rectangular structured sample S2 revealed very different concentrations for the glass front and the glass back substrates with the front glass substrate containing up to 255 mg indium per kg glass and the back glass substrate as low as 143.3 mg/kg glass. However, sample S4, which also had a rectangular subcell structure, contained much lower concentrations of indium than samples S1 and S2. Sample S4 revealed an indium concentration of 60.9 mg/kg glass on the front substrate and as low as 37.4 mg/kg glass on the back substrate (see Table 2.3).

As the ICP-OAS revealed a large amount of variation in the indium concentrations, all 10 samples were then prepared for AAS. The ICP-OAS and AAS for samples S1, S2 and S4 correlated well with some slight variation, as would be expected given the site-specific nature of the samples. The ASS testing revealed a continuation of the wide variation in indium concentrations for the different liquid crystal panels. Sample S8 with the V-subcell structure revealed a high indium concentration on the front substrate of



Spectrum Location (Weight % Concentrations):

Spectrum 1: N (38.98), Al (6.22), Si (50.56), Ca (3.6), Zn (0.32), As (0.32), In (0), Sn (0).

Spectrum 2: N (33.98), Al (19.04), Si (38.59), Ca (1.82), Zn (0), As (0), In (5.7), Sn (0.88).

Spectrum 3: N (39.63), Al (18.57), Si (39.68), Ca (2.12), Zn (0), As (0), In (0), Sn (0).

Spectrum 4: N (41.06), Al (6.06), Si (49.24), Ca (3.37), Zn (0), As (0.28), In (0), Sn (0).

Spectrum location (Weight % concentrations):

Spectrum 1: Al (0), Si (25.1), Cl (4.24), Ca (4.74), Ni (4.87), Cu (1.97), Br (40.42), Au (18.66).

Spectrum 2: Al (0), Si (36.54), Cl (4.06), Ca (6.29), Ni (4.41), Cu (2.33), Br (46.36), Au (0).

Spectrum 3: Al (0), Si (16.91), Cl (83.09), Ca (0), Ni (0), Cu (0), Br (0), Au (0).

Spectrum 4: Al (9.92), Si (51.8), Cl (26.73), Ca (11.55), Ni (0), Cu (0), Br (0), Au (0).

Spectrum 5: Al (9.86), Si (57.2), Cl (21.59), Ca (11.35), Ni (0), Cu (0), Br (0), Au (0).

Figure 2.7. Location of the transparent electrode on the panel internal surfaces of sample S8. (a) S8 glass back internal surface SEM micrograph and corresponding surface EDS results; (b) S8 glass front internal surface SEM micrograph and corresponding surface EDS results.

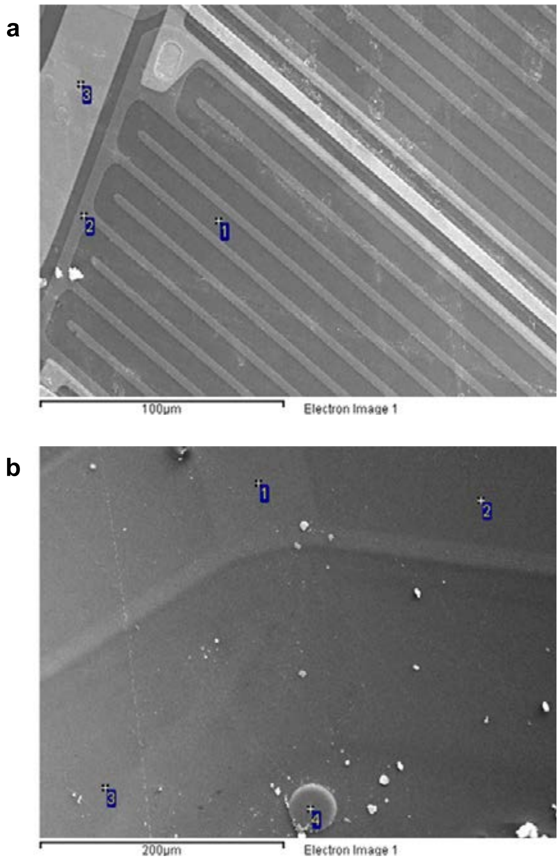
285.7 mg indium per kg glass, while the back substrate of the same sample recorded just 14 mg indium per kg glass. The plain V-cell structured sample S10 also revealed low indium concentrations but with more consistent levels on the glass front (46.2 mg/kg) and the glass back (34.6 mg/kg) substrates (see Table 2.4).

2.3.5 Detailed characterisation of liquid crystal panel internal surfaces

In order to characterise the liquid crystal panels and augment the information regarding their structure and composition, the samples were prepared for XRD analysis.

Glass back substrates results

Plain rectangular cell structured samples S1 and S5 both showed significant peaks at approximately 31 degrees 2θ, with samples S1 exhibiting additional dominant peaks at 40 and 43 2θ degrees. Subcell structured rectangular cells samples S2, S3, S6, S7 and S9 all showed dominant or significant peaks around 40 and 43 degrees 2θ with a minor peak around 31 degrees (except S3 where the 31 degree peak was dominant). However, sample S4, which also has a subcell rectangular cell structure, showed a different trend with one single dominant peak at 44 degrees 2θ. This trend corresponds well with the EDS



Spectrum location (Weight % concentrations):

Spectrum 1: N (32.12), O (13.5), Mg (0.54), Al (6.12), Si (43.92), Ca (3.8), Ti (0), Cu (0), In (0), Sn (0).

Spectrum 2: N (24.62), O (17.05), Mg (0.46), Al (5.42), Si (42.06), Ca (3.32), Ti (0), Cu (0), In (6.22), Sn (0.86).

Spectrum 3: N (31.23), O (4.61), Mg (0), Al (0.98), Si (30.43), Ca (1.31), Ti (0.35), Cu (31.08), In (0), Sn (0).

Spectrum Location (Weight % Concentrations):

Spectrum 1: O (36.61), Mg (0), Al (0), Si (14.09), S (0), Cl (2.27), Ca (1.2), Ni (4.71), Cu (1.87), Br(38.25).

Spectrum 2: O (19.37), Mg (0.57), Al (0), Si (27.36), S (0), Cl (3.24), Ca (2.99), Ni (4.29), Cu (1.59), Br(40.58).

Spectrum 3: O (23.15), Mg (1.17), Al (9.53), Si (48.39), S (0), Cl (10.31), Ca (7.45), Ni (0), Cu (0), Br(0).

Spectrum 4: O (40.96), Mg (0), Al (5.77), Si (34.82), S (34.82), Cl (4.18), Ca (5.39), Ni (0), Cu (7.47), Br(0).

Figure 2.8. Location of the transparent electrode on the panel internal surfaces of sample S10. (a) S10 glass back internal surface SEM micrograph and corresponding surface EDS results; (b) S10 glass front internal surface SEM micrograph and corresponding surface EDS results.

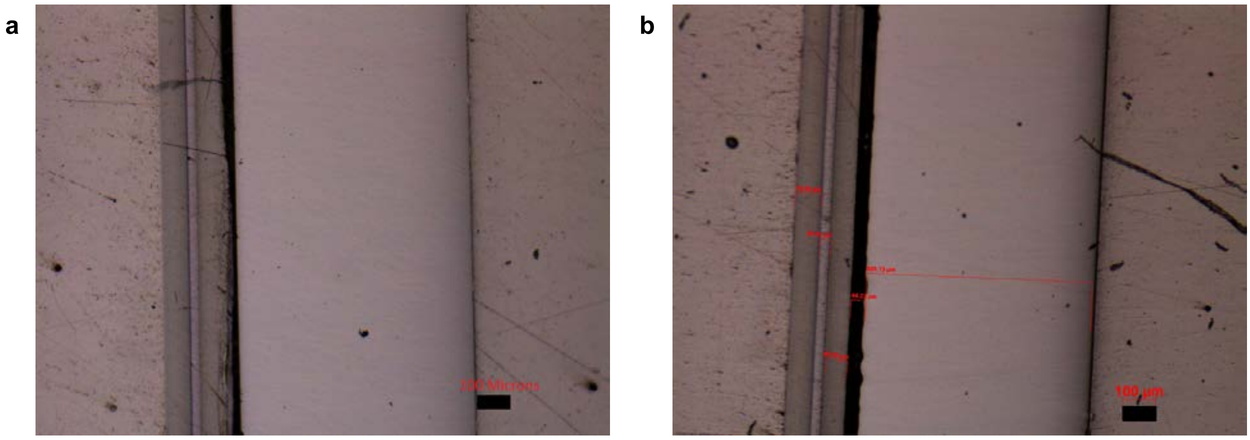


Figure 2.9. Optical images of cross sections of the glass front and glass back substrates of the liquid crystal panels. (a) S1 glass back optical cross-section – ITO thickness 1.58 µm; (b) S1 glass front optical cross-section – ITO thickness 1.86 µm.

Table 2.3. ICP-OAS data for samples S1, S2, and S4

Sample	Indium (mg/kg glass substrate)
S1 GF	181.1
S1 GB	186.5
S2 GF	255.3
S2 GB	143.3
S4 GF	60.90
S4 GB	37.40

GB, glass back; GF, glass front.

Table 2.4. AAS of the liquid crystal panel samples S1 to S10

Sample	Indium (mg/kg glass substrate)
S1 GF	162.5
S1 GB	166.7
S2 GF	254.2
S2 GB	129.2
S3 GF	254.5
S3 GB	72.7
S4 GF	38.5
S4 GB	0.0
S5 GF	275.0
S5 GB	91.7
S6 GF	292.3
S6 GB	46.2
S7 GF	250.0
S7 GB	91.7
S8 GF	285.7
S8 GB	14.3
S9 GF	234.6
S9 GB	65.4
S10 GF	46.2
S10 GB	34.6

GB, glass back; GF, glass front.

and AAS results. The plain V-cell structure sample S10 showed an identical 44 degree 2θ peak to the S4 sample, while the XRD pattern of the V-subcell structured sample S8 detected high levels of noise during the scan (see Figure 2.10).

Glass front substrate results

Plain rectangular cell structured samples S1 and S5 both showed significant peaks at approximately 31 degrees 2θ, with samples S1 exhibiting additional dominant peaks at 40 and 43 2θ degrees. This result was similar to the glass back XRD pattern. Subcell structured rectangular cell samples S2, S3, S6, S7 and S9 all showed dominant or significant peaks around 36, 52 and 61 degrees 2θ with a minor peak around 31 degrees (except S3, where the 31 degree peak was dominant). In contrast, the XRD pattern of sample S4 only detected noise during the scan. The XRD analysis for both the plain V-cell structure sample S10 and the V-subcell structured sample S8 detected high levels of noise during the scan (see Figure 2.11).

In order to examine the structure of the transparent electrode, two of the most representative samples, S1 and S2, were chosen for analysis under the FIB microscope to see if any microstructural characterisations were present. Figure 2.12 displays the methodology and location of the FIB mill into the glass substrate and the pinpointing of the transparent electrode layer in cross-section, while Figure 2.13 reveals the micrographs taken of samples S1 and S2. Unfortunately, the FIB micrographs did not reveal any characteristic details of the sample; grain boundaries and/or texture/orientations were not evident.

2.4 Results and Discussion

2.4.1 Structure of liquid crystal panel internal surfaces

The initial optical microscopy analysis of section 2.3.1 was performed on both substrates of the glass panel contained within each sample, namely the glass front and the glass back. The patterns exhibited on both substrates of each of the glass panels within the one sample appeared to be similar. Usually, the glass back substrate exhibited a detailed pattern of cells, while the glass front substrate image was usually more blurred due to the presence of the pixel colour filters. The optical surface images of the glass front panels generally revealed the presence of the three colour filters, namely red, green and blue.

Sample S1 revealed a simple rectangular cell pattern on both sides, while sample S2, also exhibiting a rectangular cell pattern, had a more detailed substructure visible within each rectangle. This substructure consisted of diagonal line tracks within each rectangular cell and was visible on both the front and back glass panels. However, the glass back

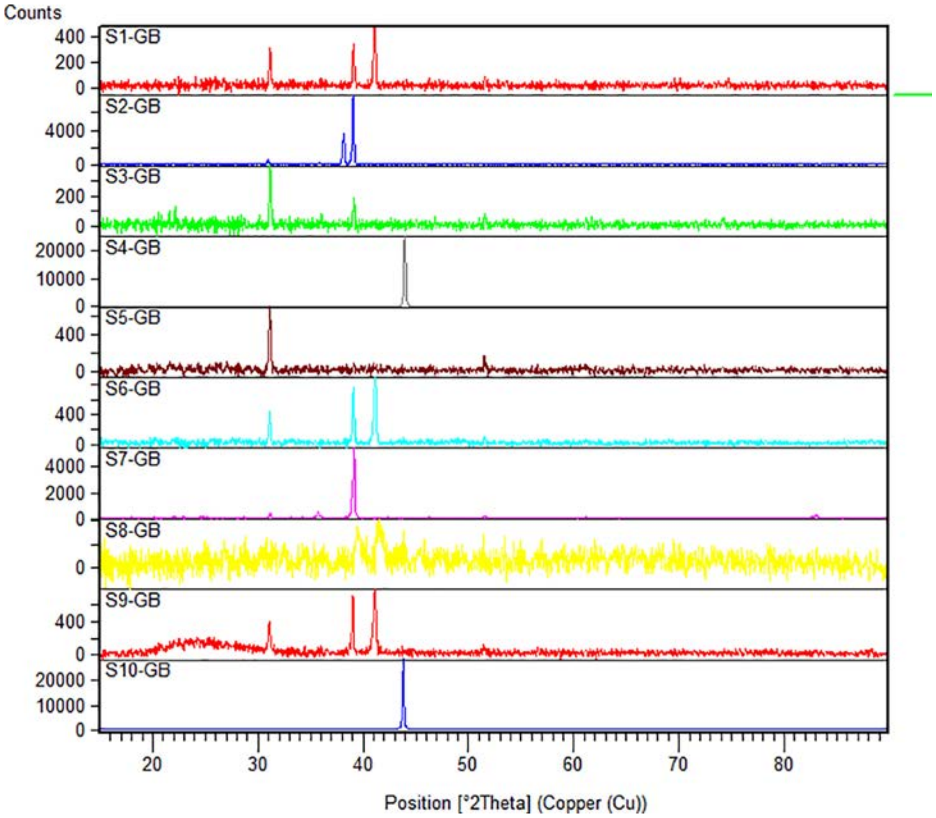


Figure 2.10. XRD micrographs of liquid crystal panel internal surfaces of the glass back substrate.

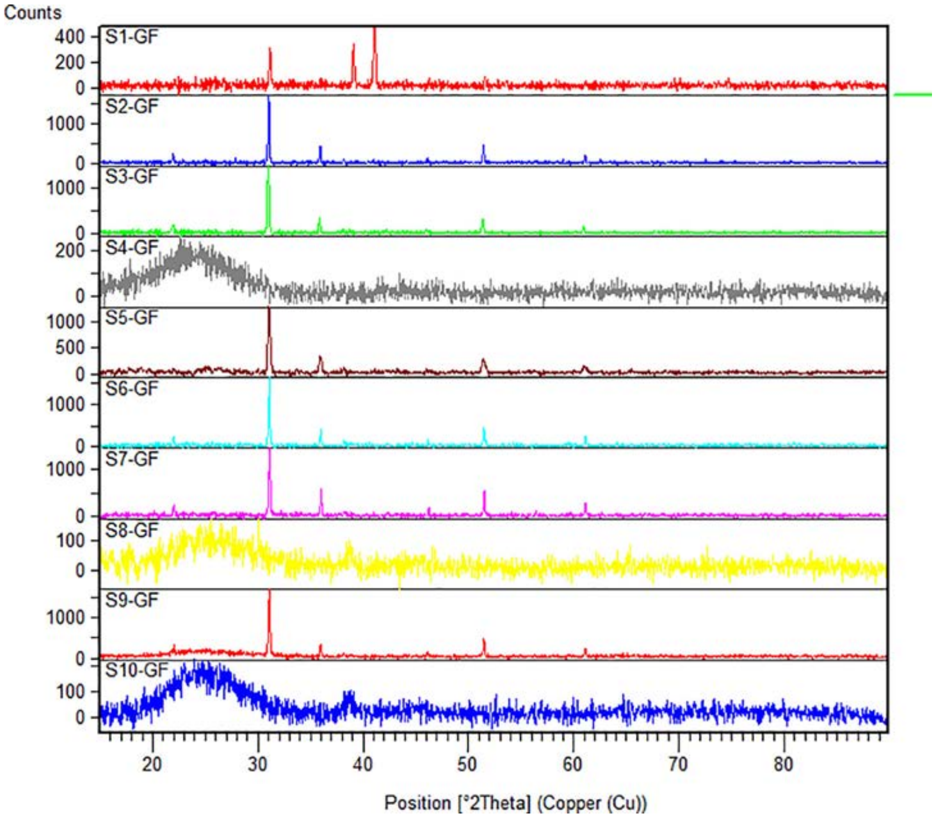


Figure 2.11. XRD micrographs of liquid crystal panel internal surfaces of the glass front substrate.

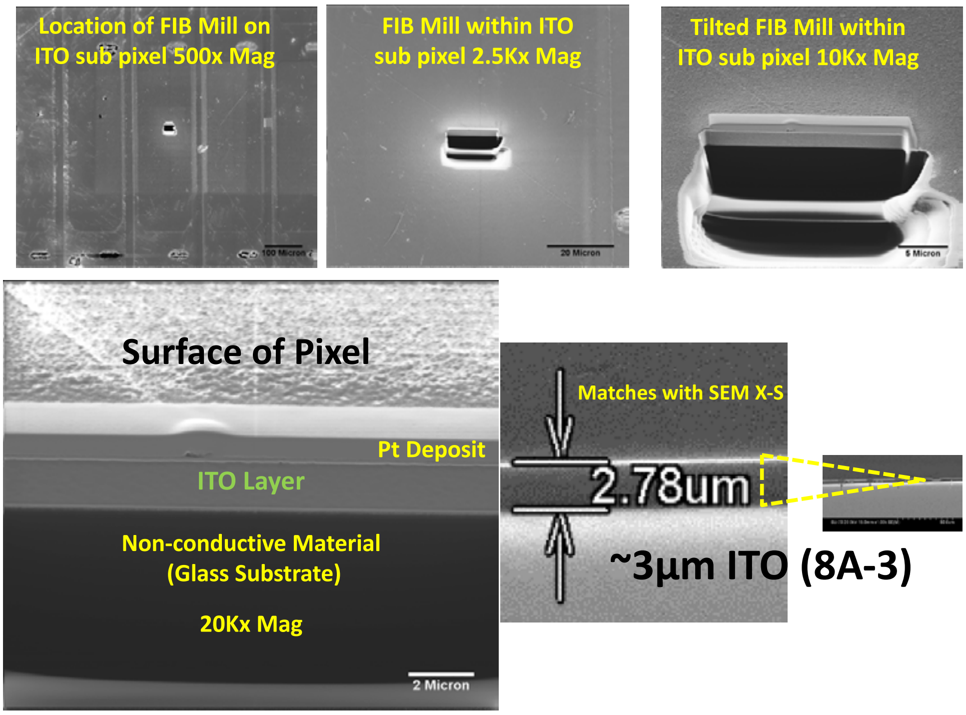


Figure 2.12. Overview of FIB micrographs of the liquid crystal panel internal surfaces of sample S1.

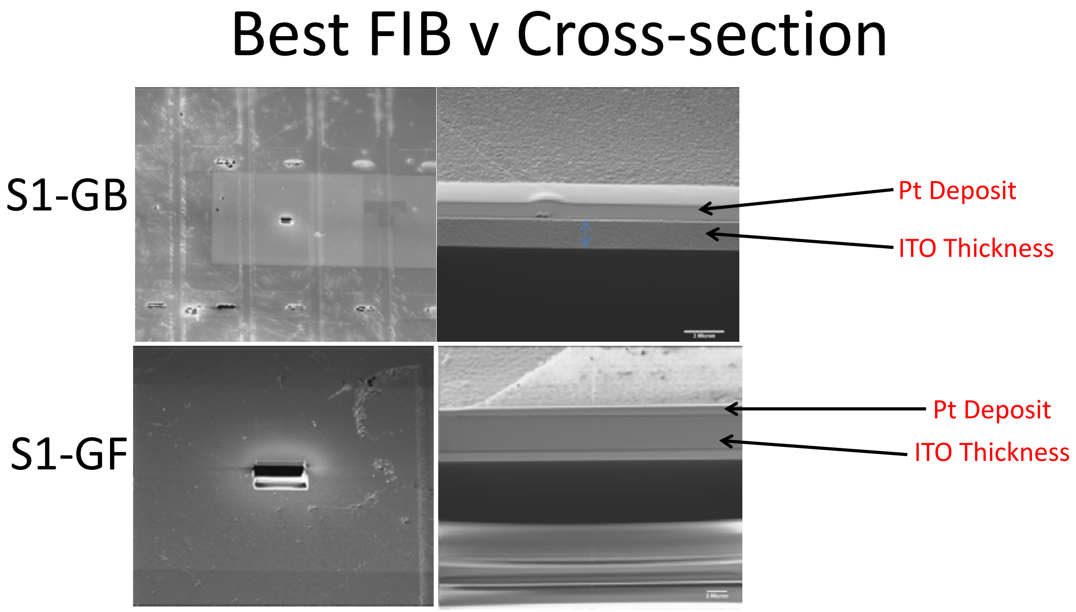


Figure 2.13. Micrograph of FIB mill of the internal surface of sample S1.

surface also revealed the presence of more tracks on the surface running parallel to the rows of rectangular cells.

Sample S3 revealed a similar structure to sample S2; however, the more detailed tracks were not present. Sample S4 had a rectangular cell structure with a subcell, while sample S5 had a simple rectangular cell structure. Sample S6 and sample S7 showed a rectangular cell structure with a diagonal line substructure contained within and the glass back panel revealed a track system running through the individual cells. Sample S9 showed a rectangular structure with a substructure of diagonal tracks similar to sample S2.

Samples S8 and S10 exhibited a different structure where the cell shape was a flattened “V” shape. Sample S8 revealed a substructure of lines or tracks running though the cell, while sample S10 did not exhibit such a substructure.

It is clear that a lot of variety exists between the patterns on the glass panels of different LCD displays. In some cases, for example samples S6 and S7, the LCDs were both manufactured by Sony and exhibited similar structures on the glass panels. However, in the case of samples S8 and S9, which were both manufactured by Philips, it was clear that very different structures existed between the panels of the same manufacturer.

Table 2.5 summarises the structures observed within the 10 samples analysed. The rectangular cell with subcell structure was the most common within the examined samples.

2.4.2 Location of transparent electrode

The EDS mapping results of the samples in section 2.3.2 reveal the location of any indium present on the surface of the samples. The samples with rectangular cell structures, namely samples S1 and S5 from the optical examination, revealed EDS maps with highly defined indium-containing regions. The EDS map of the back glass substrate of the liquid crystal panels of both S1 and S5 registered indium within the cell region, while the surrounding peripheries did not contain indium. On the glass front side of these samples, the EDS map revealed high contrast in all regions indicating high levels of indium were present across the entire surface.

In general, the samples that exhibited rectangular cell structures with the presence of a subcell structure showed similar EDS maps for the indium presence as above; however, the patterns lacked the high level of contrast and definition as seen for the plain rectangular cell samples. Indium generally resided within the subcell structure on the glass back and across the entire surface on the glass front. However, sample S4 did not reveal the presence of any indium on the glass back.

Table 2.5. Structures observed within the 10 samples analysed

Structure	Samples
Rectangular cell structure	S1, S5
Rectangular cell with subcell structure	S2, S3, S4, S6, S7, S9
V-cell structure	S10
V-cell with subcell structure	S8

The V-cell structures in general showed lower concentrations of indium and, in the case of sample S8 (V-cell with subcell structure), indium was not detected on the surfaces of the samples that were tested.

These EDS mapping results were then correlated with the point EDS/SEM data also displayed in section 2.3.2. The SEM allowed high magnification images of the internal surface of the panels, which showed in detail the complex geometries and topographies of the panels as well as the indium location.

Sample S1 findings:

- The front panel displayed high readings of indium and tin, supporting the use of ITO as the transparent electrode.
- Indium readings on the front panel of up to 76 percentage by weight (wt.%) accompanied by a tin reading of 9.33 wt.% alluded to an 8:1 ratio of indium to tin. Oxygen readings are usually not accurately readable by EDS; therefore, the composition measures are not absolute values.
- EDS readings of the front panel also picked up a significant reading for aluminium (5–8 wt.%), silicon (10–35 wt.%) and copper (approximately 1 wt.%), which were believed to be associated with the glass panel itself, while readings for chlorine and bromine were believed to be associated with residues of the liquid crystal component itself.
- EDS analysis of the back panel also recorded indium and tin readings with greatly varying ratios and at much lower quantities, where the highest reading of 12.33 wt.% indium was measured along with 0.4 wt.% tin.
- Much higher levels of aluminium (approximately 20 wt.%) and silicon (approximately 32–74 wt.%) were recorded on the back panel accompanied by significant levels of molybdenum (26–33 wt.%).

Sample S2 findings:

- The front panel revealed high levels of indium and tin with the highest reading in the range of 52.52 wt.% indium and 6.07 wt.% tin with an approximate 8:1 ratio. Relatively low levels of aluminium (approximately 1–4 wt.%), silicon (4–9 wt.%) and copper (approximately 1 wt.%)

were detected. This was similar to that seen in sample S1.
- Chlorine and bromine were also present in the reading and were believed to be associated with the liquid crystal components.
- The back panel exhibited the same trend as sample S1 with lower levels of indium (0.32–26 wt.%) and tin (0.5–2.4 wt.%) being picked up, while aluminium (12–27 wt.%) and silicon (31–54 wt.%) levels increased. A significant presence of titanium (15–23 wt.%) on the back panel was detected, which had not been present in the front panel.

Sample S3 findings:

- The front panel revealed high levels of indium and tin with the highest reading in the range of 50–71 wt.% indium and 3–10 wt.% tin. There were relatively low levels of aluminium (approximately 3–6 wt.%) and silicon (14–26 wt.%), while copper was not present.
- Chlorine was detected (without the presence of bromine) and was believed to be associated with the liquid crystal components.
- The back panel exhibited lower levels of indium (13–16 wt.%) and relatively high levels of tin (2–21 wt.%), potentially indicating that segregation of the indium and tin phases may have occurred.
- Silicon (38–92 wt.%) levels were particularly high, while aluminium levels were approximately 9–28 wt.%. No additional elements were present in the back panel analysis of sample S3.

Sample S4 findings:

- Relatively low levels of indium (0.6–4.58 wt.%) were recorded from the front panel of sample S4, while tin levels were in the range of 4–6 wt.%. Aluminium and silicon readings were significant in the ranges of 10–35 wt.% and 53–55 wt.%, respectively.
- The back panel also had relatively low indium levels (0.4–7.9 wt.%), with tin generally in the range of 4 wt.%. Silicon readings were particularly high in the range of 62–83 wt.%, while aluminium was approximately 9–10 wt.%. No other elements were detected on the back panel.

Sample S5 findings:

- Significant levels of indium (45–81 wt.%) and 4–7 wt.% tin were recorded on the front panel for sample S5. Readings for aluminium (6–10 wt.%) and silicon (8–32 wt.%) were accompanied by a smaller proportion of magnesium (approximately 1 wt.%).
- Chlorine believed to be associated with the liquid crystal components was also present up to 7 wt.%.
- The back panel in this case showed lower levels of indium (9–15 wt.%) and tin (approximately 1 wt.%) with consistent levels of aluminium (approximately 20 wt.% average), silicon (48 wt.% average) and magnesium (approximately 1 wt.%).
- However, the back panel of sample S5 also exhibited significant readings for molybdenum (16–33 wt.%) and strontium (0.6–21 wt.%).

Sample S6 findings:

- Sample S6 displayed high readings for indium (47–63 wt.%) and tin (4–5 wt.%), and typical levels of aluminium (4–8 wt.%) and silicon (16–33 wt.%) in the front panel.
- The back panel showed lower levels of indium (5–7 wt.%) and tin (0.5–1 wt.%) accompanied by aluminium of 5–18 wt.% and silicon of 33–51 wt.%. Molybdenum was detected in one specific location of the surface at 8 wt.%. However, the back panel recorded high levels of nitrogen in all regions in the range of 31–38 wt.%.

Sample S7 findings:

- The front panel of sample S7 showed high levels of indium (46–51 wt.%) and tin (4–7 wt.%) with typical levels of aluminium (3–8 wt.%) and silicon (10–31 wt.%). Magnesium was present in two of the regions analysed in the range of 0.9–1.6 wt.% and strontium was detected at 5.51 wt.% in one of the regions.
- The back panel of sample S7 showed typically lower readings of indium (8–12 wt.%) and tin (0.8–1.2 wt.%) with significant aluminium (5–14 wt.%) and silicon (28–48 wt.%).
- Nitrogen was detected in a relevantly high reading (31–40 wt.%) on the back panel, with some regions exhibiting titanium and magnesium readings.

Sample S8 findings:

- The EDS analysis of sample S8 did not register any indium or tin present on the front panel. Silicon was abundant in the sample in the range of 16–57 wt.%. Aluminium, nickel and copper were also present in specific regions analysed within the sample and were believed to be linked to the glass substrate.
- Chlorine, calcium and bromine were also detected and were believed to be linked to the liquid crystal component.
- The front panel also exhibited a very high gold reading in a specific region, which could not be accounted for as part of the sample gold coating.
- The back panel revealed the presence of indium in only one region analysed at a value of 5.7 wt.% and accompanied by 0.88 wt.% tin. More traditional levels of aluminium (6–18 wt.%) and silicon (38–50 wt.%) were also evident in the back panel accompanied by a high reading of nitrogen from all regions analysed (in the range of 33–41 wt.%).
- The presence of zinc (0.32 wt.%) was also detected in the back panel, with arsenic (0.32 wt.%) in specific locations as well.

Sample S9 findings:

- Sample S9 showed high readings of indium (51–70 wt.%) and tin (5 wt.%) in all regions analysed on the front panel. Aluminium and silicon readings were in the range of 3–7 wt.% and 8–30 wt.%, respectively.
- Magnesium was also detected in two of the three regions analysed at approximately 1 wt.%.
- Analysis of the back panel revealed lower indium levels of 8 wt.% and tin 0.7 wt.% and they were only detected in one of the regions analysed. Aluminium and silicon readings were in the range of 6–20 wt.% and 26–51 wt.% and all regions were accompanied by high nitrogen readings (28–38 wt.%). A relatively high presence of molybdenum was also found in one of the regions analysed.

Sample S10 findings:

- The front panel of sample S10 did not register any indium or tin presence. However, it did register more standard readings for aluminium (5–9 wt.%) and silicon (14–48 wt.%) in different regions.
- The back panel did register indium and tin presence on one of the regions analysed at 6.33 wt.% and 0.38 wt.%, respectively, while aluminium and silicon readings were in the range of 0.9–6 wt.% and 30–43 wt.%, respectively.
- Titanium and magnesium were also present in different regions of the sample in varying quantities.

The above analyses, performed across the internal surface for both substrates of all the samples, have underlined the variation in the indium present and the location of the indium, with predominantly high readings for indium presence on the front panel and typically lower levels on the back panel. In addition, the dominant presence of elements from the glass substrate included aluminium and silicon, with some samples showing high readings for the presence of nitrogen on the back panels. In addition, the presence of other elements such as titanium, nickel, magnesium and strontium have been recorded at varying levels in specific samples only. From a structural perspective, rectangular cell structure liquid crystal panels appear to have a continuous coating presence of indium-based ITO on the internal surface of the front panel and cell pattern presence of ITO on the international surface of the back glass substrate. Liquid crystal panels with the V-cell structure have very low indium concentrations present on the front glass substrate only.

Understanding the infeed composition of liquid crystal panels will be fundamental to designing a metallurgical recovery system that is capable of managing the variations in key parameters. Table 2.6 summarises and correlates the results of the EDS mapping and the point EDS data according to structure type of the liquid crystal panel.

2.4.3 Thickness of the transparent electrode

The optical microscopy of the cross-sections of the panels shown in section 2.3.3 revealed that both the front and back glass substrates had a thickness of approximately 550–600 µm (glass thickness). On the external surface of both glass substrates, two associated layers on top of the glass with a thickness

Table 2.6. Table of correlations from EDS mapping and point EDS data for samples with different cell structures

Structure	Samples	EDS mapping results	Point EDS correlations
Rectangular cell	S1, S5	Indium only present on cell pads of the glass back panel Indium recorded as present across the entire surface of the glass front	Lower levels of indium detected on the glass back panel and only in specific locations within cells Higher levels of indium detected on all regions of the front glass substrate up to and above 70 wt.%
Rectangular cell with subcell	S2, S3, S4, S6, S7, S9	Samples S2, S3, S6, S7 and S9 showed similar results Indium location within the subcell structure of the cell pads on the glass back Indium recorded as present across the entire surface of the glass front S9 revealed more bright contrast across the entire glass back indicating greater indium presence and dispersion Sample S4 gave a different result No indium was present on the glass back No indium was present on the glass front	Samples S2, S3, S6, S7 and S9 showed: Lower levels of indium detected on the glass back panel and only in specific locations within cells Higher levels of indium detected on all regions of the front glass substrate up to and above 50 wt.%
V-cell	S10	Low contrast indicates the indium is present on the glass back but in lower concentrations compared to other samples EDS map of glass front was not available	No indium detected on the glass back panel Low levels of indium up to 6 wt.% detected on the glass front panel
V-cell with subcell	S8	No indium detected on the glass back No indium detected on the glass front	No indium detected on the glass back panel Low levels of indium up to 5.7 wt.% detected on the glass front panel

of approximately 70–100 µm were observed. These layers were believed to be the polarisation and passivation layers on the external surfaces (termed external layers) of each substrate.

As the internal surface of the glass front and glass back substrates contain the transparent electrodes, high magnification images of these internal surfaces were taken. In most cases, high magnification images of the internal surfaces of the glass panels revealed the presence of thin coatings. The findings are presented in Table 2.7.

The above analysis has captured the range of coatings present on the surfaces of the samples and illustrated the large variations in coating thickness, which will have a significant effect on the calculation of the volume of the coating present on any liquid crystal panel. It is important to understand the variation and level of accuracy that can be expected when trying to predict the quantity of transparent electrode material present in an average panel in order to determine the maximum recoverability that can be expected from a recovery process. Even liquid crystal panels from the same manufacturer, as in samples S6 and S7 (Sony) and samples S8 and S9 (Philips), showed a large range of variation in the presence of the transparent electrode.

2.4.4 Quantification of indium concentrations on the liquid crystal panel internal surfaces

The ICP-OAS was conducted on three of the samples that were sent to IDO Labs, Germany for testing. The results showed a large disparity of indium presence in the samples demonstrating indium levels of 60, 181 and 255 mg/kg glass from liquid crystal front glass substrates. This confirmed that a significant disparity in indium levels does exist and this correlates with the large differences in indium reported from different authors. In order to investigate this further, the full set of 10 samples were analysed in-house under AAS to further understand the level of variation regarding indium concentrations.

Table 2.7. Readings and observations from the cross-sectional analysis of the internal surfaces of the liquid crystal glass substrates

Sample S1 revealed the presence of a coating of approximately 1.58 µm thickness on the internal surface of the glass back substrate; however, the coating was not uniform across the surface and was only present in specific locations. This was believed to be owing to the pattern of the transparent electrode on the internal surface of the glass as illustrated in the surface optical images. However, a high magnification image of the internal surface of the glass front substrate revealed the presence of a more uniform layer of thickness 1.86 µm
Sample S2 revealed the presence of a coating on the internal surface of the glass back substrate with a thickness of 1.56–1.8 µm, again with an intermittent pattern on the surface; the glass front surface showed a varying thickness coating of 1.1–2.12 µm
Sample S3 showed an intermittent pattern of both coatings present on the internal surface of the glass back and front substrates, with a thickness of 1.86 µm and 1.54 µm, respectively
Sample S4 showed more uniform coatings present on the internal surface of both front and back substrate with a thickness of 1.65 µm and 1.67 µm, respectively
Sample S5 displayed thicker coatings on the internal surface of both back and front substrates of 2.29 µm (intermittent coating) and 2.46 µm (uniform coating).
Sample S6 displayed a mixed thickness of coatings with 1.47 µm (intermittent coating on back substrate) and 2.46 µm (uniform coating on front substrate)
Sample S7 also displayed a mixed thickness of coatings with 1.48 µm (intermittent coating on back substrate) and 1.47 µm (uniform coating on front substrate)
Sample S8 displayed an intermittent coating on both substrates with a thickness of 1.13 µm for the back substrate and 1.56 µm for the front substrate
Sample S9 displayed a continuous coating on the back substrate with a thickness of 0.84–1.13 µm and an intermittent coating on the front substrate of 1.56 µm
Sample S10 showed a uniform coating on both substrates with a thickness of 1.15 µm for the back substrate and 2.27 µm on the front substrate

The AAS corroborated the surface SEM and EDS results and trends, with indium concentration always being higher on the glass front and lower on the glass back panels of the liquid crystals. Of the 10 samples, the highest indium concentration on the glass front substrate was observed at 292 mg/kg glass and the lowest was 38 mg/kg glass, while for the back substrate the highest reading for indium concentration was 162 mg/kg glass and the lowest reading showed no indium present in the sample. The V-cell structure panels displayed the lowest indium readings, while the more common rectangular cell structure showed the highest indium concentrations (see Table 2.8).

2.4.5 Detailed characterisation of liquid crystal panel internal surfaces

XRD analysis

Plain rectangular cell structured samples S1 and S5 both showed significant peaks at approximately 31 degrees 2θ, with sample S1 exhibiting additional dominant peaks at 40 and 43 2θ angles. Sample S5 exhibited a similar pattern to the reference pattern 01–088–0773 for $In_4Sn_3O_{12}$ with the dominant peak at 31 degrees 2θ. However, while sample S1 had a significant peak at 31 degrees 2θ, it exhibited its dominant peak at 42 degrees 2θ. In comparison with the AAS indium concentration results, these samples for both glass front and glass back revealed relevantly high indium levels above 90 mg/kg glass in all cases. The EDS reading had previously detected molybdenum in both samples at relevantly high concentrations, while S5 also had strontium present, which may account for the variation seen here. The glass front substrate revealed very similar patterns to the results for the glass back substrates.

Subcell rectangular structured samples S2, S3, S6, S7 and S9 all showed dominant or significant peaks at the higher peaks of 40 and 43 degrees 2θ with a minor peak around 31 degrees (except S3 where the 31 degree peak was dominant) for the glass back substrate. All these samples showed significant levels of indium on the glass back at approximately 65–90 mg/kg glass. The lower indium concentration may account for the change observed in the dominant peak, while the lower peaks were still present at the 31 degree 2θ angle. On the glass front, the traditional pattern was visible with the dominant peak at the 31 degree 2θ angle and these correlated with the AAS results where indium levels were approximately 230–250 mg/kg. However, sample S4, which also had a subcell rectangular cell structure, showed a different

Table 2.8. Structures and observations revealed on liquid crystal glass panels

Structure	Samples	Observations
Rectangular cell structure	S1, S5	S1 revealed similar concentrations on GF and GB (approximately 160 mg/kg for both) while S5 showed variation with 275 and 91 mg/kg for GF and GB, respectively
Rectangular cell with subcell structure	S2, S3, S4, S6, S7, S9	These samples showed a very similar and consistent trend with high indium readings on the GF and lower readings on the GB. The highest indium reading was 292.3 mg/kg glass for sample S6 on the GF and most of the samples ranged from 230–250 mg indium/kg glass front. On the glass back, the highest reading was 129 mg/kg for sample S2, while most samples were within the range 65–90 mg/kg glass back. Sample S4 was the only sample not to follow the above trends, exhibiting 38 mg indium on the GF and no indium on the GB, perhaps suggesting the use of an alternative transparent electrode. The EDS reading for this panel had revealed a very high silicon reading on the GB, presumably from the glass substrate
V-cell structure	S10	The S10 sample revealed low indium levels on both the glass front and the glass back of 46 and 34 mg indium/kg glass, respectively. This can be compared with the EDS results, where indium had not been detected on the GF and only low levels on the GB. Titanium and magnesium as well as aluminium and silicon had been detected in significant concentrations
V-cell with subcell structure	S8	This sample showed very high indium levels on the GF of 285 mg/kg glass and very low levels on the GB of 14 mg/kg glass. The EDS results again had not managed to detect indium on the GF and had picked up high silicon levels presumably from the glass beneath as well as aluminium, nickel and copper. While the EDS on the GB had low levels of indium and also the presence of zinc and arsenic suggesting the presence of an alternative electrode composition

GB, glass back; GF, glass front.

trend with a single dominant peak at 44 degrees 2θ. This trend corresponded well with the AAS results where indium levels were much lower than the other subcell rectangular samples, with 38.5 mg indium on the glass front substrate and no indium present on the glass back substrate.

The plain V-cell structured sample S10 showed an identical 44 degree 2θ peak to the S4 sample. This sample also exhibited similar low indium concentrations of 46.2 and 34.6 mg/kg in the glass front and glass back substrates, respectively. From this it can be deduced that low indium levels (below 90 degrees 2θ) were correlating in their XRD pattern with a dominant 44 degree 2θ peak. Sample S10 in the EDS results also had significant concentrations of aluminium, titanium and magnesium. It is likely that this sample contained an alternative transparent electrode composition that was based on indium oxide. However, the XRD pattern of the V-subcell structured sample S8 detected high levels of noise during the scan on both the glass front and the glass back substrate.

FIB analysis

Two samples, S1 and S2, were selected for characterisation by FIB spectroscopy. A FIB mill was made into the sample on one of the main rectangular cell structures. Platinum coating was deposited to keep the surface intact during milling. Micrographs of sample S1 of both glass front and glass back displayed the platinum layer strip on top and the ITO layer underneath. On the glass back panel sample, the ITO layer appeared as a continuous layer, being within one of the cells where the ITO was present for this rectangular structured panel. Sample S2, which had the rectangular cell with a subcell structure, displayed a similar micrograph and result to sample S1. The subcell structure was not evident in the FIB micrograph. The FIB micrographs did not reveal any other characteristic details of the sample and grain boundaries and/or texture/orientations were not evident.

2.5 Conclusion

Comprehensive analysis of 10 different liquid crystal panels was undertaken. The specific objective of this section of the research, as set out previously, was to investigate the presence and amount of indium contained in the liquid crystal panels of LCDs. The findings detailed the structure of the panels from rectangular cells to V-cells; the location of the transparent electrode as residing predominantly within the subcells of the structured patterns on the glass back, while residing more uniformly across the glass front panel; the variation in transparent electrode thickness in different LCDs from 1.5–3 μm; the concentration levels of indium present correlating with the structures observed, where plain rectangular cell panels had high indium levels from 90–275 mg/kg glass, whereas the subcell rectangular panels generally exhibited more consistent levels of indium, with higher levels on the glass front of 230–250 mg/kg and 65–90 mg/kg on the glass back. This was consistent with the microscopy results, which showed an intermittent transparent electrode layer on the glass back and a uniform one on the glass front. The less common V-cell structures generally exhibited lower concentrations of indium present, and the presence of other elements such as zinc, arsenic, titanium, aluminium and magnesium indicated a strong potential for alternative electrode compositions.

It is clear that a significant amount of variation exists between different LCDs, even those produced by the same manufacturers, and that any recovery process for indium will have to acknowledge that key parameters such as intake indium concentration, the location and presence of other metals will affect expected recovery rates. Examination of the samples revealed a pattern to the indium levels present that correlated with the cell structure. It has also been shown that XRD analysis can be a useful tool to indicate both the cell structure and the indium levels by correlations of the patterns with the typical structures.

This knowledge may be used to optimise the recovery process by designing tailored preparation techniques, for example, splitting the glass back and the glass front in cases where indium is generally more concentrated on the glass front; a maximum time for dissolutions related to ITO thickness; the grinding, milling or shredding of panels before treatment; an optical pre-check of screens to identify structure (XRD) and likely indium concentration range. Furthermore, characterisation of a wider sample set using specific techniques (optical, AAS, cross-sectional analysis) would allow the formation of a significant database that could guide the recycler on optical recovery strategies for indium from LCDs.

3 Review of Indium Recovery Techniques

3.1 Objectives

The objective of this section of the research was to undertake a review of the best available techniques (BATs) applicable to indium recovery from waste LCD displays. This involved a review of current state-of-the-art techniques reported from industry, patents and/or publications. A comparison of the techniques was undertaken regarding reported efficiencies for indium recovery achieved by various processes.

3.2 Introduction to Indium Recovery Techniques

Indium recovery techniques generally come under the umbrella of hydrometallurgy, which is defined as a technique within the field of extractive metallurgy involving the use of aqueous chemistry for the recovery of ores, concentrated and recycled or residual materials. Hydrometallurgy is typically subdivided into three general areas, in order of application (see Figure 3.1):

1. leaching;
2. solution concentration and purification;
3. metal recovery.

Leaching is an extractive metallurgical step that involves immersing the source material in an aqueous media to convert the metal into a soluble salt. The leaching step strips the indium metal in the source material of an electron, which allows the metal ion to go into solution.

The second step, concentration and purification, is to concentrate the indium metal in solution and to rid it of any impurities; this step ultimately determines the efficiency of the process. The concentration step can involve selective precipitation, cementation, solvent extraction, ion exchange or electrolysis (Ogi *et al.*, 2012). All of these separation methods can be used in integrated processes in the hydrometallurgical industry and they can be repeated a number of times depending on the complexity of the source material and the desired purity of the final product. Despite their widespread use in the recovery of valuable material, it must be noted that none of these concentration steps have 100% efficiencies. Furthermore, there are several other aspects to be considered when using these concentration methods, such as:

- energy costs;
- chemical costs;
- toxic by-products;
- capital costs.

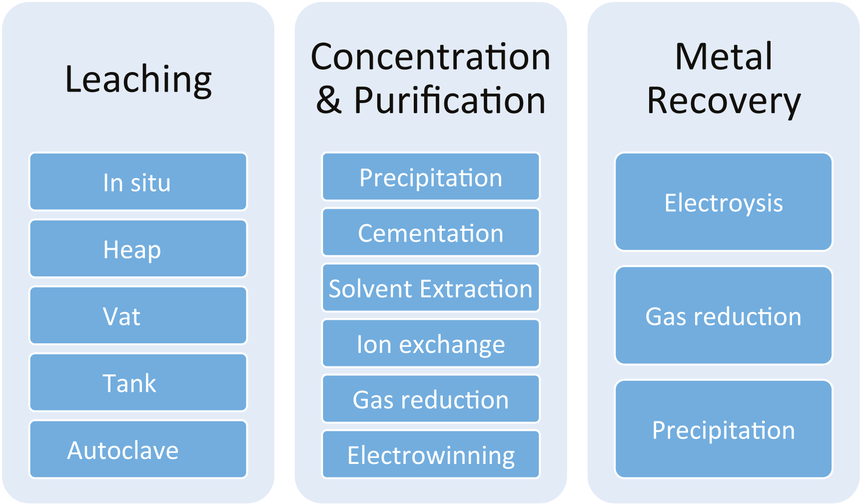


Figure 3.1. Summary of hydrometallurgy techniques.

The metal recovery phase is the final step in the hydrometallurgical process and metals suitable for supply as a raw material are directly produced from this step. Sometimes, however, further refining is required if ultra-high purity metals are required. The primary types of metal recovery processes are electrolysis, gaseous reduction and precipitation.

3.3 Comparison of Indium Recovery Techniques Reported in the Literature

In the recovery of valuable metal from waste electronics, the two most important hydrometallurgical separation methods are solvent extraction and ion exchange, of which solvent extraction is the most widely studied. Many different solvent extraction studies have been developed because this technique allows the recovery of very trace amounts of indium from a wide variety of sources, such as flue dust, leaching solutions, slags and residues.

3.3.1 Solvent extraction

In solvent extraction there are two solvent phases, an aqueous phase and an organic stripping phase, with both phases being immiscible in each other. The two phases are forced to mix through applied agitation (emulsification) and this enables any solutes to migrate from one solvent to the other. When the agitation is removed the molecules will preferentially migrate to the solvent where they have the greatest solubility. If the molecules are very polar they will generally favour the aqueous phase but if they are non-polar they will favour the organic phase.

The solvent extraction process can be generalised as follows.

1. The first step requires the use of a suitable complexing agent. This complexing agent is added to the aqueous phase to ensure that the extractable complex is of sufficiently low charge density to maximise the transfer of atoms to the organic phase.
2. The agitation step is critical in ensuring that the equilibrium separation is achieved. The two phases completely physically separate out from one another. The polar molecules remain in the aqueous phase and the non-polar molecules remain in the organic phase.
3. The final step is to extract the top aqueous phase and to recover the desired non-polar molecules from the organic phase.

The most widely reported method used to extract indium metal from waste electronic and electrical equipment (WEEE) is solvent extraction (SX). This approach has been applied to the recovery of indium from LCDs and examples of the effectiveness of this approach are shown in Table 3.1. The biggest attraction of this technique is the very high recovery rates achievable. Another advantage is the wide variety of solvents, acids and chelating systems developed for the recovery of indium.

Some of the main extractant types (aqueous phase, sometimes referred to as "leach liquor") used in

Table 3.1. Effectiveness of the solvent extraction approach reported in the literature

Author	Recovery source	Details	Efficiencies obtained
Yang *et al*. (2014)	LCDs	Acid/SX (D2EHPA) in kerosene	>95%
Yang *et al*. (2014)	LCDs	Acid/SX (D2EHPA) in kerosene	>98
Kato *et al*. (2013)	LCDs	Acid/SX/chelant	96.70%
Yang *et al*. (2013)	ITO etching waste cake	Aqua regia/SX/in kerosene	90%
Ruan *et al*. (2012)	LCDs	Liquid N_2/Acid/SX (D2EHPA)	97%
Yang (2012)	LCDs	Mixed acids/SX	Not given
Kang *et al*. (2011)	ITO etching waste cake	NaOH/SX/electrorefining	100%
Virolainen *et al*. (2011)	LCD waste	SX/ion exchange	Not given
Kang *et al*. (2011)	LCDs	SX (PC88A)	99.997%
Nimshihaa *et al*. (1999)	Indium residue from zinc tailings	Leach/SX	98.90%

D2EHPA, di-(2-ethylhexyl)phosphoric acid; liquid N_2, liquid nitrogen; NaOH, sodium hydroxide; PC88A, 2-ethylexyl hydrogen 2-ethylhexyl phosphonate.

solvent extraction are outlined in Table 3.2. The large number of aqueous phase extractants developed is representative of the large amount of research carried out into the use of solvent extraction as a method for metal recovery, and indium recovery in particular. The most reported on extractant in the literature for indium recovery is D2EHPA.

A similarly large number of organic reagents have been employed for the recovery of indium (see Table 3.3). The main drawback with solvent extraction is that it is a potentially polluting technology owing to the involvement of organic solvents. However, some solvent extraction systems are more polluting than others and the replacement of toxic phosphorous compounds with specific chelating extractants such as hydroxyoximes or β-diketones would be an important contribution to better industrial waste management (Paiva, 2001).

The literature on solvent extraction of indium is large and complex and reviewing all the different phases and stripping processes is a considerable undertaking. Paiva (2001) produced a comprehensive literature review on the various phases and their performance and these are detailed in Tables 3.4–3.6.

The widely reported use of solvent extraction for the recovery of indium allows comparison of the settings and recovery rates for a multitude of reagents (see Table 3.7). It is apparent that there is a significant amount of variation in settings between the various reagents used. It is also evident that very high recovery rates can be achieved using a variety of different reagents, provided that the most suitable parameter settings have been established.

Most papers published on solvent extraction used a pre-mechanical treatment step in an effort to increase the efficiency of the extraction process. Some of these mechanical techniques are outlined in Table 3.8, along with the process considerations associated with them.

Rocchetti *et al*. (2015) presented a cross-current leaching technique to mobilise indium from crushed glass panels of end-of-life LCDs (fragments). Cross-current leaching involves carrying out a series of treatments to leach indium in sulfuric acid at 80°C for 10 minutes to mobilise the indium. They filtered the leach liquor produced in the initial leaching step and used it to treat a second amount of LCD fragments, adding a small amount of fresh H_2SO_4 to restore any amounts lost due to the high temperature of the process. The authors reported that the cross-current leaching allowed for the concentration of indium, saving of reagents required, and reduced the emission of CO_2 due to the recovery of indium. In a subsequent publication, Rocchetti *et al*. (2016) reported that they had followed the cross-current leaching step with a cementation process to recover the indium. Cementation with zinc powder was reportedly optimised through the investigation of the effects of different variables (zinc concentration, pH,

Table 3.2. Main extractant (aqueous phase extractants) types used in the solvent extraction of indium

Extractant type	Examples	Author
Carboxylic acids	CA-12, CA-100	Zhao *et al*. (2003)
Phosphoric acid derivative	D2EHPA	Ruan *et al*. (2012)
Chelating compounds	Hydroxyoximes, azoles	Kato *et al*. (2013)
Solvating extractants	TBP, TOPO, MIBK	Yang *et al*. (2013)

D2EHPA, di-(2-ethylhexyl)phosphoric acid; MIBK, methyl isobutyl ketone; TBP, tri-*N*-butylphosphine; TOPO, trioctylphosphine oxide.

Table 3.3. Chemical structure of some common reagents (organic phase) used in the solvent extraction of indium

Reagents	Chemistry	Author
TOPO	Trioctylphosphine oxide	Yang (2013)
Cyanex 923	Mixture of four trialkyl-phosphine oxides	Yang (2013)
TBP	Tri-*N*-butylphosphine	Virolainen *et al*. (2011)
Aliquat336	*N*-Methyl-*N*,*N*,*N*-trioctyloctan-1-ammonium chloride	Inoue and Alam (2015)
TIOA	Triisooctylamine	Inoue and Alam (2015)

Table 3.4. Extractants used for indium extraction from aqueous solutions (data from Paiva, 2001)

Organic extractant phase	Aqueous phase	Final stripping phase	Additional comments
Fatty acids (C_{11}-$C_{15}CO_2H$)	Aq. H_2SO_4	n/a	Extracted complex [In2(OH)L5.5HL]x
Saturated fatty acids	Diluted solutions from Zn or Pb refinery residues	H_2SO_4 or HCl (pH 3–5)	Fe(III), Ga, Sn co-extracted; kerosene used as diluent
Versatic 10 (carboxylic acid)	Zn residue treatment, aq. H_2SO_4	n/a	In, Ga, Sn co-extracted: kerosene used as diluent
Versatic 10, 2-bromodec-anoic,naphtenic, 3, 5-diiso-propylsalicylic acids	Aq. $HNO_3 + NaNO_3$ or aq. HCl + NaCl	n/a	In and Ga selectively recovered
2-bromodec acid	Aq. H_2SO_4	Aq. H_2SO_5	Several data on metal ion interferences in In extraction (Cu, Zn, Cd, Hg, Pd, Fe(III), Al, Ga, Ti)
Di(2-ethyl-hexyl) phosphoric acid (D2EHPA)	Aq. H_2SO_4	H_2SO_4 or HCl	In, Ga selectively recovered against Zn and Al; supported liquid membrane
Di(2-ethyl-hexyl) phosphoric acid (D2EHPA)	Acidic sulfate solutions	Aq. HCl	Fe(III), Sn, Pb, Zn, Sb co-extracted; As not extracted. Stripping phase purified with TBP
Di(2-ethyl-hexyl) phosphoric acid (D2EHPA)	Sulfuric liquor from Zn processing, aq. H_2SO_4	Aq. H_2SO_5	Fe(III) co-extracted, but good In/Fe(III) concentration factor
Di(2-ethyl-hexyl) phosphoric acid (D2EHPA)	Zn leach residues	n/a	Some Fe(III) co-extracted, synergistic mixture with Y100
Di(2-ethyl-hexyl) phosphoric acid (D2EHPA)	Aq. H_2SO_4, HCl, H_2PO_4	HCl, H_3PO_4	In from H_2SO_4: good separation factors against Fe(III), Zn and Al: In separated efficiently from As and Ge
Di(2-ethyl-hexyl) phosphoric acid (D2EHPA)	Aq. $HNO_3 + HCl$	–	IR and NMR studies
Diisostearyl phosphoric acid	–	–	In selectively separated from Ga; supported liquid membrane
Di-*p*-octylphenyl phosphoric acid	Aq. H_2SO_4	–	Co-extraction of Ga: benzene as diluent
Mono(iso-ocatdecyl) phosphoric acid	Aq. HCl		Ga, Fe(III) co-extracted
2-ethylhexyl phosphonic acid	Aq. H_2SO_4	Aq. HCl or H_2SO_4	IR and NMR studies
2-ethylhexyl phosphonic acid	Aq. $HCl + HNO_3$	–	IR and NMR studies
Diphosphonate ligands	Aq. H_2SO_4	Aq. HCl	Ion exchange resin; co-extraction of Ga
Bis(2-ethylhexyl) phosphinic acid	Aq. H_2SO_5	Aq. H_2O_4, other acids	Fe(III) co-extracted
Bis(2-ethylhexyl) phosphinic acid	Aq. HNO_3	–	–
Bis(2,4,4-trimethylpentyl) phosphinic acid	Aq. HNO_3, HCl-H_2SO_4 mixed	–	–
Bis(2,4,4-trimethylpentyl) phosphinic acid	Aq. HCl, H_2SO_4, or mixed	Aq. HCl	Separation from Cd by stripping with HCl

Aq., aqueous; D2EHPA, di-(2-ethylhexyl)phosphoric acid; IR, infrared; NMR, nuclear magnetic resonance; TBP, tri-*N*-butylphosphine.

cementation time) on cementation efficiency and purity of the solid product. The authors reported that the highest purity of the indium product (62% indium in the solid product, calculated in the 4-metal system indium–aluminium–calcium–iron) was achieved after a cementation of 10 minutes, whereas the presence of impurities increased with time. The authors are currently investigating the process further with the aim of increasing the purity of indium and improving the environmental sustainability of the process.

Table 3.5. Chelating acidic-type extractants used for indium extraction from aqueous solutions (data from Paiva, 2001)

Extractant	Aqueous phase	Stripping phase	Additional comments
1-phenyl-3-methyl-4-acyl-5-hydroxy-pyrazoles	$HClO_4$ or $NaClO_4$, nitrate chloride	–	Synergistic mixtures with solvating extractants, TBP, TOPO, MIBK, etc.
3-hydroxy-2-methyl-4-pyridone derivatives	HNO_3/HCl/NaCl	–	UV to follow formation of complexes
Acetylacetone benzoyl acetone	$NaClO_4$	–	Good separation from Ga and Al. Synergistic effect with di-chlorophenol
2-thenolyl trifluoro-acetone	Buffers	–	Synergistic mixtures with TOPO
4-acyl-3-phenylisozazol	Aq. HNO_3 and HCl	–	Synergistic mixture with TOPO
N-nitroso-*N*-*p*-octadecyl-phenylhydroxyl-amine ammonium salt	Aq. HCl and buffers	–	Transport through impregnated polypropylene membrane
N-alkyl-carbonyl-*N*-phenyl-hydroxyl amines	–	–	Separation of In, Ga, Al
4-isoproyl tropolone	Aq. $NaNO_3$	–	Chloroform as diluent
Pseudo calixarenes (PCA)	Aq. chloride (pH 3–5)	–	–
Hydroxyoxime derivated such as LIX63, 984 or 973N	Aq. HCl, buffers, Ag. sulfate	22, 73–75	LIX 984 selectively separated In from Zn and Pb

Aq., aqueous; MIBK, methyl isobutyl ketone; PCA, principal components analysis; TBP, tri-*N*-butylphosphine; TOPO, trioctylphosphine oxide; UV, ultraviolet.

Table 3.6. Solvating-type extractants used for indium extraction from aqueous solutions (data from Paiva, 2001)

Extractant	Aqueous phase	Stripping phase	Additional comments
Tri-*N*-butyl phosphate (TBP)	Aq. HCl	Aq. HCl	Separation factor: interference of several ions, supported liquid membrane
Tri-*N*-butyl phosphate (TBP)	Aq. bromide, iodide, thiocyanate	–	–
Tri-*N*-octyl phosphone oxide (TOPO)	Aq. HCl	–	IR, IR and NMR studies
Tri-*N*-butyl phosphone oxide (TBPO)	Aq. HCl	–	Several organic diluents
Triphenyl phosphine oxide (TPPO)	Aq. HCl	–	In is extracted but not separated from Ga and Ti
Cyclohexanone	Aq. bromide, iodide, HBr, H_2SO_4	–	–
Methylisobutyl ketone (MIBK)	Aq. chloride, iodide, HBr, H_2SO_4	Aq. HNO_3	Interference of Sn, Bi, Sb, Ga, As and other metal ions
Mesityl oxide	Aq. HCl and HBr	–	Sb(III), SCN^- and $C_2SO_4^{2-}$ interfere; separation and determination of Ga, In and Al
Diethylether, diisopro-pylether	Aq. HCl and HBr	6M HCl	Raman studies
Tris(2-ethy-hexyl phosphate)	Aq. sodium salicylate	H_2O	Separation and determination of Ga, In and Al
Triphenylarsine oxide (TPASO)	Aq. sodium salicylate	H_2O	Separation and determination of Ga, In and Al

Aq., aqueous; IR, infrared; NMR, nuclear magnetic resonance.

Table 3.7. Comparison of reported settings and efficiencies of various solvent extraction indium recovery techniques

Main reagent/material	°C	pH	Pressure (kPa)	Recovery (%)	Author
LZX973N	20	3–4	Atm.	95.0	Alguacil (1999)
Chromosorb 108 resin	25	8–9	Atm.	95–100	Tuzen and Soylak (2006)
Chelating agent/supercritical CO_2	60–70	2–3	13790	90.9	Chou and Yang (2008)
Nanofiltration membranes	5–40	6–8	460–1470	95.0	Wu *et al.* (2004)
D2EPHA	25	1.4	Atm.	89.7	Lee *et al.* (2002)
P507 extraction resin	10–40	2.0	Atm.	99.6	Liu *et al.* (2006)
P204	25	0.4	Atm.	90.0	Gao *et al.* (2010)
$Na_5P_3O_{10}$	25–65	2.6	Atm.	97.0	Jiang *et al.* (2011)

Atm., atmosphere; D2EHPA, di-(2-ethylhexyl)phosphoric acid; kPa, kilopascal.

Table 3.8. Main mechanical processing steps employed in the recovery of indium

Mechanical treatment	Considerations	Author
Conventional milling	Electricity/equipment/time	Terakado (2011)
Shredding	Electricity/equipment/time	Li *et al.* (2009)
High-impact ball milling	Electricity/equipment/time	Lee *et al.* (2013)
Mechanochemical milling	Electricity/equipment/chemicals/time	Hasegawa *et al.* (2013)
Roll milling	Electricity/equipment/time	Dodbiba *et al.* (2012)

3.4 Comparison of Indium Recovery Techniques Reported in Patents

The indium recovery techniques found in patents are similar to those found in scientific journals. Unsurprisingly, given the number of systems developed, solvent extraction techniques are the most patented technology with claims of up to 100% indium recovery rates (see Table 3.9).

Patents on the recovery of indium specifically from TFT-LCDs reveal a mix of techniques have been proposed including microbe-technologies, pyrolysis and roasting. The sources of indium are mainly waste LCDs or ITO etching waste (see Table 3.10).

Table 3.9. Patents based on the recovery of pure indium metal from ore and leached solutions

Patent number (year)	Recovery source	Details	Efficiencies obtained
US 1912590 (Murray, 1933)	Ore	Roasting/leaching/neutralising/rendering	not given
US 3180812 (Beau, 1965)	In_2Cl_3	Solvent extraction	not given
US 4292284 (Tomii and Tsuchida, 1981)	Leached solutions	Solvent extraction	100%
US 5019363 (Le Quesne and Fossi, 1991)	Leached solutions	Solvent extraction	99%
EP 1905855 B1 (Kita *et al.*, 2007)	Indium material	HCl acid extraction	not given
US 20110132146 A1 (Miki *et al.*, 2011)	Etching waste	Precipitation-inducing metal	90%
US 8834818 B2 (Ferron, 2014)	Recycling scrap	Leaching/solvent extraction	not given
WO 2014084445-A1 (Lee *et al.*, 2014)	Waste solutions	Solvent extraction/selective absorption	87%

Table 3.10. Patents based on recovery of indium from LCD-flat panel displays (LCD-FPDs) and ITO waste

Patent number (year)	Recovery source	Details	Efficiencies obtained
CN 102002591 A (Weiqing, 2010)	Waste LCDs	Leaching	99%
EP 2722409A1 (Konishi *et al*., 2014)	ITO etching waste	Leaching/microbe	Not given
CN 103602815 A (Xiaoyong *et al*., 2014)	LCD waste	Leaching/back extraction	99.13%
CN 102207640 A (Ruisheng, 2014)	LCDs	Ageing via roasting	Not given
CN 102671921 B (Ming and Xin, 2014)	LCDs	Pyrolysis/reduction	>90%
CN 102002592 A (Weiqing, 2011)	LCDs	Leaching/extracting agent	Not given
CN 103191903 B (Zhifeng *et al*., 2015)	ITO etching waste	Leaching/evaporation	Not given

3.5 Novel Indium Recovery Methods with the Potential for Use as a Best Available Technique

3.5.1 Novel indium recovery techniques from the literature

Choi *et al*. (2014) successfully recovered over 90% of ITO from TFT-LCD panel scraps using the "lift-off" method outlined below. The authors concluded that this technique could easily be applied to the large-scale recovery business dealing with large LCD panels from TVs and other displays (see Figure 3.2).

The lift-off method involves the dissolution of the colour filter (CF) layer using an alkaline mixture of KOH and NaOH at 80°C for 30 minutes, which allows the ITO layer to be separated from the glass substrate in a non-destructive manner. One prerequisite for this method is that the TFT-LCD is first pre-disassembled in a controlled manner. Choi *et al*. (2014) described the use of a manual edge-cutting method to pre-separate the TFT-LCD into upper and lower layers. One big advantage of this method is that it does not have the energy costs associated with mechanical pre-treatment of the LCD screen and it also avoids mixing

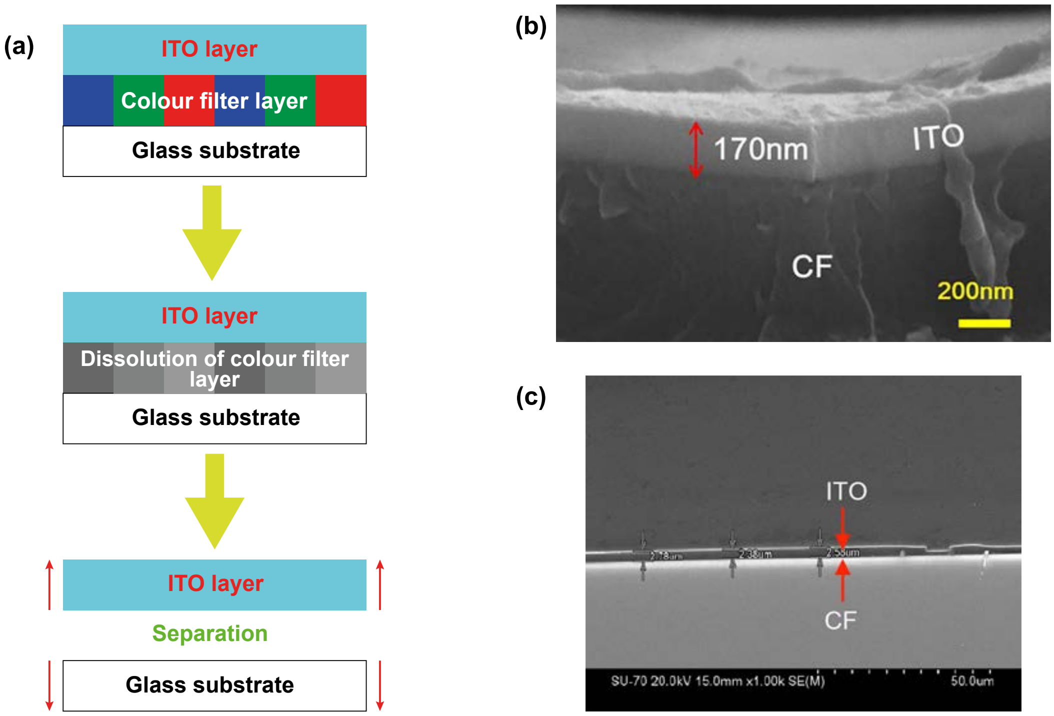


Figure 3.2. The lift-off technique outlined by Choi *et al.* (2014) showing (a) the sequential steps involved; (b) the thin (170 nm) layers of ITO recovered; (c) the much thicker ITO layers (3 µm) measured on LCD-TFTs by O'Donoghue *et al.* (2014).

the glass and ITO with other materials. The removal of this step greatly reduces the complexity of the overall recovery process as well as avoiding needless energy use.

Yoshida *et al*. (2014) investigated the recovery of indium from TFT and CF glass using subcritical water. This type of water is actually harmless water that is heated to temperatures between its boiling point (100°C) and its critical point (374°C) but under pressure to suppress vaporisation. Under these conditions the water acts as a very good solvent for hydrophobic matter and the magnitude of the ionic product of water increases threefold compared to room temperature. Yoshida *et al*. (2014) reported that these properties of subcritical water are advantageous for hydrolysis and decomposition of organic compounds including polymeric films. The authors claimed that this technique had never before been applied to the recovery of indium and that it would provide a novel method without the need for chemical treatment and without destroying the glass substrate. The approach achieved 83% recovery of indium from the colour filter and 7% from the TFT glass. In their experimental procedure, the authors broke the colour filter glass into 5–10 mm size pieces in order to fit it in the reactor. However, a much larger reactor would be required to avoid the mechanical destruction of the glass.

Yoshida *et al*. (2015) further developed this technique for the recovery of indium from TFTs and colour filter glass from LCD waste. The authors combined the subcritical water approach with the addition of NaOH, with the joint aims of increasing the recovery rate of indium and reducing the reaction temperature. The results showed a significant improvement in indium recovery rates from colour filter glass, with an increase from 83% at 360°C without NaOH to over 99% at 160°C with NaOH. Recovery rates for the TFT glass showed dramatic improvements using NaOH, going from 10% without NaOH to 95% with NaOH at the lower temperature of 220°C. Since no indium dissolved in the liquid phase, this method demonstrates a considerable advantage over acid dissolution and ion-exchange methods. Rapid recovery rates of only 5 minutes offset the relatively high temperatures involved. The halving of processing temperatures with the addition of NaOH would represent a large saving in energy costs. The relatively cheap and easy to obtain chemicals used in this process make this method a potentially economically viable technique for the recovery of indium from TFT-LCDs.

Another promising technology that is a potential alternative to the established hydrometallurgy processes is "biohydrometallurgy". This technology exploits microbiological processes for the recovery of heavy metal ions. Jadhav and Hocheng (2015) outline how this method has been applied to the recovery of heavy metal ions from various sulfide minerals or low grade ores. Biohydrometallurgy has already been exploited commercially for many years for metal recovery, with Brierley (2008) reporting that bioleaching has been successfully and commercially applied in biohydrometallurgy for extracting copper and precious metals from low-grade ores and tailings. Brierley claimed that biotechnology achieves metal recycling by processes similar to that in nature and therefore is environmentally friendly with low costs and low energy requirements. Although still mainly at lab scale, this technology has been shown by Brierley to achieve high extraction yields in excess of 90%. Jadhav and Hocheng (2015) state that there is a need to advance commercial application of biohydrometallurgical processes.

The application of biohydrometallurgy to the bacterial leaching of metals from electronic scrap was examined by Willner and Fornalczyk (2013). They aimed to exploit the natural ability of microorganisms to transform solid metals present in waste into a dissolved form. This so-called "bioleaching" process is cheaper and less complex than conventional recovery techniques. It is also considered to be an incredibly flexible technology in that microorganisms are very adaptable to both changing and extreme living conditions. Willner and Fornalczyk (2013) reported laboratory results on the recovery of metals from e-waste using biohydrometallurgical methods with a particular emphasis on copper. They achieved isolation rates of copper at 98–99% at low doses (20%) of bacteria. They concluded their research by saying that further studies are needed to optimise bioleaching from electronics, not only for copper, but also for other useful metals.

Namias (2013) compared pyrometallurgy, hydrometallurgy and biometallurgy for the processing of electronic waste, finding that the recovery of valuable components from e-waste has been gaining popularity over the years and that bioleaching and biosorption are the two main methods developed. Currently, research in this field is focusing on finding the most effective organism for the bioleaching process. Kamberović *et al*. (2011) reported that the benefits of processing e-waste using biometallurgy are low operating costs, minimisation of hazardous chemicals and high efficiencies in detoxifying effluents. A review by Willner *et al*. (2015) listed studies conducted on WEEE containing metals such as gallium, aluminium and zinc using bioleaching and a variety of microorganisms. Although the recovery of indium from FPD-LCDs using biometallurgy has not yet been reported on, the VTT Technical Research Centre of Finland apparently used fungi to filter gold from mobile phones with more than 60% gold recovered compared to 10% using more commonly used harmful chemicals (www.urbanmining.org).

3.6 Comparison of the Viability of Various Indium Recovery Techniques

A review of indium recovery processes in actual operation or being undertaken commercially reveals the following:

- The American company, Indium Corporation, offers reclaiming services of indium, germanium, gallium and tin for environmental and/or economic purposes. They reclaim materials such as: ITO, IZO, gallium indium zinc oxide (GIZO), spent targets and other leftover materials from bonding, sputtering and other processes. They state that most elements can be reclaimed into finished metallic or compound form. The recovery processes are reported to be conducted at their facilities in the USA and South Korea.
- The French company, Coved, has reported the recovery of indium from LCD screens, working in conjunction with Camille SA and the IFTS (French Institute of Filtration and Separation Techniques). Coved is developing an industrial-scale prototype facility for the recycling of indium, which will recycle between 30 and 40 tonnes of indium every year.

However, detailed information or knowledge of the processes being used for this indium recovery is not available. It can be observed that the commercial uptake of indium recovery is generally low.

Aside from the conventional routes for recovering metals from WEEE material, there are also some novel and innovative technologies reported in the literature that could potentially be adapted and commercially exploited for the recovery of indium metal from WEEE material. Successful recycling techniques and processes need to be cost-effective and compliant with relevant legislation. High recovery rate technologies involving solvent extraction and electrochemistry are widely reported to provide high recovery yields but these processes are also complex, costly and environmentally unfriendly. Alternative novel technologies have been reviewed in this BAT report that can minimise the use of chemicals and high-energy mechanical processes.

This review allowed a comparison between the various potential technologies available for the recovery of indium from LCD displays. Table 3.11 outlines the technology reviewed in this report and compares their individual potential merits.

The review highlights that while well-developed techniques such as solvent extraction and ion exchange are well established, the market take-up for indium recovery from LCDs is low (only a small number of companies report recovering indium). This correlates with the fact that the economic and environmental aspects of set-up and operation of these processes is significant and an investment into a technique or process to recover indium requires a clear understanding of the medium to long-term markets for indium and the potential to capitalise on the investment. While novel techniques have been proposed and attempt to address different aspects individually, including cost, complexity and time taken for indium recovery, the recycling market has yet to take up any process at large scale. There are many variables regarding any indium recovery process, including verified levels of indium recovery expected from a specific volume of LCDs, number of purification process steps (and hence cost) required when different metals or impurities are present in the liquid crystal panels, processing time required to leach different thicknesses of indium layers, and optimised pre-process treatment options to increase indium

Table 3.11. Summary of potential techniques for indium recovery from other industry and from existing indium recovery techniques used by LCD manufacturers

Technique	Cost	Complexity	Environmental impact	Time	Commercial viability
Solvent extraction	High	High	High	Medium	Established
Mechanical treatment	Medium	Low	Medium	Low	Established
Ion exchange	High	High	High	Medium	Established
Acid treatment	Medium	Medium	High	Low	Established
Electrochemistry	High	Medium	High	Low	Established
Lift-off method	Low	Low	Low	Low	Novel
Subcritical water	Low	Medium	Low	Low	Novel
Biometallurgy	Low	Medium	Low	High	Novel
Mechanochemical treatment	High	Medium	High	Low	Novel
Cementation	Medium	Medium	Medium	Low	Novel

yields. These variables affect the market uptake of indium recovery solutions at scale and pose a risk from a process investment perspective. It was believed that a quantification of the variability range would assist the optimisation of existing and/or novel indium recovery processes.

4 Industry Perspective of Indium Recovery

4.1 Objectives

The objectives were to gather information on the perspective of key players from industry regarding indium recovery, including recyclers, manufacturers and industrial associations. This involved the generation of a survey for industry to investigate the key parameters that would influence the uptake of an indium recovery technology.

4.2 Survey Methodology

A survey of nine key questions was developed to investigate and capture the knowledge and opinions of individual companies and organisations regarding the indium recovery from LCDs. A list of relevant organisations was created and the researchers undertook the surveys by telephone either by:

(a) warm lead: where a member of the research group had a contact person in a relevant company and used an email introduction to set up the call and undertake the survey;

(b) cold call: where no contact person was available in the relevant company and the researcher contacted the main reception and asked to be directed to the relevant department or personnel who would have the relevant expertise to undertake the survey.

A script was developed for the researchers to follow, including an introduction to the research project, the university and funding body of the study, and an explanation of the purpose of the survey. The results of the survey were offered for sharing with the survey participants with a confirmation that all participating organisations were treated anonymously and participants only referenced according to their position in the value chain, i.e. electronics manufacturer, recycler, organisation association. It was also confirmed that the survey results would be incorporated into a public report. At the end of the survey, the researchers asked if the organisation would like a follow-up regarding the report and, if so, an email address was sought. Finally, the participant was asked if they could recommend any other organisations or companies that would be relevant or interested in providing input on the topic.

4.3 Survey Results

The questions and the survey results are presented in the box below from participating organisations from Ireland, the USA, France and Denmark.

1. With regards to your organisation, do you see a benefit or a need for the recovery of indium from waste LCD displays?

Manufacturer: Both benefit and need; benefit to reduce supply risk, mitigate environmental impact and close the supply loop of indium.

Recycler: Yes, there is a benefit as this is used a lot.

Manufacturer: Personally, so little in displays it’s not necessary/not expensive for organisation.

Association: Yes, benefit, as material is very important for the future/will the producers be willing to buy the recycled product?

Association:

- EU looking at and pushing REE (rare earth elements) recovery;
- a lot of interest;
- yes, if able to remove and market for product;

- no, if costly and no market for it;
- look for companies that would purchase the materials.

Association: Yes, market – don't know if these would be.

2. Are you aware of industry or pilot-level indium recovery operations for LCDs in Europe or elsewhere? If yes, where, what organisation and what type of process is used?

Manufacturer: Yes, hydrometal recovery, mainly Asia, can't disclose company.

Recycler: No, not aware of any.

Manufacturer: No.

- Some studies in JRC/not aware of any industry work in the area.

Association: Some projects

- Suppliers working on extraction of REE from products.
- First problem is to increase speed of recycling process then look at metals recovered and which metal would be suitable.

Association: French government helps a lot in R&D in this area.

- Paris, France.
- Terenova, France.

Association: No, not aware of any.

3. If an indium recovery technology from waste LCDs was available, would it affect, influence or potentially adjust your organisation's business model or value chain? Please elaborate your response.

Manufacturer: It depends on the supply chain; change of product:

- type of materials used;
- quantity of material.

Recycler: Yes, we would like to build a factory in our country for this specific purpose.

Manufacturer: Cost-driven decision for this company:

- more expensive/influence in that respect;
- if more expensive, then business model would be affected.

Association:

- potential, yes;
- depends on cost;
- no law, if convenient, would be an influencing factor.

Association:

- no change really;
- cost-driven decision;
- internal or external recovery would depend on costing.

Association: Don't think it would; recycled externally.

4. Regarding an indium recovery technology, what do you believe would be the key influencing parameters that would affect the uptake of indium recovery in Europe from your organisation's point of view? Notes: for example, indium price, cost of technology, cost of operation, purity of indium recovered, etc.

Manufacturer: Not cost; if prices increase would push company.

- purity of indium and environmental impact of process;
- would like a holistic approach to recycling, not just partial recycling.

Recycler:

- cost of technology;
- purity of indium recovered (they are getting 99.99% purity from indium tin oxide).

Manufacturer: In my opinion, if it meant an increase in the cost of recycling, it would deter.

- costs better used elsewhere in the company.

Association:

- cost of recovery;
- legislation driven;
- effect on end price of goods.

Association: Must be profit-generating for company otherwise not of interest.

Association: Don't know what would influence it.

5. In your opinion, do you believe that governmental legislation to encourage the indium recovery from waste LCDs would be beneficial?

Manufacturer: No, push more from the private industry.

Recycler:

- yes, losing critical material when not recycling;
- need government to push recovery.

Manufacturer:

- force it to happen;
- justification for regulation?
- companies may design it out of the display.

Association: They should but won't in the short term.

Association:

- all governments should drive REE recovery;
- all recyclers should do better;
- look at Umicore (http://www.umicore.com).

Association: Externally recycled in organisation, yes would be benefit.

6. In terms of implementing an indium recovery process, what do you believe would be the main driving force for such implementation?

Manufacturer: Needs to be a benefit or need for it.

Recycler: Cost.

Manufacturer:

- costing;
- ease of recycling;
- speed of recycling;
- large quantity recovery;
- Is there a market?

Association:

- cost;
- labour;
- logistics;
- all factors affect price at end;
- no market, as companies have own suppliers; if producers would be willing to buy recycled indium, then this would potentially drive that.

Association: Cost reduction would be the main one.

Association: Cost of recovery of indium.

7. Do you believe a scaled-up indium recovery operation could be viable? If so, what do you consider to be the key influencing factors?

Manufacturer: Based on technology now – no. In future – yes, if technology introduced. Everything now is lab-based.

Recycler: Yes, good technology and speed would be the key influencing parameters.

Manufacturer: Not viable, too little in product.

Association: Don't know how much it would cost; must be large-scale production to be viable at all, as purchasers are large companies.

Association: No, don't think so.

Association: No, would not be viable.

8. In your opinion, what are the main technical and/or scale-up challenges for the recovery of indium?

Manufacturer:

- difficult to separate tin and indium;
- lose tin when indium is recovered;
- such a small amount of product;
- market not ready for indium recovery.

Recycler: Purity of indium.

Manufacturer: Too little indium in product/technology constraints to recover indium.

Association: Don’t know.

Association: Volume needed – large to make it competitive for a company.

Association: Don’t know.

9. In your opinion, do you believe there is potentially a short, medium or long-term requirement for indium usage in the ICT sector in the future? Note: short = 1–3 years; medium = 3–5 years; long = 5 + years. Please explain your answer.

Manufacturer:

- long-term use;
- using less but no full substitute.

Recycler: Yes

- used in LCD and LED;
- will be used in other products in future.

Manufacturer: Don’t know.

Association:

- yes, we use a lot of indium today;
- compare price of recycled indium to indium on market;
- depend on whether or not the producer is willing to buy the recycled product.

Association: Don’t know.

Association: Don’t know.

4.4 Discussion and Conclusion

A review of the survey results highlighted the following trends in industrial opinion among the participants:

- All organisations agreed that there was a benefit and/or need for indium recovery.
- Participant organisations reported knowledge of industrial or pilot indium recovery operations in Asia and France.
- All organisations believed that indium recovery operations would have an impact on their business; however, whether this would be a positive or negative impact would be determined

by the cost and benefits of the particular indium recovery methodology being used.

- The key parameters that would influence indium recovery were predominantly cost of recovery and purity of indium obtained as well as having an adequate value chain where the manufacturer would be willing to purchase the recycled indium.
- Regarding whether or not there was a requirement for legislation on indium recovery, most participants (apart from the manufacturers) believed that legislation could be a positive and necessary driver for indium recovery.
- When asked about current industrial drivers for indium recovery, all participant organisations indicated that cost of recovery was key and that the price of the indium from the recovery process must compare well with the price of indium from traditional sources on the market. Other driving forces were ease of recycling and the quantity of indium that the process could produce.
- In general, the impression of the viability of commercial indium recovery was pessimistic, with the majority of concerns focused on the risk associated with the low amount of indium in LCDs and being able to recover enough indium for use as a suitable supply chain for a manufacturer. However, the recyclers' opinions were more optimistic and showed an interest in the indium recovery set-up.
- Regarding the upscaling challenges for indium recovery, from a technical perspective, feedback focused on the purity of indium and the low levels of indium in LCDs.
- Interestingly enough, when asked about the future requirement for indium in products, most participant organisations generally agreed that there is a future requirement, as indium is used in a large number of products now and in the foreseeable future.

It is clear that the development of any indium recovery process should take into account these considerations and that the four key elements outlined in Figure 4.1 will be central to the viability of indium recovery. All considerations pivot around the ability of organisations to sell the recovered indium back into the ICT market, resulting in the manufacturer and their supply chain playing a key role in the overall viability. This industrial perspective is rarely cited among the proposed considerations for indium recovery.

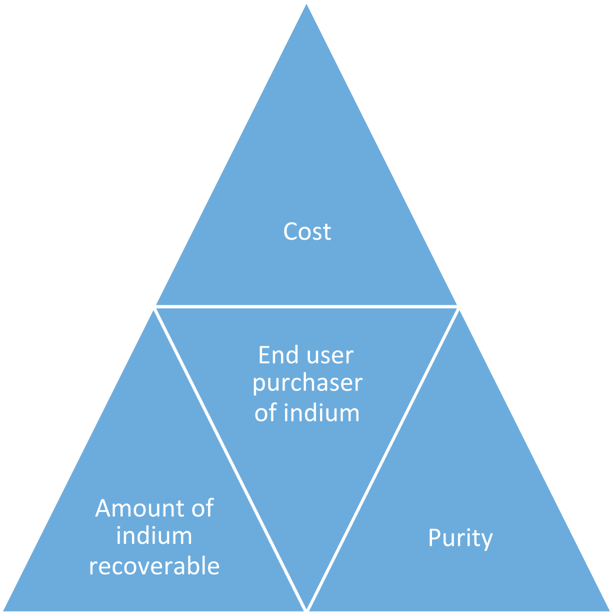


Figure 4.1. Factors to be considered in the development of an indium recovery process.

5 Policy Perspective of LCD Recycling

5.1 Objectives

The objectives were to review the WEEELABEX (WEEE label of excellence) standards with respect to LCD recycling in Ireland, assess the compliance requirements via primary research and gauge Ireland's level of compliance with these standards.

5.2 Overview of the WEEE Directive

In order to reduce the potential impact on the environment when electrical products reach their end of life (EoL), the EU has introduced a number of directives, the most significant being the WEEE Directive, the main objectives of which are:

- reducing the waste arising from EoL electrical and electronic equipment;
- improving and maximising recycling, reuse and other forms of recovery of waste from EoL electrical and electronic equipment;
- minimising the impact on the environment from their treatment and disposal.

Directive 2012/19/EU of the European Parliament and of the Council of 4 July 2012 on waste electrical and electronic equipment (WEEE) (recast) is the latest adopted version of the Directive (WEEE Directive, 2013). Annex VII of this Directive specifies the "selective treatment for materials and components of waste electrical and electronic equipment". Within this section, the specific clauses that deal with LCDs and their components are as follows:

4. As a minimum the following substances, mixtures and components have to be removed from any separately collected WEEE:

- polychlorinated biphenyls (PCB) containing capacitors in accordance with Council Directive 96/59/EC of 16 September 1996 on the disposal of polychlorinated biphenyls and polychlorinated terphenyls (PCB/PCT) (1),
- mercury containing components, such as switches or backlighting lamps,
- printed circuit boards of mobile phones generally, and of other devices if the surface of the printed circuit board is greater than 10 square centimetres,
- chlorofluorocarbons (CFC), hydrochlorofluorocarbons (HCFC) or hydrofluorocarbons (HFC), hydrocarbons (HC),
- liquid crystal displays (together with their casing where appropriate) of a surface greater than 100 square centimetres and all those back-lighted with gas discharge lamps,
- external electric cables.

It specifies that these substances, mixtures and components shall be disposed of or recovered in compliance with Waste Framework Directive 2008/98/EC.

In addition to the WEEE Directive, focus is now drawn to the Europe 2020 policy. One of Europe 2020's flagship initiatives is "Resource efficiency", which promotes extraction and recycling with the aim of using waste as a resource. It recognises the strategic importance of avoiding risks to supplies of resources, such as rare earth elements, which are contained within LCDs. Rare earth materials that are contained within the liquid crystal panel have been classified as "critical raw materials" (EC, 2010).

In addition to the above, Pan-European organisations such as the WEEE Forum are implementing recycling standards (WEEELABEX) to level the playing field for recycling across the different EU countries. The purpose of this is to address the variation that can exist when different countries transpose EU legislation into their individual national laws. The WEEELABEX has laid down a set of technical requirements with respect to WEEE recycling called the "WEEELABEX standards" or normative documents, which will be explored in the next section.

5.3 Overview of the WEEELABEX Standards

5.3.1 Introduction to the WEEELABEX standards

Prior to 2008, the environmental policy relating to WEEE was largely governed by the individual Member States of the EU. Austria, Belgium, the Netherlands, Norway, Sweden and Switzerland were among the first countries to develop producer responsibility legislation in an effort to address the growing mountain of WEEE. It was quickly recognised that there was a real and urgent need for harmonisation of the approach to WEEE in Europe. The WEEE Forum was set up in the early 2000s to address this issue and it now operates in 23 European countries including Ireland and the UK. The WEEE Forum describes itself as a non-profit association that, as of 2013, represents 39 WEEE compliance schemes and is the largest of its kind in the world (2014). Such WEEE compliance schemes are typically non-profit organisations that take care of the collection, treatment and recycling of WEEE on behalf of its producer members. The objectives of the forum are to encourage and promote co-operation between WEEE compliance schemes and to develop a defined set of standards to follow. A stated goal of the forum is to act as a centre of competence that allows member organisations to make constructive contributions to the general debate on electrical and electronic waste policy matters. The association aims to assist its members in the development of their activities in a sustainable manner within the existing regulatory and legislative framework (www.weee-forum.org).

In 2007, the WEEE Forum made the suggestion to harmonise contractual requirements for all types of WEEE. A project plan was developed and submitted to the European Commission under the LIFE programme, an EU financing instrument that promotes, among other things, environmental governance. In August 2008, the European Community awarded funding of €1,064,600 for the setting up of a project by the WEEE Forum. This multi-annual project was titled WEEELABEX from a portmanteau of WEEE Label of Excellence.

The WEEELABEX project was a multi-stakeholder project and was run by the WEEE Forum in co-operation with stakeholders from the producers' community and WEEE processing industry. The objectives of the project were twofold. One objective was to design a set of standards with respect to the collection, sorting, storage, transport, preparation for re-use, treatment, processing and disposal of all kinds of WEEE. The WEEELABEX standards package structurally consists of three documents: the first aimed at operators performing collections of WEEE; the second aimed at logistics operators; and the third aimed at treatment operators. These three documents are freely available on the WEEELABEX website. The other key objective was to put in place a process of monitoring companies through audits conducted by auditors trained by its office. Successful audits result in operators and processes being listed on a publicly accessible WEEELABEX website. This procedure, whereby processes are audited and listed, is referred to as Conformity Verification (CV).

The WEEELABEX standards aim to provide a coherent and comprehensive set of technical requirements with respect to WEEE operations. The WEEE Forum states that the WEEELABEX standards are currently the most comprehensive and coherent set of requirements for all operations that deal with the collection and disposal of WEEE (www.weee-forum.org).

Structure of the WEEELABEX organisation

Following the introduction of the WEEELABEX standards, various WEEE compliance schemes joined together in 2011 to form the WEEELABEX organisation with a mandate to implement the standards across Europe. The WEEELABEX organisation is a stand-alone legal entity, headquartered in Prague, that manages the training of auditors, monitors auditing activities and approves WEEELABEX operators and WEEELABEX auditors. Through its activities, the WEEELABEX organisation actively promotes the WEEELABEX standards to operators and Member States. The structure of the WEEELABEX organisation consists of three constituent bodies (see Figure 5.1):

- WEEELABEX General Assembly – composed of all members of the WEEELABEX systems;
- WEEELABEX Governing Council – the executive body with the authority to enforce orders and to ensure that they are carried out as intended;
- WEEELABEX Office – its functions include secretariat and notary.

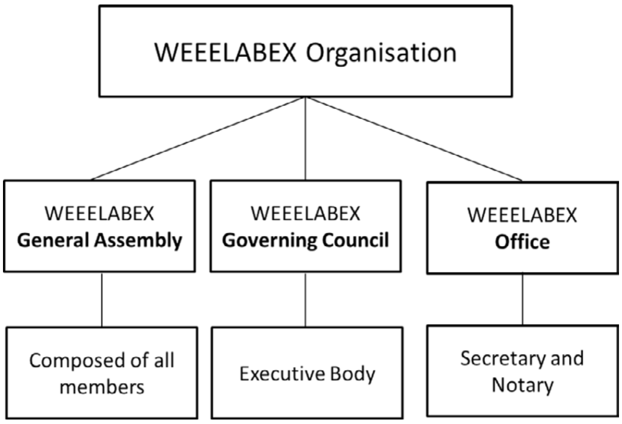


Figure 5.1. The structure of the WEEELABEX organisation.

The WEEELABEX organisation applies a dual approach to implementing the WEEE Directive with an environmental aspect and an efficiency aspect. The environmental part seeks to improve the overall WEEE chain through depollution, whereas the efficiency aspect seeks to create a level playing field for all WEEE operators by imposing a harmonised set of rules, requirements, roadmaps, reports and auditing. The WEEELABEX organisation is open to any compliance scheme or WEEE operator. Schemes that are members of the organisation and all current members are listed on the WEEELABEX website.

Operation of the WEEELABEX standards

The WEEELABEX standards provide a means for assessing those involved in the processing of WEEE within the EU such as manufacturers, retailers, recyclers, local authorities and various compliance schemes. WEEELABEX audits are performed against several treatment process types enabling treatment operators to become approved for one or more process streams depending on the type of treatment activity that they perform, see Table 5.1.

Table 5.1. The treatment types and associated process streams (www.WEEELABEX.org)

Treatment process	Process stream
Type 1	Manual dismantling, including all or some depollution.
Type 2	Mechanical treatment (pre-treatment and intermediate treatment), including some or all depollution (where indicated).
Type 3	Advanced mechanical treatment, including some or all depollution (where indicated).
Type 4	End-processing (pure fractions), or incineration/energy from waste facilities.

WEEELABEX audits can also be performed for individual WEEE categories and this enables treatment operators to become approved for one or more waste streams depending on the type of treatment activity that they perform. These waste streams are as follows:

- large appliances (WEEE Categories 1 and 10, excluding temperature exchange equipment);
- mixed appliances (WEEE Categories 2, 3, 4, 5, 6, 7 and 9, excluding display units that contain cathode ray tubes or cold cathode fluorescent lamps);
- temperature exchange equipment (WEEE Category 1 – fridges, freezers, air-conditioning units, heat exchange tumble dryers, etc.);
- display units cathode ray tubes (CRT) (WEEE Categories 3 and 4 – televisions and monitors containing CRTs);
- display units FPD (WEEE Categories 3 and 4 – televisions and monitors containing cold cathode fluorescent lamps);
- gas discharge lamps (WEEE Category 5).

5.3.2 *The current status of the WEEELABEX standards*

The original WEEE Directive was published in 2003 and the updated Directive was published in 2012. The main changes introduced in the updated Directive are:

- to broaden the range of electronic and electrical equipment covered by the Directive;
- to put greater emphasis on the re-use of WEEE products;
- an effort to limit the red tape and cost burdens for businesses;
- to fight the illegal export of WEEE more effectively;
- increased recovery targets for Member States;
- a like-for-like exchange requirement for small WEEE products at retail level.

The WEEELABEX standards focused on being aligned with other standards operating in the EU. In August 2009 the WEEELABEX organisation signed a contract of co-operation with the European Committee for Electrotechnical Standardization (CENELEC), one of the three official EU standards bodies. The development of the WEEELABEX standards into formal EN standards is planned, which will confer a clear set of rules for all operators on the market to comply with the WEEE Directive (WEEELABEX).[1] An update regarding the status of these standards was undertaken by primary researchers involving surveys, which are detailed in next section.

In 2013, the Green Electronics Council (EPEAT), the American global rating system for green electronics, announced that the WEEELABEX standards had satisfied all of its environmental requirements (WEEE Forum, 2014). So far, only two other recycling standards have met the EPEAT requirements for responsible recycling of electronics in Europe and both of these are in fact US standards: the R2:2013 standards and the e-Stewards standards for Responsible Recycling and Reuse of Electronic Equipment. It is expected that this will place the WEEELABEX standards at a distinct advantage to other standards, particularly in relation to attracting transatlantic business.

5.3.3 The WEEELABEX standards in Ireland

The WEEELABEX standards are intended to apply to all types and sizes of organisations involved in WEEE operations from collection to disposal. Producers, in conjunction with a given compliance scheme, are charged with implementing the WEEELABEX standards. The WEEELABEX standards deal with all 10 of the WEEE categories listed in Table 5.2.

Table 5.2. WEEE category listings (from WEEE Ireland, 2014)

Large Household Appliances
Small Household Appliances
IT and Telecommunications Equipment
Consumer Equipment
Lighting Equipment (except household luminaries)
Electrical and Electronic
Toys, Leisure and Sports Equipment
Medical Devices (except implants and infected products)
Monitoring and Control Instruments
Automatic Dispensers

The WEEE categories that encompass FPD technology are category 3.0 (IT and Telecommunications Equipment) and category 4.0 (Consumer Equipment). Regarding Irish compliance, three Irish operators are approved by WEEELABEX, see Table 5.3.

WEEELABEX impact within Ireland

When the WEEE Directive was implemented in Ireland in 2005, it was initially met with a high level of compliance with relatively minor levels of non-conformance. In later years, however, WEEE Ireland reported a large increase in the problem of the scavenging and harvesting of large WEEE items. This practice later progressed to the targeting of smaller household appliances. WEEE Ireland observed that the introduction of the WEEELABEX standards in Ireland (in April 2011) had assisted in alleviating scavenging and harvesting. They stated that this improvement is mainly as a result of the increased organisation and targeted approach to WEEE recycling promoted by the WEEELABEX standards. The WEEELABEX organisation has been involved in training Irish auditors to approve and monitor all indigenous WEEE treatment processes. Introduction of the standards has also seen increased participation of scrap metal merchants who are encouraged to report any attempt at illegal dumping of WEEE.

LCD technology is rapidly evolving and responding to this is a problem for all producers and recyclers of WEEE products including those based in Ireland. As the frequency with which consumers update their LCD-containing equipment increases, problems have begun to arise with the safe storage of this particular type of WEEE, according to WEEE Ireland. They report that the cost of securing these sites currently falls on the individual recyclers and this problem can inevitably add to the overall cost of recycling, which ultimately renders proper LCD recycling not as cost effective (www.weeeireland.ie). It has been suggested that government subsidisation of WEEE operators through grants could help to offset this cost. Alternatively, more rapid and efficient recycling technologies for the cost-effective depollution of WEEE such as LCDs would be particularly welcome. Greater research at a national level into the depollution of LCDs would ensure Ireland's position at the forefront of environmental

---

[1] http://www.weeelabex.org/

Table 5.3. Irish operators approved by WEEELABEX

Operator	Treatment process streams audited	Commissioned by	Lead auditor	City	Type of operator
Irish lamp recycling Co. Ltd	Lamps	Treatment operator	Julie-Ann Adams	Athy	1
KMK Metals Recycling Ltd	SHA, CRT	Treatment operator	Luca Campadello	Tullamore	2
The Recycling Village Ltd	FPD, CRT	ERP	Angel Valor	Duleek	2

CRT, cathode ray tube; ERP, European Recycling Platform; SHA, small household appliances.

innovation and help to contribute the scientific knowledge that can ultimately be used to meet robust environmental standards such as the WEEELABEX.

5.3.4 The relevance of the WEEELABEX standards to LCD recycling

The WEEELABEX standards have developed specific requirements for the treatment of FPDs. This normative document defines additional requirements to those defined in the WEEELABEX general requirements for collection, logistics and treatment of WEEE and addresses the specific requirements for treatment of FPDs as part of the WEEE stream, including all components, fractions thereof and associated powders, liquids and gases.

The WEEELABEX standards outline the administration and technical requirements for all stages of the recycling of LCDs and govern their preparation for treatment, the general depollution of hazardous components (mercury, ITO and fluorescent coatings) and the various monitoring processes. The requirements are outlined in the following sections:

Administrative and organisational requirements

Technical and infrastructure conditions

- Collection, handling and transport of flat panel displays shall be performed in such a way that the integrity of the displays is not affected. Crushing or compacting is not permitted prior to treatment.
- Flat panel displays and their components shall be stored under weatherproof covering.

Note 1: Components of flat panel displays may include: backlight lamps, fluorescent coatings and/or lithium containing batteries.

Note 2: Examples of weatherproof covering include: roof, closed containers.

Transport

- Containers shall be loaded carefully. When stacking one container on top of another, precautions shall be in place to prevent breakage of the flat panel displays in the container underneath.
- During transport of flat panel displays, weatherproof covering shall be ensured.

Information materials

- Employee training materials and information shall be available at the workplace and easily accessible to workers at all times. Materials and information shall document specific risks inherent to flat panel displays.

Note: Potential risks include physical injury, exposure to mercury, lead and/or indium tin oxide, and inhalation of dust and/or fluorescent coatings.

Technical requirements

Preparation for treatment

- If treatment technology requires separation of different types of flat panel display, employees shall be qualified and trained in the appropriate methods to carry out the sorting tasks.

General depollution

- Substances and components contained in flat panel displays shall be removed in accordance with Annex A and B (depollution guidelines and monitoring) of this normative document.
- The treatment of flat panel displays shall consider the different types of displays, the fractions and components thereof, and the specific requirements for:
  - mercury,
  - fluorescent coatings, and

  - indium tin oxide.
- Mechanical treatment of flat panel displays shall be carried out in a dedicated treatment facility, which can document that no contamination occurs with other treated streams.

Mercury

- For treatment of flat panel displays with CCFL backlights, evidence shall be provided showing that at least a given percentage in mass of mercury from backlight lamps is removed from the input content of the non-treated appliances.
- This can also be demonstrated by verifying that less than a given amount (in mg/m^3 or mg/kg) of mercury is present in the fractions that are intended to be recycled and that the fractions where the mercury is concentrated are directed to appropriate disposal.
- Broken CCFL backlights from manual dismantling shall be stored and transported in closed containers, in order to avoid mercury emissions. Such containers shall be stored in places that are not exposed to heat.
- All CCFL backlights from manual dismantling, whether broken or not broken, shall be treated in special treatment plants for lamps or sent for appropriate disposal in accordance with national legislation.

Indium tin oxide (ITO)

- When liquid crystal display panels or their fractions are sent to treatment facilities that intend to concentrate ITO, recovery and disposal, operations shall be subject to the requirements of the WEEELABEX normative document on Treatment – Part I (General Requirements) (WEEELABEX Standards).

Fluorescent coatings

- Flat panel displays and fractions shall be sent to treatment facilities that guarantee recovery or disposal of the fluorescent coatings and glass in accordance with Clause 5.8.2 of WEEELABEX normative document on Treatment – Part I (General Requirements).
- Fluorescent coatings and fractions containing fluorescent coatings shall be disposed of in landfill, or treated by suitable thermal processes, designed and approved for hazardous substances.

Monitoring

- The operator shall develop protocols and procedures appropriate to his technology, to demonstrate the fulfilment of the pollutants' removal target, as stipulated in Clause 5.2.
- The processing of flat panel displays shall be carried out in a controlled atmosphere. Suitable ventilation equipment and filters shall ensure that occupational exposure limits (OEL) and air emission limit values on heavy metals and dust can be complied with at all times. Accumulation of heavy metals in dust shall also be measured periodically.
- The airborne mercury content of workplaces where processing of LCD with CCFL backlights takes place and of storage areas shall be monitored, following the periodicity and protocols described in the respective European legislation on Health and Safety. Occupational health monitoring of concerned employees shall include measurement of the intake of mercury by employees through analysis of mercury concentration in blood or urine.

The standards are yet to specify the depollution limits for the specific materials and substances listed above. It is expected that the working group of the WEEELABEX will finalise these requirements in 2016 and the standards will be published by CENELEC.

The information presented herein was sourced from relevant industry organisations' websites and primary research that involved surveying the key organisations involved in the WEEELABEX. The survey results are included in Appendix 1.

5.4 Findings and Conclusions

From a review of the standards and the survey results, the key findings regarding LCD recycling are summarised below:

- Transfer of the WEEELABEX guidelines into a CENELEC standard will aid the uptake of these guidelines, which will promote resource-efficient and environmentally sound recycling of LCDs.

- The WEEELABEX working group are currently determining the limits regarding mercury fractions from LCDs post recycling.
- The surveys reveal that FPDs are still only on the rise from low levels at recycling facilities across the EU.
- Any market conditions that help to generate a revenue regarding LCD recycling are key and need to be reinforced with appropriate legislation and standards.
- Favourable market conditions include efficient and fast recycling processes, recovery of additional metals with marketable value.
- It is also evident from the surveys that any player in the market cannot operate in isolation and decisions regarding recycling methodologies or metal recovery processes must involve the upstream and downstream players to be successful, including compliance schemes and operators who supply the separated fractions to the end user purchaser.
- Regarding the recovery of ITO, it was generally reported to be dependent on market needs and the standards would support efficient and environmental recovery technologies where applicable. However, at present this is not a mandatory requirement regarding the recycling of LCDs.

6 Conclusions and Recommendations

The objectives of the research were to investigate and explore the potential for indium recovery from LCDs and our recommendations are listed together with the specific objectives below:

To investigate the presence and amount of indium contained in the liquid crystal panels of LCDs

- The findings from the 10 samples tested revealed that indium content varied significantly from 38 to 292 mg indium per kg glass panel where the LCD is made up of two of these panels.
- Generally, it was found that the glass front substrate contains higher concentrations of indium than the glass back substrate, correlating with a uniform electrode layer and an intermittent electrode layer, respectively.
- Optical analysis revealed different surface structures of the electrode coatings where transparent electrodes exhibited either a rectangular cell or V-cell structure. It was found that rectangular cell structures generally contained higher indium concentrations and were the more common structure of the samples examined.
- Other elements were regularly present including aluminium, silicon, molybdenum and titanium.
- Recommendations:
  - Understanding the trend of variation of the LCDs and the indium content allows the recycler to make informed decisions regarding a metal recovery process.
  - Larger sample testing of LCD panels to create a database for indium level and internal structure on a large scale would assist recyclers and smelters regarding parameter decisions for a hydrometallurgical process.
  - The potential to correlate the cell structure (using simple optical microscope or X-ray) with the indium level could be a fast and cheap process-control method to separate out panels with high indium concentration.

To review potential methodologies and best available techniques for the recovery of indium from LCDs

- While some new plants have been reported to be undertaking indium recovery in France, Spain and Asia, information regarding processing is not available.
- Generally, hydrometallurgy is the well-known and traditional route to recover metal with the predominant techniques for indium recovery focused on solvent extraction and ion exchange. These technologies are usually operated at scale but require optimisation regarding the recovery of indium from LCDs to deal with low indium concentration, presence of other metals and best pre-processing steps.
- Novel techniques have also been reported, such as the lift-off method, subcritical water process, biotreaments and mechanochemical treatments. However, these are mainly at the laboratory scale testing phase and are not reported as being deployed commercially.
- The review has highlighted both the interest in indium from a research and industrial perspective but also that finding an economical process to recover indium at scale is still required. The influence of the business case on the technology is a significant facet of rolling out a large scale recovery plant and this aspect requires future development.
- Recommendations:
  - Whether using traditional hydrometallurgy or novel techniques, the adaptability of the process to account for variations in indium concentration and the presence of other metals is key.
  - Further research regarding the specific effects of input indium concentration thresholds, the presence of other metals, and indium thickness on each of the hydrometallurgical steps for recovery would aid the development

  of an optimised process. The tailoring of a pre-process preparation step would also be central to the optimisation of the indium recovery where options could include the separation of front and back panels, an optical check for structure/high indium concentrations, and leaching performed with panel intact or pre-shredding of panel, while recovery options would include tailoring parameters such as time, temperature, number of metal recovery runs or purification steps required.
  - Comparison with control samples of indium supplied traditionally from the market regarding purity and characterisation.
  - Justification of the business case regarding indium recovery from a plant operation costs perspective and potential revenue generated from such activities.

To review the requirement for indium recovery from an industrial perspective

- All organisations believed that indium recovery operations would have an impact on their business; however, whether this was a positive or negative impact would be determined by the cost and benefits of the particular indium recovery methodology being used.
- The key parameters that would influence indium recovery were predominantly cost of recovery and purity of indium obtained as well as having an adequate value chain where the manufacturer would be willing to purchase the recycled indium.
- When asked about current industrial drivers for indium recovery, all participant organisations indicated that cost of recovery was key and that the price of the indium from the recovery process must compare well with the price of indium from traditional sources on the market. Other driving forces were ease of recycling and the quantity of indium that the process could produce.
- Regarding the upscaling challenges for indium recovery, from a technical perspective feedback focused on the purity of indium and the low levels of indium in the LCDs.
- When asked about the future requirement for indium in products, most participant organisations generally agreed that there is a future requirement, as indium is used in a large number of products now and in the foreseeable future.
- Recommendations:
  - Any process development research for indium recovery should focus on these key parameters: technical aspects (purity and low concentration indium recovery) and commercial aspects (cost of recovery and acceptance of recovered indium as a product for the end user, i.e. the manufacturer).
  - Therefore, regarding the technical aspects, processes should be designed utilising a knowledge bank to take into account the presence of other elements and indium concentration expected per batch as well as using pre-processing steps to ensure a minimum indium concentration per batch of panels to be processed.
  - Regarding the commercial aspects, control samples to compare and contrast recovered indium with traditionally sourced indium is important as well as engaging the end purchaser regarding quantities of indium required, characterisations of the product and any other factors that they would consider critical when potentially using recovered indium as a supply.

To review the requirement for LCD recycling from a policy perspective

- WEEELABEX is an important industry standard and is developing threshold values for mercury post-recycling for FPDs.
- The standard requires that consideration should be given to the ITO fraction of the liquid crystal panel; however, this does not require mandatory recovery.
- In general, it is believed that ITO will be driven by economic factors and market conditions.
- Recommendations:
  - As the WEEELABEX standard is in the process of being developed into the CENELEC standard with an expected completion in 2016, monitoring and inputting to the developments where opportunity arises via the compliance scheme is important.
  - Regarding both LCD recycling and indium recovery, it is clear that engagement of multiple stakeholders along the value chain

is required and not just the recycler in order to achieve change or a step forward in terms of processing capability. The drivers for implementation of technologies usually lie with the end user, in this case being a complex chain from recyclers and smelters to manufacturers (and potential metal traders in between). The recommendation regarding policy is the continued engagement of Irish industry and stakeholders with European policy development as well as increasing engagement with connections and networking opportunities such as the European Innovation Partners (WEEE 2020)[2] and Raw Materials Knowledge and Innovation Community (KIC),[3] all of which incorporate a focus on electronic recycling.

---

[2] https://ec.europa.eu/growth/tools-databases/eip-raw-materials/en/content/weee-2020-raw-material-partnership-%E2%80%93-delivering-advancements-across-weee-value-chain-improve

[3] www.EITRawMaterials.eu

References

Alguacil, F.J., 1999. Solvent extraction of indium III by LIX 973N. *Hydrometallurgy* 51: 91–102.

Beau, R., 1965. Process for the Manufacture of Indium of High Purity. US3180812.

Brierley, J.A., 2008. A perspective on developments in biohydrometallurgy. *Hydrometallurgy* 94: 2–7.

Choi, D., Kim, Y.S. and Son, Y., 2014. Recovery of indium (ITO) and glass plate from discarded TFT-LCD panels using an electrochemical method and acid treatment. *RSC Advances* 4: 50975–50980.

Dodbiba, G., Nagai, H., Wang, L.P., Okaya, K. and Fujita, T., 2012. Leaching of indium from obsolete liquid crystal displays: comparing grinding with electrical disintegration in context of LCA. *Waste Management* 32: 1937–1944.

EC (European Commission), 2008. Review of Directive 2002/96/EC of the European Parliament and of the Council of 27 January 2003 on waste electrical and electronic equipment (WEEE) “Final Report”, United Nations University, Germany. Available online: ec.europa.eu/environment/waste/pdf/weee_review.pdf (accessed 11 May 2015).

EC (European Commission), 2010. Report of the Ad-hoc Working Group on defining critical raw materials “Critical raw materials for the EU”. Brussels.

Ferron, C.G., 2014. Treatment of Indium Gallium Alloys and Recovery of Indium and Gallium. Neo Material Technologies Inc, US8834818 B2.

George, M.W., 2003. Indium. In *Minerals Yearbook – 2003*. US Geological Survey.

Hasegawa, H., Rahman, I.M., Egawa, Y., Sawai, H., Begum, Z.A., Maki, T. and Mizutani, S., 2013. Chelant-induced reclamation of indium from the spent liquid crystal display panels with the aid of microwave irradiation. *Journal of Hazardous Materials* 254–255: 10–7.

He, Y., Ma, E. and Xu, Z., 2014. Recycling indium from waste liquid crystal display panel by vacuum carbon-reduction. *Journal of Hazardous Materials* 268: 185–190.

Inoue, K. and Alam, S., 2015. Hydrometallurgical recovery of indium from flat-panel displays of spent liquid crystal televisions. *JOM* 67: 400–405.

Jadhav, U. and Hocheng, H., 2015. Enzymatic bioleaching of metals from printed circuit board. *Clean Technologies and Environmental Policy* 17: 947–956.

Kamberović, Z., Korać, M. and Ranitović, M., 2011. Hydrometallurgical process for extraction of metals from electronic waste-part ii: development of the processes for the recovery of copper from Printed Circuit Boards (PCB). *Association of Metallurgical Engineers of Serbia* 17(3): 139–149.

Kang, H.N., Lee, J.-Y. and Kim, J.-Y., 2011 Recovery of indium from etching waste by solvent extraction and electrolytic refining. *Hydrometallurgy* 110: 120–127.

Kato, T., Igarashi, S., Ishiwatari, Y., Furukawa, M. and Yamaguchi, H., 2013. Separation and concentration of indium from a liquid crystal display via homogeneous liquid–liquid extraction. *Hydrometallurgy* 137: 148–155.

Kelly, S.M., 2000. *Flat Panel displays, Advanced Organic Materials*. Royal Society of Chemistry, RSC Materials Monographs, Cambridge.

Kita, N., Nakamura, Y. and Tsukada, A., 2011. Method for Recovering Indium from Indium-Containing Material. Dowa Metals & Mining Co., Ltd, EP1905855 B1.

Konishi, Y., Saito, N. and Higashi, A., 2014. Indium Recovery Method. Osaka Prefecture University Public Corporation, EP2722409 A1.

Lee, C.H., Jeong, M.K., Kilicaslan, M.F., Lee, J.H., Hong, H.S. and Hong, S.J., 2013. Recovery of indium from used LCD panel by a time efficient and environmentally sound method assisted HEBM. *Waste Management* 33: 730–734.

Lee, M., Ahn, J. and Lee, E., 2002. Solvent extraction separation of indium and gallium from sulphate solutions using D2EHPA. *Hydrometallurgy* 63: 269–276.

Lee, U.H., Chang, J.S., Lee, S.K., Hwang, Y.K. and Jeon, D.H., 2014. Method for Recovering Indium from Solution or Mixture which Contains Indium. Korea Research Institute Of Chemical Technology, WO2014084445A1.

Le Quesne, Y. and Fossi, P., 1991. Process for Hydrometallurgical Treatment of an Indium Chloride Solution. Metaleurop Sa, WO1989004877A1.

Li, J., Gao, S., Duan, H. and Liu, L., 2009. Recovery of valuable materials from waste liquid crystal display panel. *Waste Management* 29: 2033–2039.

Liu, X-Z., Zang, S-L., Fang, D-W., Jun, L. and Wang, L-N., 2006. Thermodynamics of solvent extraction of rare and scattered metal-indium with diethylhexylmonothiophosphoric acid. *Chemical Research in Chinese Universities* 22: 111–113.

Miki, T., Sugimoto, T., Sahashi, E., Honma, T. and Maeseto, T., 2011. Method and Apparatus for Recovering Indium from Etching Waste Solution Containing Indium and Ferric Chloride. Sharp Kabushiki Kaisha, Kobelco Eco-Solutions Co., Ltd, US20110132146A1.

Ming, X. and Xin, L., 2014. Waste Liquid Crystal Display Panel Treatment and Resource Recycling Method. Shanghai Jiaotong University, CN102671921B.

Murray, W.S., 1933. Indium Recovery Process. Oneida Community Ltd, US1912590A.

Namias, J., 2013. *The Future of Electronic Waste Recycling in the United States: Obstacles and Domestic Solutions.* Dissertation. Columbia University, New York City, NY.

Nishihama, S., Hirai, T. and Komasawa, I., 1999. Separation and recovery of gallium and indium from simulated zinc refinery residue by liquid-liquid extraction. *Industrial and Engineering Chemistry Research* 38: 1032–1039.

O'Donoghue, L., Moroney, P. and Harrison, W., 2014. Novel LCD separation strategy to allow for maximum rare earth recovery. *Proceedings of the Electronics Goes Green – Care Innovation Conference*, Vienna, Austria, 17–20 November 2014.

Ogi, T., Tamaoki, K., Saitoh, N., Higashi, A. and Konishi, Y., 2012. Recovery of indium from aqueous solutions by the Gram-negative bacterium *Shewanella algae*. *Biochemical Engineering Journal* 63: 129–133.

Paiva, A.P., 2001. Recovery of indium from aqueous solutions by solvent extraction. *Separation Science and Technology* 36: 1395–1419.

Rocchetti, L., Amato, A., Fonti, V., Ubaldini, S., De Michelis, I., Kopacek, B., Vegliò, F. and Beolchini, F., 2015. Cross-current leaching of indium from end-of-life LCD panels. *Waste Management* 42: 180–187.

Rocchetti, L, Amato, A. and Beolchini, F., 2016. Recovery of indium from liquid crystal displays. *Journal of Cleaner Production* 116: 299–305.

Ruan, J., Guo, Y. and Qiao, Q., 2012. Recovery of indium from scrap TFT- LCDs by solvent extraction. *Procedia Environmental Sciences* 16: 545–551.

Ruisheng, W., 2014. Method for Recycling TFT (Thin Film Transistor) Liquid Crystal Display with Liquid Crystal Bubbles. BYD Company Limited, CN102207640B.

Statista, 2015. Worldwide sales of LCD TV by region from 2011 to 2015 (in million units). Available online: http://www.statista.com/statistics/326947/lcd-sales-by-region/ (accessed 22 February 2016).

Strategic Metal Report, 2014. Indium. Available online: http://strategic-metal.typepad.com/strategic-metal-report/indium/ (accessed 27 July 2014).

Terakado, O., Iwaki, D., Murayama, K. and Hirasawa, M., 2011. Indium recovery from indium tin oxide, ITO, thin film deposited on glass plate by chlorination treatment with ammonium chloride. *Materials Transactions* 52: 1655–1660.

Tomii, K. and Tsuchida, H., 1981. Solvent Extraction Recovery Process for Indium. Toho Aen Kabushiki Kaisha US4292284A.

Virolainen, S., Ibana, D. and Paatero, E., 2011. Recovery of indium from indium tin oxide by solvent extraction. *Hydrometallurgy* 107: 56–61.

Wang, X., Lu, X. and Zhang, S., 2013. Study on the waste liquid crystal display treatment: focus on the resource recovery. *Journal of Hazardous Materials* 244–245: 342–347.

WEEE Directive, 2013. Implementation of the waste electrical and electronic equipment (WEEE) (recast) Directive 2012/19/EU, Consultation Document, Department of the Environment, Ireland, October 2013. Available online: www.environ.ie (accessed 13 May 2015).

WEEE Forum, 2014. Equivalent conditions for the treatment of WEEE exported outside the European Union. WEEE Forum. Available online: www.weee-forum.org (accessed 12 May 2015).

WEEE Ireland, 2013. WEEE Annual environmental report 2013. WEEE Register Society Category Listings, The National WEEE Registration Body. Available online: http://www.weeeregister.ie/information (accessed March 2015).

WEEE Ireland, 2014. WEEE annual environmental report 2014. WEEE Register Society Category Listings, The National WEEE Registration Body. Available online: http://www.weeeireland.ie/resources/annual-reports-and-financial-statement-2014/ (accessed 22 February 2016).

WEEELABEX, 2011a. WEEELABEX Collection – Normative document on Collection V9.0. Available online: http://www.weeelabex.org/wp-content/uploads/2015/10/968606_841245d6de816ba82bf6cf82af30ce4b.pdf

WEEELABEX, 2011b. WEEELABEX Logistics – Normative document on Logistics V9.0. Available online: http://www.weeelabex.org/wp-content/uploads/2015/10/968606_83239faa8af3d5ac0f589df6b81ed82c.pdf

WEEELABEX, 2013. WEEELABEX Treatment – Normative document on Treatment V10.0. Available online: http://www.weeelabex.org/wp-content/uploads/2015/10/968606_0dbec6e7617cd83ae8307684f59d4244.pdf

Weiqing, F., 2010. Process for Extracting Indium from Waste Liquid Crystal Display Screen. Suzhou Rui Environmental Technology Group Ltd, CN102002591A.

Weiqing, F., 2011. Process for Treating Waste LCD. Suzhou Rui Environmental Technology Group Ltd, CN102002592A.

Willner, J. and Fornalczyk, A. 2013. Extraction of metals from electronic waste by bacterial leaching. *Environment Protection Engineering* 39: 197–208.

Willner, J., Kadukova, J., Fornalczyk, A. and Saternus, M., 2015. Biohydrometallurgical methods for metals recovery from waste materials. *Metalurgija* 54: 255–258.

Wu, Y-F., Cheng, C-H., Wang, Y-S. and Tsai, T-H., 2014. Recovery of indium from etching waste using a rotating cylinder electrode system. *Fresenius Enviromental Bulletin* 23: 51–56.

Xiaoyong, P., Peng, L., Junxi, Q., Zeping, W. and Weihua, C., 2014. Method for Recycling Indium from Waste Liquid Crystal Displays. Sichuan Changhong Electric Co., Ltd, CN103602815A.

Yang, J., 2012. *Recovery of Indium from End-of-Life Liquid Crystal Displays*. Thesis (unpublished). Chalmers University of Technology, Gothenburg.

Yang, J., Ekberg, C. and Retegan, T., 2014. Optimization of indium recovery and separation from LCD waste by solvent extraction with bis(2-ethylhexyl) phosphate (D2EHPA). *International Journal of Chemical Engineering*: 1–9.

Yoshida, H., Izhar, S., Nishio, E., Utsumi, Y., Kakimori, N. and Asghari Feridoun, S., 2014. Recovery of indium from TFT and CF glasses in LCD panel wastes using sub-critical water. *Solar Energy Materials and Solar Cells* 125: 14–19.

Yoshida, H., Izhar, S., Nishio, E., Utsumi, Y., Kakimori, N. and Asghari Feridoun, S., 2015. Recovery of indium from TFT and CF glasses of LCD wastes using NaOH-enhanced sub-critical water. *Journal of Supercritical Fluids* 104: 40–48.

Zhifeng, L., Kai, Z., Yulin, W., Qiu, C. and Hao, J., 2015. One Kind of Waste Recycling Methods LCD Panel. Hefei University of Technology, CN103191903B.

Abbreviations

AAS	Atomic absorption spectroscopy
B2B	Business-to-business
BAT	Best available technique
CCFL	Cold cathode fluorescent lighting
CENELEC	European Committee for Electrotechnical Standardization
CF	Colour filter
CRT	Cathode ray tube
EDS	Energy dispersive spectroscopy
EPEAT	Green Electronics Council
EoL	End of life
EU	European Union
FIB	Focused ion beam
FPD	Flat panel display
ICP-OAS	Inductively coupled plasma–optical absorption spectroscopy
ICP-OES	Inductively coupled plasma–optical emission spectroscopy
ICT	Information and Communications Technology
ITO	Indium tin oxide
IZO	Indium-doped zinc oxide
LCD	Liquid crystal display
LCD-LED	Liquid crystal display–light-emitting diode
OLED	Organic light-emitting diode
PCB	Polychlorinated biphenyl
REE	Rare earth element
SEM	Scanning electron microscope
SX	Solvent extraction
TFT	Thin-film transistor
WEEE	Waste electronic and electrical equipment
WEEELABEX	WEEE label of excellence
XRD	X-ray diffraction

Appendix 1 WEEELABEX Survey Results

Survey 1: Relevant Association

1. How would you rate the current level of compliance with the WEEELABEX standards across the EU regarding the depollution of LCDs?
    - 57 operators are listed as having the WEEELABEX standards with seven listed for LCDs.
    - There are another 77 operators in the process of being listed.
    - So the rate is high and increasing.

2. Five organisations have conformity verification to process FPD, are a large volume of LCDs being processed by these operators? If so, can you give an estimated volume?
    - This information is confidential. Producers might be of help here. I suggest less than 20% treated currently. Collection rates are also less than 20%.
    - 80% are still in use. 80%–90% of the 20% collected are treated correctly.
    - The only negative is for type 1 operators. For example, scavenging occurs so most of the valuable parts are sent abroad.
    - We should keep the raw material in the EU and keep jobs in the EU. Therefore, this malpractice should be stopped.

3. Regarding the technical standard, Clauses 5.3.1 and 5.3.2 look to specify particular thresholds for mercury – how and when will these threshold values be set? Please elaborate.
    - I will follow up with regarding this question.

4. Clause 5.4 relates to requirements for the ITO contained within LCDs. However, it is not mandatory to recover the ITO, do you foresee further advances in the standard regarding ITO recovery or setting of a threshold recovery target for ITO in the future? If so, please elaborate.
    - The WEEELABEX is the only valid source at the moment. The standard will be adjusted according to the new updated CENELEC standards. The CENELEC is expensive: €40/€60 per standard and there are 30 such standards. The WEEELABEX is free.
    - The WEEELABEX is a competency centre so if stakeholders do something then the WEEELABEX will update accordingly.

5. Are any of the WEEELABEX operators listed above for the FPD currently recovering the ITO? If so, please elaborate.
    - Don't know.

6. Clause 5.5 relates to requirements for the fluorescent coatings contained within LCDs. However, it is not mandatory to recover the fluorescent coating, do you foresee further advances in the standard regarding fluorescent coating recovery or setting of a threshold recovery target in the future? If so, please elaborate.
    - Yes, this is being worked on at present. When these are set will depend on industry. This type of WEEE comes under category 3 and 4. CRT and CCFLs are considered as two separate waste streams because future recovery targets will change. Since 1994 no chlorofluorocarbons (CFCs) have been placed on the market. So the targets are not relevant after 20 years. However, these standards will be valid for countries such as Russia, Africa, etc.

7. Do you foresee any forthcoming changes to the WEEELABEX standards, specifically with respect to LCD depollution?
    - Yes, but don't know what they will be.

8. Can you briefly outline the auditing procedure for an operator and how long it takes to complete an audit?
    - I can't answer this question.

9. How would you rate Ireland's compliance with the WEEELABEX standard for LCD recycling in particular? Can you elaborate?
    - Don't know.

10. Ireland has three WEEELABEX approved operators and two trained auditors: is this correct or up to date?
    - I believe so.

11. What are the future plans for the WEEELABEX standard?
    - They will adjust according to the updated CENELEC standard.

12. WEEELABEX is being registered as a CENELEC standard. When will this process be completed and what will it mean for WEEELABEX?
    - Expect this year. WEEELABEX will align with any changes.

13. What are the main obstacles for WEEE operators that you feel might hinder their full compliance with the WEEELABEX standard, specifically with respect to depollution of LCDs?
    - Cost, efficiencies and illegal exportation are the main obstacles.

14. What would you feel would further promote the recycling of LCDs across the EU?
    - Suitable recovery technology would promote recycling of LCDs.
    - Everything is dependent on market prices.

Survey 2: Relevant Organisation

15. How would you rate the current level of compliance with the WEEELABEX standards across the EU regarding the depollution of LCDs?
    - This is a difficult question to answer but compliance would be close to meeting the WEEE Directive. A rough estimate would be 70–80% compliance.

16. Five organisations have conformity verification to process FPDs. Are a large volume of LCDs being processed by these operators? If so, can you give an estimated volume?
    - I would need to know the amounts to give an answer. This would be considered commercially sensitive information so difficult to obtain. WEEELABEX auditors know this information but it is confidential.

17. Regarding the technical standard, Clauses 5.3.1 and 5.3.2 look to specify particular thresholds for mercury – how and when will these threshold values be set? Please elaborate.
    - The technical standard is complemented by another document, A10 for depollution of monitors.

18. Clause 5.4 relates to requirements for the ITO contained within LCDs. However, it is not mandatory to recover the ITO. Do you foresee further advances in the standard regarding ITO recovery or setting of a threshold recovery target for ITO in the future? If so, please elaborate.
    - Not sure. There will be nothing in the short term in advance of the CENELEC standard.

19. Are any of the WEEELABEX operators listed above for the FPD currently recovering the ITO? If so, please elaborate.
    - Perhaps WIREC (Spain) but the participant was not aware of the process. Indium content is low in FPDs so WEEE operators require large volumes. Operators may be sending ITO to other facilities.

20. Clause 5.5 relates to requirements for the fluorescent coatings contained within LCDs. However, it is not mandatory to recover the fluorescent coating. Do you foresee further advances in the standard regarding fluorescent coating recovery or setting of a threshold recovery target in the future? If so, please elaborate.
    - The CENELEC are working on limits for LCDs so the WEEELABEX will be waiting to update on these.

21. Do you foresee any forthcoming changes to the WEEELABEX standards, specifically with respect to LCD depollution?

    - No, they will be waiting for the CENELEC to be published.

22. Can you briefly outline the auditing procedure for an operator and how long it takes to complete an audit?

    (a) An application is submitted to the WEEELABEX office which is a Declaration of Intent and operators say they are ready and want an audit.

    (b) The WEEELABEX check to see if the operator is within scope.

    (c) Wait for audit fee to be received prior to setting audit date. This will depend on the number of waste streams to be audited. There is a specific audit and a general audit. It takes between 2 and 3 days. It will take an additional extra day per waste stream. This is a rough estimate. Minimum times are set to ensure the quality of the audit. The time depends on the capacity of the facility and the waste streams. The audit involves weighing output fractions, checking depollution, collecting information, checking where output fractions go, etc. All performed to obtain recovery rates accurately.

23. How would you rate Ireland's compliance with the WEEELABEX standard for LCD recycling in particular? Can you elaborate?

    - No reply. Two operators have the standard.

24. Ireland has three WEEELABEX approved operators and two trained auditors: is this correct or up to date?

    - Not sure but I think this is correct. The WEEELABEX website is up to date.

25. What are the future plans for the WEEELABEX standard?

    - The WEEELABEX will work with the CENELEC. If similar, then the purpose of the WEEELABEX would have been to prepare the CENELEC.

26. WEEELABEX is being registered as a CENELEC standard. When will this process be completed and what will it mean for WEEELABEX?

    - It is still waiting to complete this process. Five documents published so far from the CENELEC. There is still a lot under development. It is expected that this process will be completed sometime this year, end of 2016/early 2017.

27. What are the main obstacles for WEEE operators that you feel might hinder their full compliance with the WEEEBLEX standard, specifically with respect to depollution of LCDs?

    - FPDs still only account for 2–5% of total amount of TVs recycled so manual disassembly is still sufficient for the moment. The collection rates are still very low and this is not the main area of business for most recyclers (WEEE Operators). Appliances are still a relatively new technology. Operators try to increase their output fractions. Often they try to find companies downstream to sell on the FPDs to. The operators are asking where they can send FPDs to downstream. Operators are waiting for the market to merge. This information is from two years ago so maybe the situation has changed now. Only three years ago CRT treatment plans were still being built. Mainly hot wire technology applied to split CRT tubes. Or alternatively, cut them, as this is a low investment technology.

28. What would you feel would further promote the recycling of LCDs across the EU?

    - Automated technology would further promote recycling of LCDs. Only 3 or 4 operators have some form of automated technology. It is often too expensive for most mainly because they do not collect enough WEEE LCDs. A cheaper mid-scale solution requiring low investment to auto-dissemble would be good at the moment. Also market solutions for output fractions from LCDs because if operators find that they are not valuable they will request support. So it would be a good idea to find good markets for output fractions. Operators get more money if they treat waste. If downstream prices are low then operators will lose money. Operators are always looking for solutions. Efficiency, time, man hours, less time to treat, etc. are all important factors. It is all about recovery rates.

Survey 3: Relevant Organisation

29. How would you rate the current level of compliance with the WEEELABEX standards across the EU regarding the depollution of LCDs?
    - Low to medium.
    - Only three countries have introduced the standards: the Netherlands, Ireland and hopefully France.

30. Five organisations have conformity verification to process FPDs. Are a large volume of LCDs being processed by these operators? If so, can you give an estimated volume?
    - In 2014, less than 10% FPDs in this country's intake.

31. Regarding the technical standard, Clauses 5.3.1 and 5.3.2 look to specify particular thresholds for mercury – how and when will these threshold values be set? Please elaborate.
    - These have not been determined yet but will be published with the new EN Standard 50625–3 due next year. As a guide, the threshold for mercury in lamps is 5 mg.

32. Clause 5.4 relates to requirements for the ITO contained within LCDs. However, it is not mandatory to recover the ITO. Do you foresee further advances in the standard regarding ITO recovery or setting of a threshold recovery target for ITO in the future? If so, please elaborate.
    - Do not know.

33. Are any of the WEEELABEX operators listed above for the FPD currently recovering the ITO? If so, please elaborate.
    - Not aware of any at the moment.

34. Clause 5.5 relates to requirements for the fluorescent coatings contained within LCDs. However, it is not mandatory to recover the fluorescent coating. Do you foresee further advances in the standard regarding fluorescent coating recovery or setting of a threshold recovery target in the future? If so, please elaborate.
    - If this question refers to the fluorescent coating of the CCFL tubes then it will be governed by the lamp WEEELABEX standard. It is not a specific requirement under Annex VII of the WEEE Directive but it may be driven in the future by demand from manufacturers/producers.

35. Do you foresee any forthcoming changes to the WEEELABEX standards, specifically with respect to LCD depollution?
    - The WEEELABEX organisation should know the answer to this question.

36. Can you briefly outline the auditing procedure for an operator and how long it takes to complete an audit?
    - The audit process involves a number of steps:

        (a) The operator will issue a Declaration of Intent to the WEEELABEX organisation and make contact with a listed WEEELABEX lead auditor.

        (b) The auditor will conduct a site audit. The audit will take approximately one day, depending on the size of the site and the number of auditors.

        (c) They will also conduct pre-audit (off-site) checks of licences, permits, etc., that are requested as part of the pre-audit paperwork. (1 day's duration)

        (d) Batch analysis must be conducted in the presence of an approved auditor. (1 day's duration)

        (e) Reports are issued. An initial draft of findings is sent for review to the operator for comment. The operator has three months to address any non-conformity. The final report will be issued stating "pass" or "fail".

        (f) Questions used during the audit are split into "Priority 1" and "Priority 2".

- 100% of priority 1 questions must be complied with.
- 70% or greater of priority 2 question must be complied with.

37. How would you rate Ireland's compliance with the WEEELABEX standard for LCD recycling in particular? Can you elaborate?

    - Good. All FPDs collected by two compliance schemes and sent for recycling to WEEELABEX certified sites here in Ireland or Europe. Cannot say the same for business-to-business (B2B) FPDs. The EPA manages the waste management plans for B2B producers. Also a lot of B2B FPDs would never arise as waste and enter the reuse market. This is according to a report on WEEE from the United Nations University.

38. Ireland has three WEEELABEX approved operators and two trained auditors: is this correct or up to date?

    - This is correct. Auditors from the UK and Europe conducted the compliance audits for certified recyclers in Ireland.

39. What are the future plans for the WEEELABEX standard?

    - The publication of an official EU standard will supersede all other standards.

40. WEEELABEX is being registered as a CENELEC standard. When will this process be completed and what will it mean for WEEELABEX?

    - Not sure.

41. What are the main obstacles for WEEE operators that you feel might hinder their full compliance with the WEEEBLEX standard, specifically with respect to depollution of LCDs?

    - One of the main obstacles is the lack of interest in the UK.
    - There is a perceived lack of value in waste FPDs.

42. What do you feel would further promote the recycling of LCDs across the EU?

    - Manufacturer/producer awareness of the standards due to their participation in EPEAT, which forces them to look at the whole life cycle of the display products they produce. It is a voluntary standard for display and print electronics. It recognises R2 and WEEELABEX as applicable standards for downstream treatment of the products. The continuous implementation of the standards in the Netherlands, Ireland and France is proving to doubters that it can be achieved and not at the excessive cost that was predicted.

Survey 4: Relevant Organisation

43. How would you rate the current level of compliance with the WEEELABEX standards across the EU regarding the depollution of LCDs?

    - Currently, the depollution of LCDs mainly involves manually dismantling. The operator removes the backlighting with care by trying to pop the lights out without breaking them. This is an easy method but very time-consuming.
    - Shredding is also limited for correct depollution purposes, as it is difficult to know how much mercury contamination occurs. Organisation expressed reservations about standard shredding methods.
    - Also capacitors and polychlorinated biphenyls (PCBs) are problematic. Unless there is a very good picking line, hazardous components will be missed.

44. Five organisations have conformity verification to process FPDs. Are a large volume of LCDs being processed by these operators? If so, can you give an estimated volume?

    - With reference to the throughput for the whole of country; currently, 8–10% of overall TVs are FPDs. There are still a lot of CRTs coming through the waste stream. We should really begin to see a shift in the display stream soon. Hoarding is an issue. Historically, buying a CRT was a much bigger investment for consumers than today. To put this in perspective, it would typically cost a month's salary to buy a CRT 10–20 years ago, while the cost of a standard FPD equates to a week's salary. Therefore, there is a physiological attachment to CRTs and this can partially explain the delay in CRT recycling.

45. Regarding the technical standard, Clauses 5.3.1 and 5.3.2 look to specify particular thresholds for mercury – how and when will these threshold values be set? Please elaborate.

    - Mercury (Hg) is difficult to depollute, as it readily forms amalgams with other metals and hides in dust particles. Daniel Picha is the expert on the technical aspects of the WEEELABEX. Discussions are ongoing with technical specialists that inform the CENELEC. Currently trying to adopt the WEEELABEX with the CENELEC but this is a moving picture. The WEEELABEX organisation should know the answer to this.

46. Clause 5.4 relates to requirements for the ITO contained within LCDs. However, it is not mandatory to recover the ITO. Do you foresee further advances in the standard regarding ITO recovery or setting of a threshold recovery target for ITO in the future? If so, please elaborate.

    - No, I don't foresee it being mandatory to recover ITO. Unless there is an economic benefit for a treatment operator or it dictated that they should, then they will not do it voluntarily.

47. Are any of the WEEELABEX operators listed above for the FPD currently recovering the ITO? If so, please elaborate.

    - I cannot comment on this due to confidentiality agreements. You may contact operators directly.

48. Clause 5.5 relates to requirements for the fluorescent coatings contained within LCDs. However, it is not mandatory to recover the fluorescent coating. Do you foresee further advances in the standard regarding fluorescent coating recovery or setting of a threshold recovery target in the future? If so, please elaborate.

    - This would depend on the particular treatment operator and lamp operator, in particular. It would also depend on where the recycler is based due to the so-called "proximity principle". If a treatment operator is close to an incinerator, landfill, etc., this will influence how they process the WEEE.

49. Do you foresee any forthcoming changes to the WEEELABEX standards, specifically with respect to LCD depollution?

    - I don't foresee any changes at the moment. Maybe in a few years there will be some changes with respect to the rare earth elements.

50. Can you briefly outline the auditing procedure for an operator and how long it takes to complete an audit?

    - There are two types of auditors:

        (a) Independent auditor

        (b) WEEE auditor (works for a compliance scheme)

    - It has been decided that people who work for a compliance scheme will no longer audit but this decision keeps being put back. The duration of audit depends on the volume and methods involved. A rough estimate would be 1–2 days.
    - For two auditors, 1 full day is required for documents, downstream.
    - Lead auditor, 1 full day for batch auditing, depending on stock levels.
    - Don't stockpile, continuously processed.
    - 1 day to achieve 5 tonnes. Hg analysis sent to labs.
    - 3–4 days for audit/lab/report/etc.

51. How would you rate Ireland's compliance with the WEEELABEX standard for LCD recycling in particular? Can you elaborate?

    - An audit of one site for FPD was conducted (Recycling Village) and they passed.

52. Ireland has three WEEELABEX approved operators and two trained auditors. Is this correct or up to date?

    - This information is on www.weeelabex.org.
    - Conformity verification gives all auditors and operators.

- Some small operators do B2B for laptops, etc., but these are not under household WEEE.

53. What are the future plans for the WEEELABEX standard?

    - No response.

54. WEEELABEX is being registered as a CENELEC standard. When will this process be completed and what will it mean for WEEELABEX?

    - There is a group of experts working on compatibility between the two. Meetings are currently ongoing. There are lots of people asking this question but you need to ask the WEEELABEX directly. One CENELEC standard for fridge treatment is a mirror of the WEEELABEX. There is a question on how they will work together. Auditors have to audit to the CENELEC standard. If the WEEELABEX is the same then it will align with the CENELEC. It will depend on whether or not industry finds it difficult to get an auditor. It remains to be seen.

55. What are the main obstacles for WEEE operators that you feel might hinder their full compliance with the WEEELABEX standard, specifically with respect to depollution of LCDs?

    - Shredding procedures are an obstacle to Hg recovery. This raises questions about contamination of other fractions. The shredding of capacitors and PCBs are an issue. Also BFRs (Brominated Flame Retardants) are a big issue for WEEE operators.

56. What would you feel would further promote the recycling of LCDs across the EU?

    - Most LCDs can’t fit in household waste bins so they have to be taken somewhere (seller or recycling centre). Currently it is free of charge. The key is really further education. There is little take-back in the UK because there is no obligation on sellers. Currently, they pay into a distributor take-back scheme which then pays local authorities to perform this service.
    - Producer responsibility is crucial. This is where, regardless whether or not they make money, producer responsibility has to cover the recycling costs. However, it is often market forces that determine what actually happens.
    - When we first started in this industry, CRTs were still being made. Back then, manufacturers such as Samsung bought back leaded glass for smelting and reuse. So the waste fraction was considered valuable. However, now the CRT market is diminishing and it is unclear whether or not CRTs are still being made. Now you have to pay to get rid of the glass rather than making money from it. This is an example of how recycling is strongly dependent on market developments.
    - Producer responsibility keeps treatment operations recycling in spite of market conditions. Sellers such as Sony, Proctor & Gamble, etc., are all registered with a compliance scheme. Sellers wanted a one-stop shop for compliance schemes rather than burdening themselves in terms of responsibility.
    - Producers pay the compliance schemes in terms of what they put on the market. For example, if a manufacturer produces the games machine, they would first give the weight of the appliance. Then work out how much they owe and then pay the compliance scheme to go out and collect that amount. There is no government funding involved. The producer is responsible for recycling and this will then force the producers to consider re-engineering new products that are easier to recycle. This approach forces producers to look for alternatives. It is a twofold approach with focus on producer responsibility: they pay all the way down the chain. Producers build in this cost as part of the production cost. They factor it in. This takes the burden off governments. No money passes hands. Everything needs to be viewed in terms of “resource management”.

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.

EPA Research Report 201

Investigation of Liquid Crystal Displays as a Source of Indium

epaResearch
Climate - Water - Sustainability
Identifying pressures • Informing policy • Developing solutions

Authors: Lisa O’Donoghue and Paul Moroney

The objectives of the research were to investigate and explore the potential for indium recovery from LCDs.

Inform policy

A review of the requirement for LCD recycling from a policy perspective was undertaken. The WEEE Directive contains specific clauses that required the appropriate recycling of LCD displays. The Directive is now also being augmented by the industrial standards such as WEEELABEX which contains more specific targets regarding handling and recycling LCDs including threshold values for mercury and consideration of indium tin oxide fractions. As the WEEELABEX standard is currently in the process of being developed into the CENELEC standard monitoring and inputting to the developments is important from a national policy perspective.

Develop solutions

In order to develop a cost-effective solution for recovering indium from LCDs, it is first necessary to understand the amount of indium contained within LCDs and the currently available recovery methods. Findings from 10 samples tested revealed that indium levels varied significantly (38 to 292 mg indium per kg glass panel) which will ideally require a recovery process that is sensitive to these range of variations. Reported recovery methods include traditional hydrometallurgy as well as novel techniques such as lift-off method, sub critical water process, bio-treatments and mechano-chemical treatments. While specific novel techniques such as lift-off and critical water processes potentially offer a good balance between cost, environmental impact, efficiency and time, they are at the early stages of testing and development and have not yet entered the market place. These processes are detailed and reviewed within this desk based study and represent a foundation of knowledge on which to develop and test a future solution.

EPA Research: McCumiskey House,
Richview, Clonskeagh, Dublin 14.

Phone: 01 268 0100
Twitter: @EPAResearchNews
Email: research@epa.ie

www.epa.ie

EPA Research Webpages
www.epa.ie/researchandeducation/research/