detritus | Multidisciplinary Journal for Circular Economy and Sustainable Management of Residues

# CHEMICAL RECYCLING OF PVC-CONTAINING PLASTIC WASTE FOR RECYCLING OF CRITICAL METALS

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**Article Info:**
Received:
12 September 2023
Revised:
16 March 2024
Accepted:
19 March 2024
Available online:
25 March 2024

**Keywords:**
Circular economy
Chemical recycling
Critical metals
PVC-waste
LED recycling
LCD panels recycling

**ABSTRACT**
Chemical recycling of polyvinyl chloride containing plastic waste to recover critical metals is a promising way to solve two important problems (polyvinyl chloride disposal and critical metal recovery) in waste management and is being transferred on a larger scale in the "CHM-Technology" project. Various polyvinyl chloride containing plastic wastes were pyrolyzed to generate a hydrogen chloride rich vapor. This hydrogen chloride rich vapor is used in a second step to chlorinate indium in liquid crystal displays. Indium chloride has a lower boiling point than indium-tin-oxide and evaporates. This is cooled down and generates a metal concentrate together with the decomposed volatile materials from liquid crystal displays. The chlorine content in the polyvinyl chloride containing plastic waste residues is reduced. The products solid, oil, hydrochloric acid and gas can be used for new products. For metal purification the metal concentrate is mixed with water, filtrated, distilled and an electrolysis is carried out to recover metallic indium. Nine waste containing PVC were used with significant differences: when more hydrochloric acid and less volatile organic fraction was produced, more indium was transferred to the metal concentrate. The best recovery of indium (78% purity after electrolysis) was 39 wt-% from LCD panels containing 83 mg In/kg.

## 1. INTRODUCTION

Plastics are one of the greatest inventions of the 20th century. Plastic products are used in many fields such as packaging, building and construction, agriculture, and many other applications, due to their light weight, mouldability, long life and low price (Geyer, Jambeck & Law, 2017). Plastic production has continuously increased in the last decade. The annual production exceeded 330 million metric tons (Mt) from 2016 to 2020. Polyvinyl chloride (PVC) accounted for 9.6% of all plastics in 2020, ranking in third place. There are different methods to dispose plastic waste (Geyer et al., 2017; Geyer, 2020). First, incineration decreases the volume of waste, and the resulting energy can be used for heating or generating electricity. However, incineration of plastic waste emits excessive $CO_2$ and may release some hazardous substances, such as polychlorinated biphenyls, dioxins, furans, and persistent organic pollutants (POPs) (Shibamoto et al., 2007). Further problems with the incineration of wastes with PVC are deposits on heat transfer tubes and the presents of Cl-species like potassium chloride, sodium chloride, hydrochloric acid and chlorine, which results in corrosion (Ma et al., 2020, Miliute-Plepiene et al., 2021, Lu et al., 2023). The cost for PVC-containing plastic waste is 275 €/t in Germany (Hofmann, 2021). Second, plastic waste can be dumped in sanitary landfills, which is a convenient and cost-effective, but landfill space is limited in many countries and an inadequate landfill can cause serious environmental pollution (Hopewell et al., 2009; Teuten et al., 2009; Wang, Ko, Liu & Xu, 2021). In some countries, such as Germany or Austria, the landfilling of Total Organic Carbon (TOC)-containing waste is prohibited (Bundesministerium der Justiz und für Verbraucherschutz, 2009). Third, mechanical recycling is suitable for various types of plastic waste (Braun, 2002). End-of-life PVC degradation causes release of chlorinated products into the surrounding environment (Maringer et al., 2017). Mechanisms of degradation during recycling include thermo-oxidation, crosslinking, functionalization of chains and hydrodechlorination (Awaja & Pavel, 2005; Sadat-Shojai & Bakhshandeh, 2011). PVC is unstable to both thermal and photoinduced stress. PVC waste streams must be pure and highly stabilized against unwanted degradation to enable useful mechanical recycling (Schyns &

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https://doi.org/10.31025/2611-4135/2024.18360

Shaver, 2021). A fourth method called "chemical recycling" summarized the development of alternative techniques for plastics recycling like pyrolysis, gasification and liquefaction (Quicker et al., 2022; Scheirs & Kaminsky, 2006). For PVC-containing plastic waste gasification and pyrolysis are mainly in the focus of research. For example, the company Ecoloop in Goslar (Germany) produces synthesis gas from PVC-containing plastic waste, but the chlorine (Cl) is absorbed with calcium oxide, where calcium chlorine is generated (VinylPlus, 2015). In this case the hydrogen chloride (HCl) or chlorine can`t be used for further processes. Therefore, pyrolysis of PVC can be used and the techniques like two-stage pyrolysis, co-pyrolysis or ad-/absorption of halogens in the vapor phase were tested for different purposes like removing chlorine in oil from mixed plastic waste (Bockhorn & Hornung, 1998; Liu et al., 2013; Muhammad et al., 2015; Shen et al., 2016).

Electronic products are another essential component of modern society, but their importance has perhaps never been more noticeable than when the COVID-19 pandemic forced almost every aspect of human interaction going online. However, the pandemic also demonstrated that the supply chains that provide crucial raw materials for manufacturing electronics are increasingly vulnerable to social, geopolitical, and technical disruptions. These vulnerabilities are likely to increase in the future, due to global health crises, natural disasters, and worldwide political instability, all of which will be magnified by looming climate change impacts almost 40 metals and minerals that provide critical functionality to electronic products (Althaf & Babbitt, 2021). A lot of these metals used in electronic products especially specialty metals like rare earth elements, gallium or indium (In) have End-of-Life-recycling rates (EoL-RR) of less than 1% (Graedel et al., 2011). In case of indium, EoL products are considered to be a promising secondary source (Lokanc et al., 2015). In the EU, total indium consumption is estimated at 64 tonnes per year from 2012 to 2016, with 60% of the total indium used in flat panel displays (FPDs), mainly in the form of indium tin oxide (ITO). In addition, 11% and 9% of the consumption is used in solders and photovoltaics, respectively, while the remaining 20% is used in thermal interface materials, batteries, alloys/compounds, and semiconductors & light emitting diodes (LED) (European Commission, 2020). This highlights the importance of considering EoL electronic products as a viable source of indium, particularly for FPDs. The liquid crystal displays (LCDs) have the highest market share among FPDs (Zheng et al., 2023) (Figure 1).

Pyrolysis of PVC was tested to use the generated hydrogen chloride for the recovery of indium from indium-tin-oxide powder and LCDs (Park et al., 2009). The utilization of hydrogen chloride from PVC for the recovery of metals like indium from other waste streams was inspiring to do further research with different PVC-containing material for the recovery of indium from LCDs and LEDs in the projects "chlorine platform" and "CHM-Technology" (Berninger & Peer, 2022).

## 2. MATERIALS AND METHODS

### 2.1 PVC-containing plastic waste and LCD-panels

PVC-containing materials were used for experiments further explained in chapter 2.3: automotive shredder residue (4,350 ± 57 mg/kg Cl), sorting residues from lightweight packaging (2,827 ± 196 mg/kg Cl), shredder residue from waste of electrical and electronic equipment (3,400 ± 328 mg/kg Cl), window frames (237,263 ± 6,233 mg/kg Cl), cable (129,442 ± 5,479 mg/kg Cl), shutter (192,827 ± 7,120 mg/kg Cl), PVC profiles (249,023 ± 6,043 mg/kg Cl), PVC-pipes (353,762 ± 10,138 mg/kg Cl) and wall paper (58,033 ± 3,496 mg/kg Cl), where the particle sizes are lower than 10mm. EoL–LCD-panels (83 ± 5 mg/kg In) were shredded to <40mm for the experiments.

### 2.2 Energy Dispersive X-Ray Fluorescence Spectroscopy

The elemental content in the input materials and products were analyzed by the X-ray spectrometer Epsilon 3XLE from PANalytical Kassel. The device was calibrated with the "omnian" standard from PANalytical Kassel, and the evaluation of the data was performed with the Epsilon Benchtop software of the same manufacturer. The elements from sodium to americium can be analyzed. The focus of this work is on the reduction of chlorine from PVC-containing materials and the volatilization of indium from LCDs, which

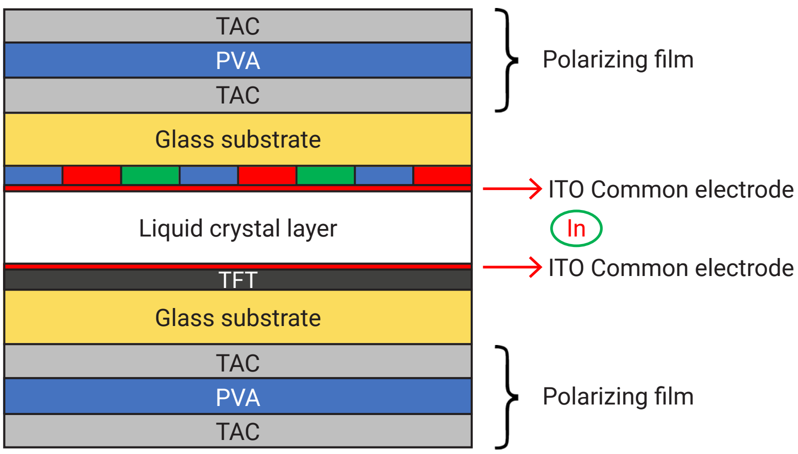


**FIGURE 1:** Lamination structure of the ITO layer from LCD panel (Zheng et al., 2023).

is why only the chlorine content is given for the PVC-containing materials and only the indium content for the LCDs. The solid samples were crushed to <1 mm with the cutting mill of the company Retsch at 600 rpm for analysis and the liquid samples were analyzed directly.

### 2.3 Experimental set-up

PVC-containing materials were treated in a semi-contınous pyrolysis plant with a halogenation reactor (Figure 1). The Halogenation reactor was filled with 2,100 g LCDs before the plant was set-up, except for wallpaper, window frame and PVC-pipes. For these three materials, the Halogenation reactor was filled with 1050 g LCDs. The halogenation reactor is self-made with 1.4571 steel, inner diameter of 50 mm, a wall thickness of 3mm and electric heated with two heaters each with a power of 3 kW. The reactor of the pyrolysis plant is self-made. Prior to treatment, the plant was set-up andflushed with nitrogen to remove the oxygen at a gas flow of 0.5 liters per minute. The pyrolysis reactor was heated up to 650°C. The halogenation with LCDs was heated up to 650°C. The polarizing film decomposed to solid, gas and oil. The oil evaporates and condenses in cooling unit 3 as well as in cooling unit 4. 750 g PVC-containing material was filled into a cartridge in six batch with each batch 125 g. Only for wallpaper, window frame and PVC-pipes the masses were 375 g and each batch 62.5 g. The mass ratio between LCDs and PVC material was 1:2.8 in all experiments. The exact masses are listed in Table 1. After inertization with nitrogen, the PVC material from the cartridge was added to the reactor. The cartridge was flushed again with nitrogen to remove pyrolysis vapors. The thermochemical treatment and decomposition took place in the pyrolysis reactor at 650°C. Coke and pyrolysis vapor is generated. The coke remained in the pyrolysis reactor, was stirred and was transferred to the char container after 30 min dwell time. Then the next batch was filled in. The vapor flowed from the pyrolysis reactor through the cooling unit 1 and cooling unit 2, where it was cooled down and oil condensed. Then the HCl rich vapor reached the halogenation reactor. Here the HCl reacted with the indium in the LCDs to indium chloride. The indium chloride evaporated at 650°C and condensed at cooling unit 3 and cooling unit 4. The unreacted HCl was removed from the gas stream by scrubbing it at first in a washing bottle with water and then in two washing bottles with sodium hydroxide. In the bottle with cotton fine liquid particles were removed from the gas stream. The gas was analyzed with a gas analyzer from the company Pollutek. The measuring principles for the gases CO, $CO_2$, $CH_4$, and $C_nH_m$ were a non-dispersive infrared sensor, $O_2$ was a electrochemical detector and for $H_2$ it was a thermal conductivity detector. The gas wascleaned with activated charcoal and released to the environment (Figure 2).

### 2.4 Recovery of the critical metal indium from metal concentrate

The indium was enriched in the liquid metal concentrate but mixed with organic material from the polarizing film decomposition from LCDs. The metal concentrate was mixed with water in a volume ratio of 1:1.75 and particles conglomerated. Then the particles were removed, by filtering two times with a 2µm filter. The metal concentrate and filtrate is shown in Figure 3. For electrolysis the indium concentration was enriched in the solution. Therefore the filtrate was distillated to remove the solvent. The indium concentrate for electrolysis was 15 gram per liter and a volume of 10 ml. The electrolysis was conducted at a current of 5.2 volts and 0.1 ampere with electrodes of platinated titanium with a diameter of 6mm for one hour.

## 3. RESULTS AND DISCUSSION

The pyrolysis of the PVC-containing materials generates the products solid residue, oil and gas. The mass of oil is containing the liquid in the bottles under cooling 1 and cooling 2 as well as the residue in the pipes from the cooling unit 1 and cooling unit 2. The masses are listed in Table 1.

The most amount of solid residue was produced from the automotive shredder fraction. Due to the low chlorine content of the input material, not much oil and gas was produced. The most gas produced in the from the input-material PVC profiles. Here the chlorine content was reduced by 60.5% (Table 2). The most oil was produced from the input-material shredder residue from waste of electrical and electronic equipment. This is caused due to the high polyolefin content. The chlorine content of the solid residues

**TABLE 1:** Input masses [g] and products solid residue [g], gas [g] and oil [g].

| PVC-containing materials | Input material [g] | Solid residue [g] | Gas [g] | Oil [g] |
|---|---|---|---|---|
| automotive shredder fraction | 752.55 | 572.45 | 115.20 | 64.90 |
| sorting residues from lightweight packaging | 751.3 | 206.05 | 262.25 | 283.00 |
| shredder residue from waste of electrical and electronic equipment | 751.00 | 201.30 | 199.65 | 350.05 |
| window frames | 375.15 | 98.05 | 232.00 | 45.10 |
| cable | 751.60 | 438.1 | 120.85 | 192.65 |
| shutter | 751.00 | 332.45 | 334.35 | 84.20 |
| PVC profiles | 751.35 | 233.45 | 414.00 | 103.90 |
| PVC-pipes | 375.7 | 151.25 | 164.60 | 59.85 |
| wallpaper | 374.95 | 145.15 | 126.55 | 103.25 |

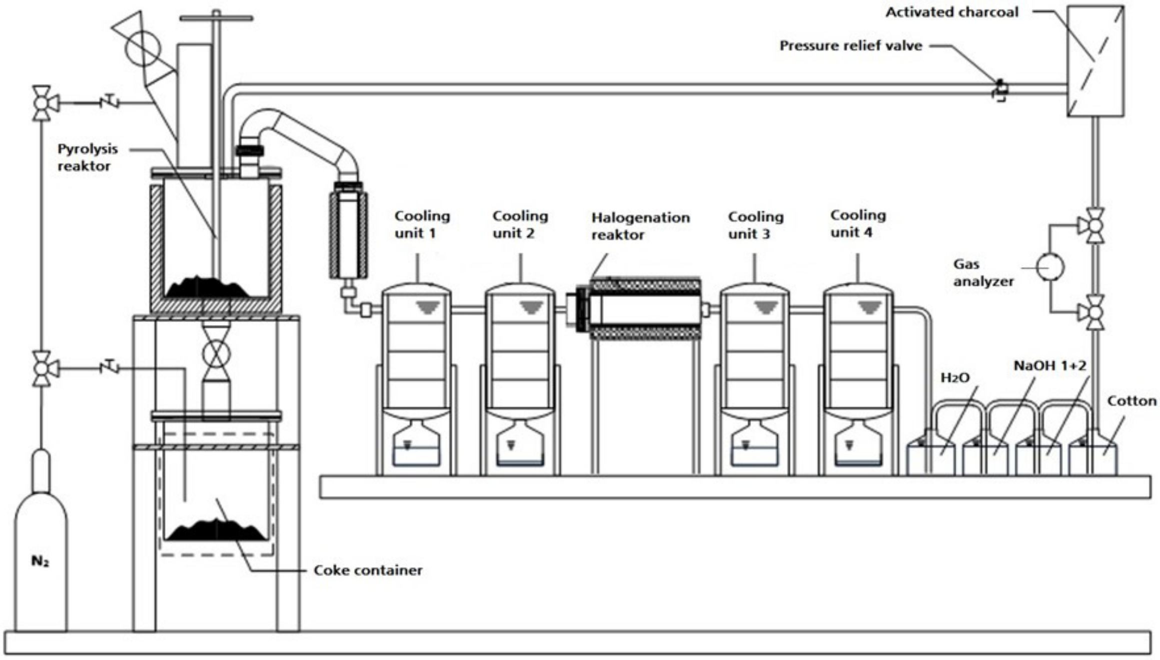


**FIGURE 2:** Schematic set-up of the plant for the experiments.

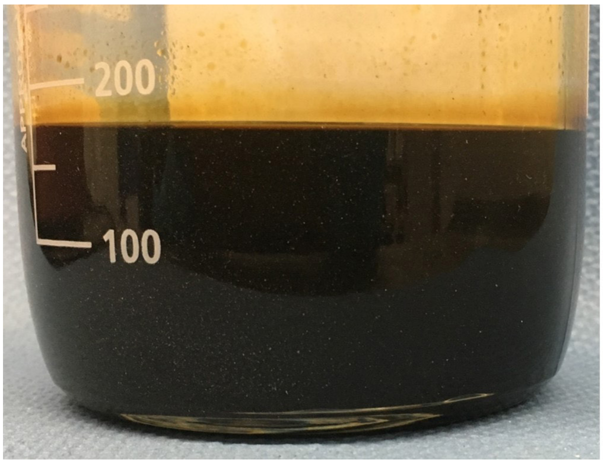


**FIGURE 3:** Metal concentrate (left) and filtrate (right).

**TABLE 2:** Chlorine removed from PVC-containing material [wt.-%], indium transferred to metal concentrate [wt.-%] and indium in metal concentrate [ppm].

| PVC-containing materials | chlorine removed [wt.-%] | Indium transferred to metal concentrate [wt.-%] | Indium in metal concentrate [ppm] |
|---|---|---|---|
| automotive shredder fraction | 8-10* | 0.9 | 11 |
| sorting residues from lightweight packaging | 8-10* | 6.2 | 61 |
| shredder residue from waste of electrical and electronic equipment | 8-10* | 26.8 | 393 |
| window frames | 94.8 | 29.9 | 335 |
| cable | 57.3 | 13.3 | 126 |
| shutter | 37.9 | 38.9 | 392 |
| PVC profiles | 60.5 | 33.3 | 1057 |
| PVC-pipes | 86.3 | 29.5 | 267 |
| wallpaper | 99.0 | 67.7 | 1230 |

** chlorine reduction calculated by subtraction of the chlorine mass from the products except solid residue from the input mass of chlorine.*

were from automotive shredder fraction 9,167 ± 871 mg/kg, sorting residues from lightweight packaging 51,432±1,430 mg/kg, shredder residue from waste of electrical and electronic equipment 14,780±1,215 mg/kg, window frames 48,209 ± 926 mg/kg, cable 95,326 ± 17,114 mg/kg, shutter 279,746 ± 5,019 mg/kg, PVC profiles 322,837 ± 1,512 mg/kg, PVC-pipes 120,479 ± 1,778 mg/kg and wallpaper 1,466 ± 291 mg/kg. The highest chlorine content in the solid residue is in the PVC-profiles.

The calorific value of the solid residues were from automotive shredder fraction 2.60MJ/kg, sorting residues from lightweight packaging 19.96 MJ/kg, shredder residue from waste of electrical and electronic equipment 8.13 MJ/kg, window frames 22.74 MJ/kg, cable 13.75 MJ/kg, shutter 10.86 MJ/kg, PVC profiles 13.01 MJ/kg, PVC-pipes 17.39 MJ/kg and wallpaper 11.44 MJ/kg. The highest calorific value of the solid residue was from the window frames.

The chlorine mass in the PVC-containing materials could be reduced between 8% and 99% via pyrolysis. The results are shown in Table 2. Hereby the reduction is the difference between the chlorine mass of the input-material and in the solid residue after pyrolysis. The indium content could be enriched in the metal concentrate up to 15 times in comparison to LCDs. This reduces the mass in the following process steps to the metallic indium and decreases costs. Between 0.9 wt.-% and 67.7 wt.-% of the indium from the LCDs were transferred to the metal concentrate. This shows that some PVC-containing materials are better suited for this recovery of indium.

Normally by increasing the weight percent from indium transferred to metal concentrate, the indium in the metal concentrate should also increase, when the metal concentrate was only containing the indium and decomposed components from the LCDs. This is not the case. Therefore, this indicates that the gas stream was not completely purified from oil after cooling unit 1 and cooling unit 2. The cooling system has to be improved for further experiments to avoid dilution with oil from PVC-containing materials. When more chlorine is removed from the PVC-containing material, then more indium should be transferred to the metal concentrate. This tendency was the case, but this does not explain the difference between window frames and wallpaper. Here the difference was in the amount of gas, which was produced. The wallpaper produced 126.55 g gas and window frames 232.00 g gas. When more hydrochloric acid and less organic gaseous fraction was produced, then more indium was transferred to the metal concentrate.

The recovery of indium from the metal concentrate was achieved by mixing with water, filtration, evaporation and electrolysis. After filtration of the mixture of the metal concentrate with water 2/3 of the indium from the metal concentrate is in the filtrate and the rest in the filter residue. After the removal of solvents, the solution contains 15 g In/L. From electrolysis 20mg indium were recovered as metal and a yield of 57% from the indium in the metal concentrate was achieved by this process steps. The purity is 78% indium. This yield could be increased if the filterresidue is used as an additional input material for the halogenation reactor and if the electrolysis solution has a higher indium content, because then the current yield is higher. The best recovery of indium (78% purity after electrolysis) was 39wt-% from LCD panels containing 83 mg In/kg.

Here no dioxin or furan analysis was performed as the research focus was more on screening and product characterization. The de-/chlorination processes should be tested in continuous processes. Another promising waste stream with a lot of critical metals are LEDs. All these aspects will be examined in the project “CHM-Technology”.

## 4. CONCLUSIONS

Essential products from the pyrolysis process of PVC-containing plastic waste like solid residue, oil and gas can be used. In a pyrolysis reactor, which is used for industrial application, the generated products must be analyzed for dioxins/furans to be sure that no hazardous products are generated. There are PVC-containing plastic wastes like window frames, where a lot of chlorine remains in the solid residue. The pyrolysis process and combination with the total indium recovery yield of 30 wt-% with 78% purity are up to now only in small technical scale. This must be upscaled in a continuous process. The recovery of indium from LCDs is possible with the combination and further process steps. Here the limits are the heaters and material for the halogenation reactor to handle the heat together with the corrosive environment. For further byproducts from the process steps from the metal concentrate to the metal indium recycling opportunities should be found, to keep the cost for an industrial process low.

The chemical recycling of PVC-containing plastic waste for recycling of critical metals is a promising route to solve two important problems in waste management.


## ACKNOWLEDGEMENTS

The project “CHM-Technology“ is part of the joint research project “ForCYCLE Technikum“ funded by the Bavarian State Ministry of the Environment and Consumer Protection (StMUV) and coordinated by the Centre for Basic Materials Efficiency (REZ).


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