DISSOLUTION-PRECIPITATION RECYCLING OF POLYOLEFINS

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Abstract

Only a small fraction of the plastics produced are being recycled, with the great majority being landfilled or released into the environment. Mechanical recycling is currently used to recycle plastic, however, this method is efficient only for homogeneous and non-contaminated feedstock, and for easily identifiable objects such as bottles made of PET or HDPE. Polyolefins in the plastic waste stream can be processed via pyrolysis, the most common among chemical recycling processes. Pyrolysis, however, decomposes the polymers, resulting in undesirable greenhouse gas (GHG) emissions. Further, pyrolysis is not viewed as constituting recycling when its product, pyrolysis oil, is not converted into new polymers. Plastics recycling research in our group utilizes physical, solvent-based processes that do not break down the polymer chains. This constitutes true recycling, as the recovered polymer is the same as the starting material. Such molecular recycling processes leave the polymer chains intact, thus maintaining their embodied energy and emitting relatively little GHG. This paper addresses the mechanism of semicrystalline polyolefin dissolution as revealed through joint in-situ infrared spectroscopy experiments and diffusion kinetics modeling. The paper also highlights the application for polyolefin recovery of switchable hydrophilicity solvents (SHS) that can cycle between a form that dissolves the target polymer and a form that does not, hence facilitating closed-loop solvent cycling. The insights obtained from these studies facilitate the design of solvent systems and processing conditions for the molecular recycling of polyolefins via dissolution-precipitation. Dissolution-precipitation is an energy-efficient and environment-friendly recycling process that can recover specific polymer types from mixtures, blends, or multi-material films, and purify them from additives, without negatively affecting the properties of the original polymers.

Keywords: Polyolefins, advanced recycling, chemical recycling, solvent purification, circular economy, sustainability

1. Introduction and Motivation

Plastics are useful because their properties are modular and amenable to first principles design. We are approaching half a billion tons of plastic annual production worldwide, which is one trillion (!) pounds, 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]

Mechanical recycling is the most common method used in the United States, but it struggles to separate polymers that have similar physical properties, causing cross-contamination in the recycled stream. Additives present in plastics also remain in the recycled products because mechanical recycling is not efficient and advanced enough to remove them. As a result, recycled plastic often shows poor mechanical performance, leading to downcycled products rather than true closed-loop recycling. It is evident that: (i) plastics offer unique, often irreplaceable, properties and will continue to be used;[4] (ii) plastics are ubiquitous and their waste threatens the environment;[5] (iii) even if sorted, flexible films and multi-material plastics cannot be reprocessed and are being landfilled;[6, 7] and, (iv) value from such plastics can be recovered by chemical recycling such as pyrolysis, but this involves high energy demand and high greenhouse gas (GHG) emissions.[8] These basic premises underscore our research on the solvent-assisted molecular recycling of plastics with a focus on polyolefins and multi-material multilayer films.

Upcycling of sorted plastic can be achieved via solvent based processes through dissolution of select types of plastic in environmentally responsible solvents to recover desirable materials such as polyolefins, and separate them from additives or impurities.[9, 10] This enables the recovery of higher quality recycled materials which are suitable for reuse, thus creating a circular economy in plastic industry. The innovations advanced here will enable the recovery from post-industrial and post-consumer waste of useful plastics such as polyolefins, for which great demand exists for incorporation into new products with certain minimum recycled plastic content.

2. Review of Related Work

Plastic 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.[11] Recycling of plastic waste is needed. The following processes can be used to this end: mechanical recycling, pyrolysis, and dissolution-precipitation recycling.

2.1. Mechanical Recycling

Mechanical recycling is the prevalent method used for plastic waste (for a schematic, refer to Figure 1). The great majority of current mechanical recycling processes do not handle plastic films.[12] 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[13].

Despite these issues, there have been positive developments in the mechanical recycling of plastic films.[14] AMP Robotics is utilizing an AI-powered sorting system to properly sort films, which may be retrofitted onto existing processes.[15] Improvements to sorting technology are valuable to MRFs due to challenges posed by multilayer film waste.[16, 17] 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.[18] Should advancements in sortation and mechanical recycling continue, plastic films could eventually be a viable feedstock for MRFs.

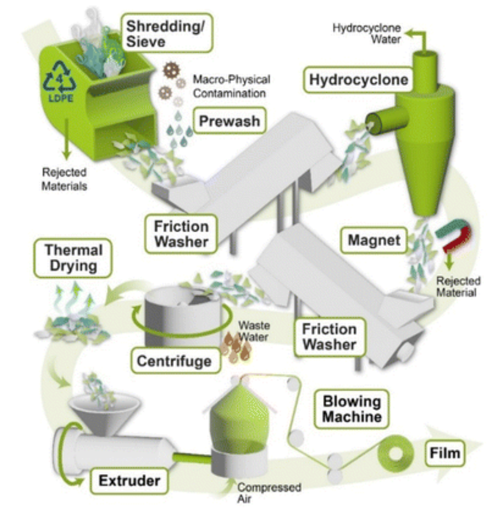


Figure 1: Schematic of mechanical recycling.

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 (for a schematic, refer to Figure 2).[19] For pyrolysis, the liquid products of diesel and naphtha are desirable for use as fuel and plastic production respectively.[20] 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.[20] The various hydrocarbons produced from pyrolysis need further processing to be turned in a desired liquid product.[20] 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 post-consumer waste plastic into fuel requires large processing volumes to be profitable, and the profitability is very sensitive to the (volatile) price of petroleum.[21]

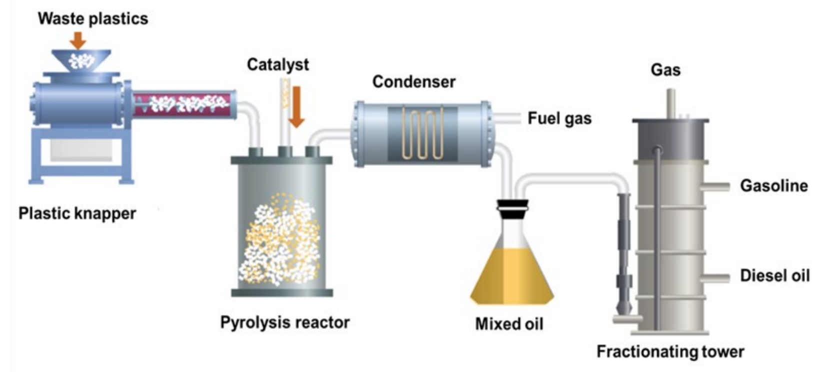


Figure 2: Schematic of pyrolysis process and products.

Pyrolysis can handle mixed plastic waste films, including contaminated feedstocks.[22] TotalEnergies has pyrolysis plants in Spain, France, and the US that can use flexible plastics as feedstock.[23, 24] 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.[23] Offering a complement or alternative to mechanical recycling is important, however generating controversial products and GHG, and requiring high energy consumption restrict how widespread pyrolysis of plastic film waste becomes.

2.3. Dissolution-Precipitation Recycling

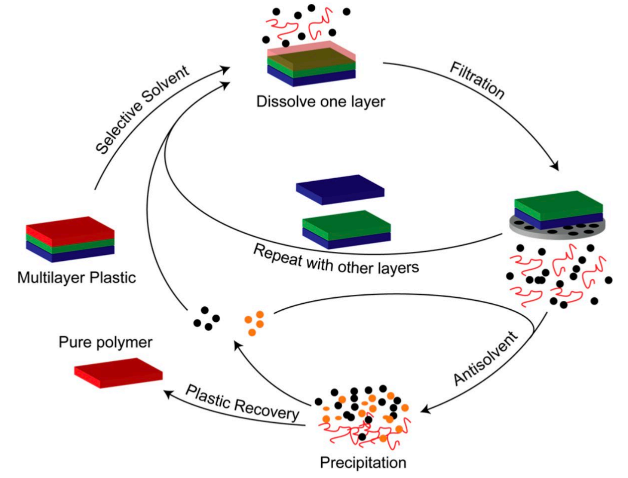


Fig. 3: Schematic of dissolution-precipitation process.

Dissolution-precipitation provides a means of recycling mixed plastic waste without the high energy consumption and emissions of pyrolysis. Dissolution-precipitation recycling works by dissolving each of the different polymers, one at a time.[25] Following filtration of undissolved solids, dissolved material is collected via precipitation (Figure 3).[26] Precipitation is generally performed using an antisolvent, cooling, or a combination of the two.[6] Solvent selection is based on solvent affinity solubility parameters such as Hansen Solubility Parameters. Solvent selection should be improved upon, as hazardous solvents like toluene are often used based on compatible solvent-solute affinity.[6] 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 uses specific solvents and antisolvents to dissolve all polymers present sequentially, layer by layer.[27]

The potential of dissolution–precipitation for recycling polyolefins is demonstrated by its adoption in industrial practice by, e.g., PureCycle Technologies that utilizes dissolution–precipitation to remove color, odor, and contaminants, and produce resin with properties comparable to virgin polypropylene (PP).[28]

3. Technology Approach

3.1 In-situ, Real-time Investigation of Polyolefin Decrystallization

Thin-film plastics, predominantly polyolefins including high-density polyethylene (HDPE), low-density polyethylene (LDPE), linear low-density polyethylene (LLDPE), and PP, pose significant recycling challenges at MRFs owing to their inherent flexibility, low density, and multilayer compositions, however, recent studies indicate economic viability through enhanced recycled product yields and pricing.[29-31] Achieving a closed-loop recycling system for such packaging films could be facilitated by solvent-based separation processes.[32, 33]

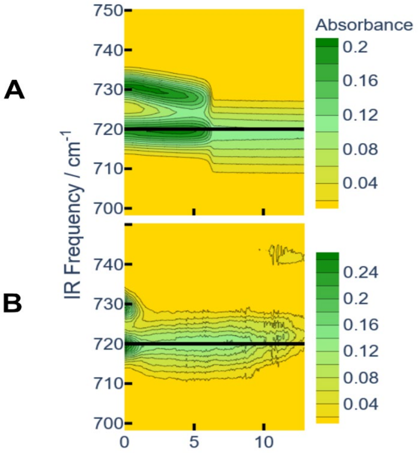


Figure 4: Two-dimensional contour plot of (A) HDPE melting and (B) HDPE dissolution at 106 °C in p-xylene.

Understanding the crystalline-amorphous boundary in semi-crystalline polyethylene (PE) during decrystallization is essential for advancing polymer science and enabling efficient plastic recycling pathways. This process involves the breakdown of ordered structures, influencing material properties and recyclability. We have explored PE thin films subjected to melting or dissolution through a specialized temperature-regulated liquid flow-cell system, capturing real-time molecular insights via in situ spectroscopy.[34] This research introduced the first real-time in situ mid-infrared (MIR, 4000–700 cm^{-1}) and near-infrared (NIR, 6000–4000 cm^{-1}) spectral analysis of PE dissolution. Integration of hetero-spectral two-dimensional correlation spectroscopy (2D-COS) (Figure 4) correlated fundamental vibrations, overtones, and combination bands to reveal chain disentanglement at a molecular scale.[34] This approach outperforms traditional methods by providing dynamic, multi-spectral data, enhancing models for dissolution-precipitation, membrane production, and foundational crystallization theories in macromolecules, areas long challenged by incomplete mechanistic understanding. Spectroscopic monitoring uncovered distinct decrystallization pathways: melting disrupts van der Waals forces primarily through thermal energy, while dissolution involves solvent-induced solvation shifts, evident in temporal changes to peak intensities and frequencies.[34] Key correlations linked conformational bands with CH_2 rocking modes, combination vibrations, and terminal CH_3 groups, refining parametric simulations. The study also validated and revised conventional MIR/NIR assignments, uncovering novel IR-active modes via 2D-COS, offering fresh tools for polymer characterization and sustainable material design. [34]

3.2 Polyolefin Dissolution Kinetics Revealed by Integrated Experiments and Modeling

Despite polyolefins being the most widely produced polymers and the feedstock for industrial-scale dissolution-precipitation recycling plants, surprisingly little is known about their dissolution. To address this gap in knowledge, we developed a model that describes the different phenomena involved in the dissolution of semicrystalline polyolefins, and validated the model with experimental results on the decrystallization and dissolution kinetics of HDPE films and PP pellets.[35] According to this model, the dissolution of semicrystalline polyolefins involves diffusion of solvent into the solid material, solvent-induced decrystallization of crystalline polymer domains, swelling with solvent of amorphous polymer domains, disentanglement of polymer chains from the solid material, and subsequent diffusion of polymer chains into the solution. Along the way, the polyolefin films or particles initially swell in size, and then shrink, until they completely dissolve (refer to Figure 5).[35]

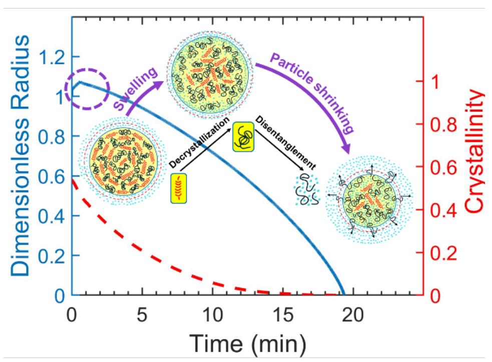


Figure 5: Time evolution of dissolving PP pellet radius and degree of crystallinity.

The model provides detailed time-resolved and position-resolved information on composition (i.e., crystalline polymer domains, amorphous polymer domains, and solvent) and solvent diffusivity (which depends on composition) across the dissolving polymer film or pellet, in different solvents and temperatures. Such information is unavailable experimentally or difficult to obtain.[35] The key fitted parameters that capture decrystallization and polymer chain disentanglement decrease with increasing temperature following an Arrhenius relationship, with activation energies higher than that for crystallization and comparable to that for melt viscosity. Both decrystallization and dissolution times increase with film thickness or particle size. For smaller particles, decrystallization and dissolution occur nearly simultaneously, while, for larger particles, their interior remains solvent-poor and crystalline for longer times.[35] This work offers insights into the interplay of decrystallization and polymer chain disentanglement during the time-course of semicrystalline polyolefin dissolution. Further, this work facilitates the design and optimization of dissolution–precipitation recycling processes that can unlock value from the million tons annually of polyolefins that are currently being landfilled or incinerated following their use.[35]

3.3 Dissolution-Precipitation Process for Plastics Recycling

The dissolution–precipitation process is a promising solvent-based technique for plastic recycling, as it is a physical separation method that preserves the polymer's chemical structure, making it a true form of recycling. In this process (Figure 6), the target polymer is selectively dissolved in a suitable solvent, while undesired polymers, additives, and impurities remain undissolved, which are removed through filtration or centrifugation. The dissolved polymer is then recovered by adding an antisolvent, which is miscible with the solvent but in which the polymer is insoluble, forcing the polymer to precipitate out and enabling its recovery and reuse.[36]

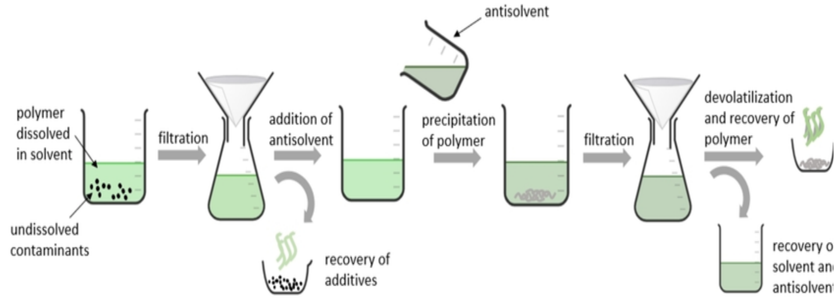


Figure 6: Dissolution-Precipitation Process.

Although dissolution–precipitation offers high polymer recovery and near virgin quality polymer, most reported systems rely on hazardous solvents and require an additional antisolvent to precipitate the polymer. Using antisolvents introduces extra steps for solvent separation, which are typically carried out through energy-intensive operations such as distillation, leading to large energy use and associated CO_2 emissions.[37, 38] Recent studies have addressed this limitation by using switchable hydrophilicity solvents (SHS), which can switch reversibly between a state capable of dissolving the polymer and another that causes the polymer to precipitate after switching SHS hydrophilicity.[39, 40] By changing their properties on demand, SHS eliminates the need for antisolvents and reduces dependence on energy-intensive separation processes, making solvent-based plastic recycling more sustainable.

4. Perspective on Dissolution-Precipitation Molecular Separation of Plastics

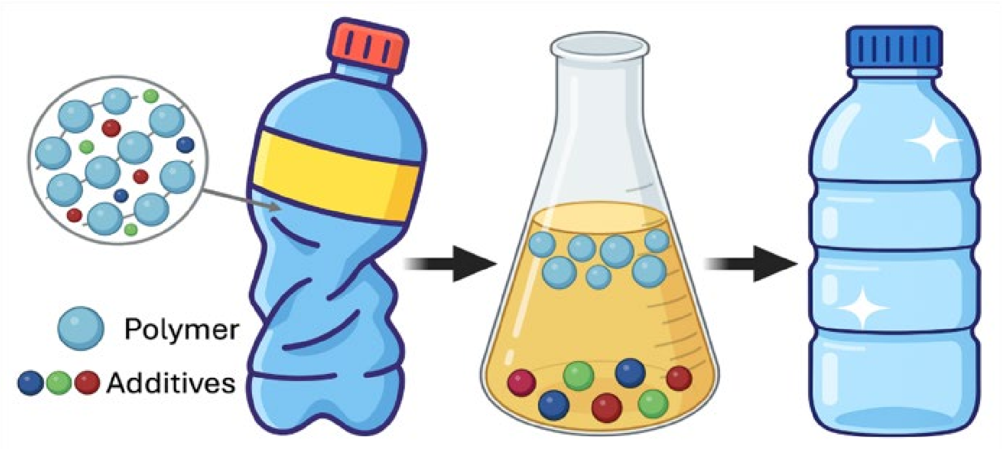
Solvent-based recycling processes based on physical phenomena, such as dissolution–precipitation, offer a more environmentally friendly approach to plastics recycling compared to pyrolysis chemical recycling.[41, 42] Switching from incineration to dissolution-precipitation recycling can significantly reduce CO_2 emissions by up to 75 %.[43] Life cycle assessment (LCA) findings highlight favorable results for dissolution-precipitation employing the SHS process that utilizes CO_2 to switch between a solvent form that dissolves polyolefins and a form that does not, and thus allows polymer dissolution and precipitation to be accomplished in the same solvent with no need for antisolvent and the associated solvent separation steps and expenses.[40] This study emphasized the significance of integrating green chemistry principles at the initial design phase. This involves implementing strategies to minimize waste production, like utilizing recyclable solvents, and enhancing the overall efficiency of the process.[40]

In our research we develop an efficient dissolution-precipitation recycling process targeted to polyolefins such as LDPE, HDPE, and PP. We investigate solvents like amine-based SHS, non-amine-based SHS, and non-SHS and address challenges related to incomplete polymer precipitation. Successful implementation could elevate recycling rates, contribute to a circular plastic economy, and pave the way for recycling diverse polymer types while mitigating environmental and health risks associated with hazardous additives.

5. Conclusions and Recommendations

Polyolefins comprise the largest volume of commodity plastics, but they are difficult to recycle to valuable products due to the molecular-level incorporation of other plastics, additives, colorants, and/or fillers. The solvent-based polyolefin separation methodology that we pursue is environmentally responsible on the basis of the solvents selected and the greatly reduced CO_2 emissions, and can be readily implemented in industrial practice and extended to the separation of other types of plastics. The different types of polyolefins to be recovered from the solvent separation process can meet the pressing demand by both customers and corporations to incorporate recycled plastics into products while maintaining desirable specifications.

Beyond the important step of technology development, collaboration with industry stakeholders, policymakers, and waste management systems is crucial for practical application of any recycling technology. Adapting methods to meet industry standards and recognizing regulatory landscapes are vital for successful implementation. A multidisciplinary approach, integrating technological innovation, economic feasibility, and collaboration, is essential to transition research from the laboratory to industrial practices, making a tangible impact on plastic recycling.



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Ms. Shikha Solanki is a PhD student in the Chemical and Biological Engineering Department at SUNY-Buffalo advised by Professors Alexandridis and Tsianou. Shikha is investigating the dissolution-precipitation of polyolefins in green solvents for molecular recycling. She has a Masters degree in Chemical Engineering from the Indian Institute of Technology (IIT), Guwahati, India.

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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.

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