DELAMINATION RECYCLING OF MULTILAYER PLASTIC FILMS

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Abstract

Only a small fraction of the plastics produced are recycled, with the great majority landfilled or released into the environment. Mechanical recycling currently used to recycle plastic cannot handle films which constitute about 40% of all plastic packaging. Polyolefin-rich films are suitable feedstock for pyrolysis; however pyrolysis breaks down the polymer chains to produce hydrocarbons that are typically used as fuel, which is not plastic-to-plastic recycling. Research in our group advances molecular recycling, whereby polymers are separated and recovered through selective interactions with solvent. Because the polymer chains do not break and retain their embodied energy, this is a promising, low-energy, low-greenhouse gas (GHG) plastics recycling methodology. This project addresses the recycling of multi-material films which comprise multiple layers of polymer combined into a single film to meet consumer specifications such as preserving food and medicine, acting as oxygen or moisture barrier, and keeping products sterile. This paper highlights the solvent-assisted delamination process that we have developed, which recovers from multilayer multi-material films the majority component polyethylene in the solid form, hence greatly reducing solvent amounts and the corresponding energy needs and GHG emissions compared to dissolution-precipitation recycling. Delamination recycling presents an energy efficient and environment-friendly approach to recover value from the ~17 million metric tons of multilayer plastic films that are produced every year globally.

Keywords: flexible films, advanced recycling, chemical recycling, solvent purification, circular economy

1. Introduction and Motivation

Plastics are useful because their properties are modular and amenable to first principles design. We are approaching one trillion pounds of plastic annual production worldwide, and further growth is expected.[1] Durability, low density, color, and low cost make plastics desirable and long-lasting, but disposable. Thus, plastic waste is increasing, and less than 10% is recycled in the US, with most plastic being landfilled.[2] Addressing environmental concerns, governments are banning certain plastics – despite favorable technical properties. Meanwhile, corporations are committing to incorporating recycled plastic content in products. Opportunities abound for recovering plastic, but technical and economic challenges limit plastics recycling.[3]

Packaging consumes a large fraction (close to half) of the plastics used world-wide. Plastic film production devotes an estimated 25% of production volume to manufacturing multilayer films.[4, 5] Multilayer multi-material films are beneficial, even essential, in several applications, notably in food and pharmaceutics.[6] Multilayer packaging achieves superior protection and durability through the integration of the unique functionalities of the constituent polymers (Figure 1). The major component of multilayer packaging is a polyolefin, on the basis of its low cost, easy processing, excellent moisture barrier properties, and chemical inertness, however, most multilayer packages consist of three or more layers of various polymers, often with an added barrier layer or coating that is based on AlOx and/or other non-polymer materials.[6] As the expectations and specifications for plastic packaging functions become ever more demanding, the complexity of packaging is increasing. This complexity has been presented as an argument (or a pretext?) against the recycling of polymer-based multilayer packaging. Up until now, the great majority of polymer-based multi-material packaging ends up in landfills. This leaves a lot of value to be recovered.

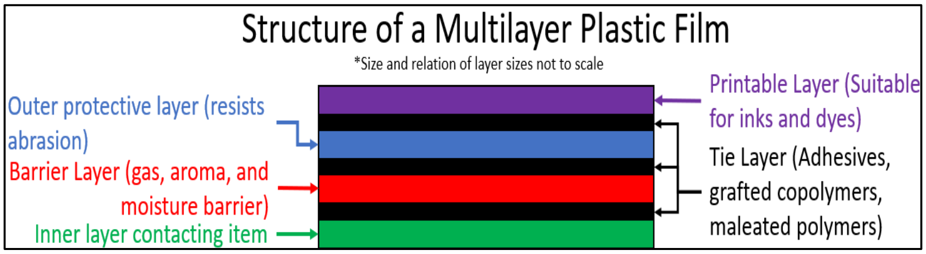


Figure 1: Schematic of a multilayer multi-material film.

It is evident that: (i) plastics offer unique, often irreplaceable, properties and will continue to be used;[6] (ii) plastics are ubiquitous and their waste threatens the environment;[7] (iii) even if sorted, multi-material plastics and flexible films cannot be reprocessed and are being landfilled;[4, 8] and, (iv) value from such plastics can be recovered by chemical recycling such as pyrolysis, but this involves high energy demand and high GHG emissions.[9] These basic premises underscore our research on the solvent-assisted molecular recycling of plastics with a focus on polyolefins and multi-material films.

Upcycling of sorted plastic is achieved through dissolution of select types of plastic in environmentally responsible solvents to recover desirable materials (i.e., polyolefins), separate them from additives or impurities, and render them suitable for reuse. New routes are developed for the recycling of polymer-based multilayer packaging by addressing the delamination of multilayer packaging into the individual constituent films, and the fractionation of the different types of plastic. The innovations advanced here will enable the recovery from post-consumer multilayer packaging waste of useful plastics such as polyolefins, for which great demand exists for incorporation into new products with certain minimum recycled plastic content. Delamination is energy-efficient and low-GHG emitting, as it leaves the majority of the polymer content of multilayer films in its original, solid state, with no polymer degradation involved.

2. Review of Related Work

Plastic film waste buildup has become a global problem. Attempting to reduce use or substitute plastics are good steps to reduce overall film waste volume, but are insufficient.[10] Recycling of plastic film waste is needed. The following processes can be used to this end: mechanical recycling, pyrolysis, and dissolution-precipitation.

2.1. Mechanical Recycling

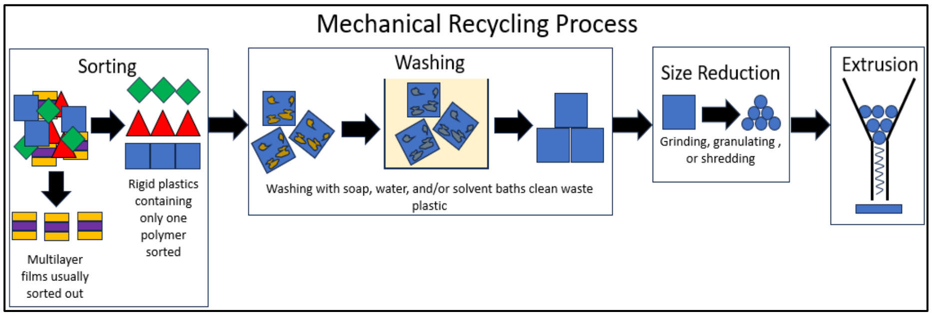


Figure 2: Schematic of mechanical recycling.

Mechanical recycling is the prevalent method used for plastic waste (Figure 2), yet the great majority of current mechanical recycling processes do not handle plastic films. Material Recovery Facilities (MRF) avoid films due to the extra costs in cleaning and handling, and the low-quality of the recycled polymer obtained from plastic film waste[11].

Despite these issues, there have been positive developments in the mechanical recycling of plastic films. For example, Waste Management disclosed a pilot scheme for recovering plastic film waste in Illinois.[12] AMP Robotics is utilizing an AI-powered sorting system to properly sort films, which may be retrofitted onto existing processes.[13] Improvements to sorting technology are valuable to MRFs due to challenges posed by multilayer film waste.[14, 15] EREMA is an example of a company that produces extruders capable of handling both post-industrial and post-consumer plastic film waste for mechanical recycling.[16] Should advancements in sortation and mechanical recycling continue, plastic films could eventually be a viable feedstock for MRFs.

2.2. Pyrolysis

When mechanical recycling is not an option, chemical recycling can be an alternative. The most common method of chemical recycling, pyrolysis, involves thermal decomposition of long-chain polymers at high temperatures and no oxygen (Figure 3).[17] For pyrolysis, the liquid products of diesel and naphtha are desirable for use as fuel and plastic production respectively.[17] Concerns for pyrolysis include its energy consumption and products. Most pyrolysis reactors run at a minimum of 500 °C in order to decompose mixed plastic film waste.[17] The various hydrocarbons produced from pyrolysis need further processing to be turned in a desired liquid product.[17] Regarding desired products, burning diesel as a fuel gives off carbon emissions, and the use of naphtha to produce more plastics could be seen as contributing to environmental problems. Pyrolysis of postconsumer waste plastic into fuel requires large processing volumes to be profitable, and the profitability is very sensitive to the (volatile) price of petroleum.[18]

Pyrolysis has benefits, as it can handle mixed plastic waste films, including contaminated feedstocks.[19] TotalEnergies has pyrolysis plants in Spain, France, and the US that can use flexible plastics as feedstock.[20, 21] According to TotalEnergies, products from its pyrolysis plants are intended to produce high quality materials with a portion to be used for food-grade resins, something not commonly done with plastic film waste.[20] Offering a complement to mechanical recycling is important, however generating non-plastic products and GHG, and involving high energy consumption and costs restrict how widespread pyrolysis of plastic film waste becomes.

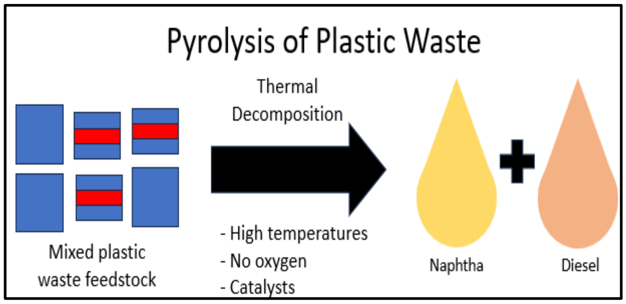


Figure 3: Pyrolysis process products.

2.3. Dissolution-Precipitation Recycling of Plastic Films

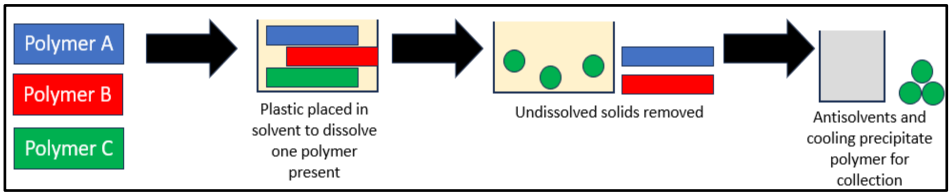


Figure 4: Simplified schematic of dissolution-precipitation process.

Dissolution-precipitation offers the potential of recycling plastic film waste without the high energy consumption and emissions of pyrolysis. Dissolution-precipitation recycling works by selectively dissolving each of the polymer layers (one at a time) (Figure 4).[22] Following filtration of undissolved solids, dissolved material is collected via precipitation.[22] Precipitation is generally performed using an antisolvent, cooling, or a combination of the two.[8] Solvent selection is based on solvent affinity solubility parameters such as Hansen Solubility Parameters. Solvent selection should be improved upon to recognize dangers inherent to specific solvents, as hazardous solvents like toluene are often used based on compatible solvent-solute affinity.[8]

An application of dissolution-precipitation to the case of plastic film is the so-called solvent targeted recovery and precipitation (STRAP) process that works by taking a characterized and identified multilayer plastic film and using specific solvents and antisolvents to dissolve all polymers present sequentially, layer by layer.[23] STRAP is rather energy intensive due to the successive layer-by-layer dissolution and precipitation processes.[8]

3. Technology Approach: Delamination of Multilayer Plastic Films

The flexible films used in packaging often involve more than one type of plastic (Figure 1) and more than just plastic, e.g., paper or metal like aluminum. This multi-material nature leads to waste that is difficult to recycle due to being heterogenous.[24] Delamination, or separation of layers, focuses on sequestering one type of polymer. This can be done by targeting adhesives to separate layers, or through attacking the layer that the plastic is adhered to, such as aluminum.[24] Once the multilayer film is separated via delamination, the remaining strips can then be extracted as monolayer film strips (Figure 5). Compared to the dissolution-precipitation-based process, delamination is less energy and emission intensive as it does not dissolve the majority of plastic. One of the drawbacks of delamination is solvent selection, as there is no standardization for adhesive usage. This can make selecting a proper solvent difficult as the adhesive or tie layer can be composed of many different materials.[25] Delamination is beneficial for dealing with flexible plastic as it recovers solid plastic layers without the polymers degradating[26]. Pretreatment of the

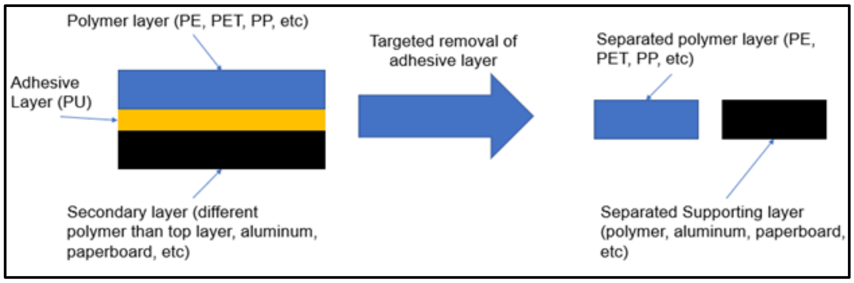


Figure 5: Simplified schematic of delamination process.

plastic waste, such as rinsing of external contaminants and size reduction, would benefit the delamination process.[27] Delamination relies on targeting for recovery a specific layer of the plastic film waste. One method is through selective glycolysis of the adhesives present.[28] Removal of an adhesive layer would promote separation and allow for recovery of the different layers present in the flexible plastic waste.[28] Work was also done to show solvent recovery and carbon emission modeling regarding the solvent selection.[28] Carboxylic acids are reported as good delaminating agents for PET/PE (polyethylene terephthalate / polyethylene) films, but the environmental impact that they have may present a hindrance on a large scale.[26] Use of Aspen Plus® software allowed for the kinetic modeling of a known delamination process.[26] Generated kinetics data facilitated the optimization of the delamination process while also considering carbon emissions and energy consumption in order to improve the process.[26]

In our research we have achieved solvent-based delamination in under an hour in four different multilayer plastic film compositions: LLDPE/Nylon 6/LLDPE, LLDPE/EVOH/LLDPE, HDPE/EVOH/HDPE, and LLDPE/PET. Delamination is induced by dissolving or degrading a minority by mass binding layer (Figure 6) or by partial dissolution and swelling of a targeted polymer layer. Solvents used include carboxylic acids and phenols. The delamination process is performed at a relatively mild temperature (80°C) and results in complete recovery of the PE present initially in the film. Recovered PE purity ranges from 98% to 100% based on solvent selection, temperature, and the time that film spends in the solvent. The delaminated films are recovered through density separation in the delaminating solvent, with all PE being skimmed off the surface of the solvent. The remaining solvent is collected and recycled in a closed-loop process.

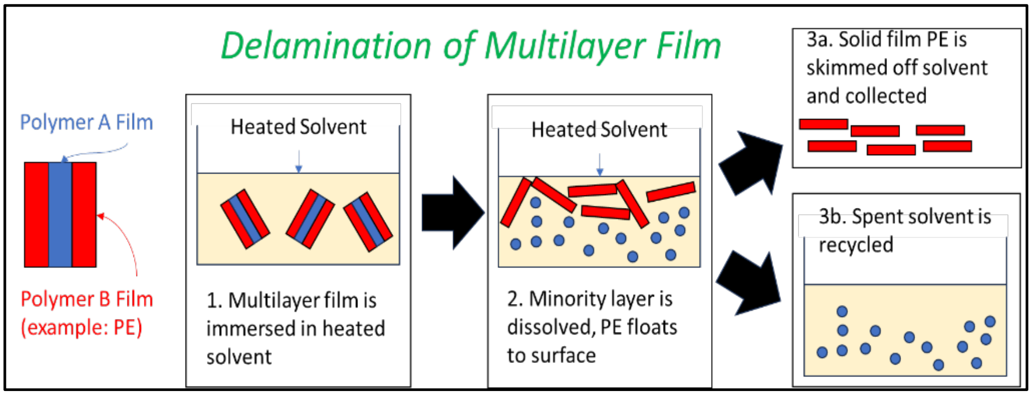


Figure 6: Delamination process via dissolution of minority by mass layer.

Delamination via adhesive removal is exemplified here for the case of LDPE/OPET film in 88% formic acid where the adhesive layer is dissolved or degraded by formic acid, recovering the PE and PET layers as single-polymer solid films in under 30 minutes and with no noticeable solvent contamination. This work is the first to demonstrate formic acid's ability to dissolve or degrade PEI when it is the adhesive layer in a multilayer film structure. Temperatures from 60 °C to 80 °C were tested, with the delamination time ranging from 9 minutes to under 1 minute, respectively to recover one solid PE film and one PET solid film. A key takeaway being increasing temperature accelerates delamination, and it can be achieved in under 1 minute starting at 75 °C to recover two solid films.

The delamination of LLDPE/PET film is induced via swelling of a target layer, PET in this case. We established that m-cresol will not dissolve any PE present in the film up to 90 °C. This work is the first to apply m-cresol as a swelling solvent for PET-containing multilayer films. In addition to releasing solid PE film, delamination via swelling allows also the PET layer to be recovered as solid film, or it may remain in heated solvent for an extended period of time of 24 hours to fully dissolve. Delamination of LLDPE/PET in m-cresol comes with the benefit of having the delaminated solid films being sorted by density in m-cresol. The density of m-cresol, 1.03 g/cm^3, is ideal for having PE layers of film float to the surface to be collected via surface skimming, and the PET layers sink to the bottom of the delaminating solvent, readily recoverable by filtering. Figure 7 shows the rapid delamination times possible via this method.

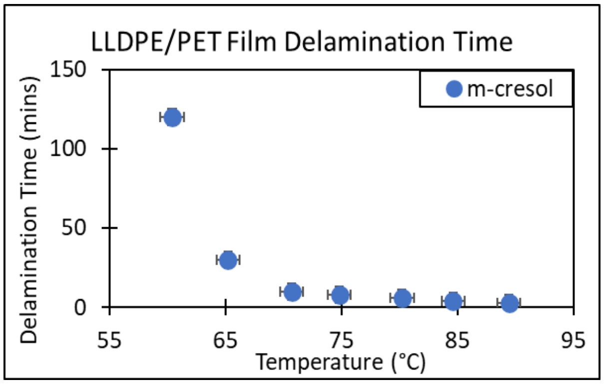


Figure 7: LLDPE/PET delamination time via swelling as a function of temperature. The time required for delamination to be induced is plotted vs temperature.

Delamination of LLDPE/Nylon 6/LLDPE, LLDPE/EVOH/LLDPE, and HDPE/EVOH/HDPE films is achieved via dissolution of a minority by mass layer; both 88% formic acid and m-cresol are found as excellent solvents to this end. DMSO is great for delaminating EVOH-containing films. Once the minority by mass specialty layer has been dissolved, the outer PE layers are unbound and recoverable as solid film. This method results in little to no noticeable solvent contamination on the outer surface of the PE layer, while the inner PE surface (that adhered to the minority-by-mass specialty layer) may have undissolved specialty layer with some solvent contamination present in it. Should residual specialty layer remain on the recovered film strips, this work recommends increasing residence time in heated solvent, to ensure that all targeted material may be dissolved prior to retrieval of delaminated solid film

4. Perspective and Future Directions on the Delamination of Plastic Films

Our work has successfully induced delamination in under an hour for four different film compositions. The recovered solid films are of high purity and recover all polyethylene initially present in the films. Solvent-based delamination can act as a separation technique for multilayer films, and enable the recycling of the materials within.

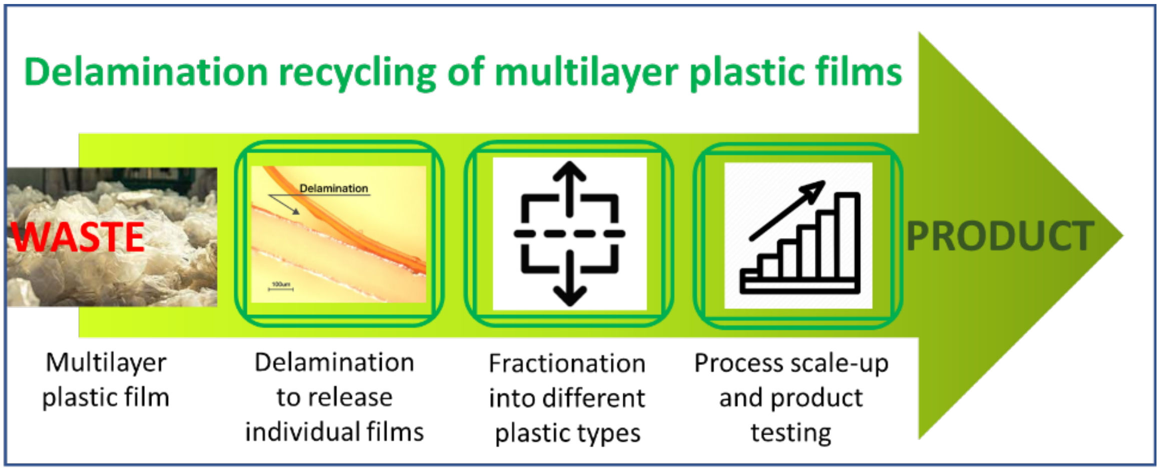
Delamination is a physical process for separating polymers for recycling. Since no antisolvents or precipitation steps are required, delamination recycling methods presented here are much less energy- and material-intensive than dissolution-precipitation recycling, where every polymer present in the multi-material film must first be dissolved and then precipitated, resulting in less solvent consumption overall, as the majority of the multilayer film remains solid throughout the process. Solid films recovered have no adverse effects from solvents used. This work has shown delamination to be complete in under an hour at temperatures under 90 °C, which is either on par or superior to previously published delamination times and temperatures. The delamination process is simple in design requiring no additional preparation of film other than size reduction.

Further work towards the precipitation of dissolved materials form solvent, the recycling of solvent for reuse in the delamination process, and a better understanding of the energy and materials cost of the process are required. Precipitating out dissolved materials allows for the recovery of all materials present and not just the desired polyethylene. Additionally, spent solvent should be reused for as many process cycles as possible, and the efficiency of recycled solvent in the delamination process should be documented.

5. Conclusions and Recommendations

Our team aims to develop and validate a novel recycling process for handling multilayer multi-material films that constitute a large fraction of all flexible plastic films which, in turn, are a major fraction of the 1 trillion pounds of plastic produced annually worldwide. The process developed here is based on the delamination of multilayer films which leaves intact, in the solid state, polyethylene which is the majority component of the film and main target for recovery and recycling. This solvent-based multilayer film delamination process is environmentally responsible on the basis of low energy usage (relatively mild temperatures and low solvent usage as the majority of the polymer remains solid) and low GHG emissions (no polymer chains break down). Polyolefins recovered using this process can meet demand by customers and corporations to incorporate recycled plastics into products.

Collaboration with industry stakeholders, policymakers, and waste management systems is crucial for practical application of the multilayer film delamination approach developed in this project. Adapting methods to meet industry standards and recognizing regulatory landscapes are vital for successful implementation. in industrial practices.



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Dr. Jacob Licht received a B.S. degree in Chemical Engineering from SUNY-Buffalo, and in 2025 completed his PhD on "Solvent-based Delamination of Multilayer Films for Plastics Recycling Applications" working with Professors Paschalis Alexandridis and Marina Tsianou in the Chemical and Biological Engineering Department at SUNY-Buffalo.

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Prof. Tsianou leads research that involves the design, development, and characterization of molecularly-engineered materials with desirable functionalities. She contributes to plastic recycling with expertise in nanostructured polymers in films and on surfaces, polymer dissolution, nano- and meso-scale organization and characterization.

Paschalis Alexandridis

Prof. Alexandridis' research utilizes molecular interactions and supramolecular assemblies to develop processes that are environment friendly and energy efficient, and products with desired properties and function. He is leading projects on chemical recycling, recycling of multilayer films, and recycling of textiles. He has authored over 200 articles and 6 US patents (Google Scholar h-index 86 and 27,500 citations). https://www.cbe.buffalo.edu/alexandridis

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