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Laboratory Experiment

Hands-on Laboratory Experiments for Demonstrating Mixed Plastic Recycling

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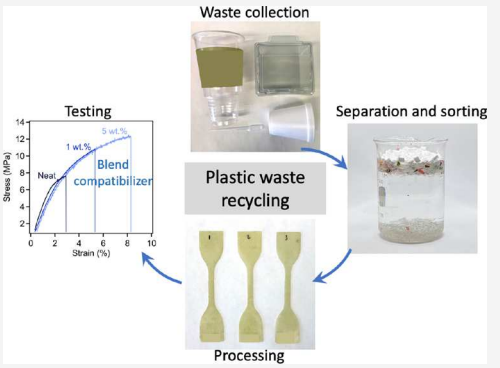
ABSTRACT: Plastic waste is a growing concern in recent years due to the massive increase in single-use plastic applications. Despite the ever-increasing amount of waste, recycling plastics is not a simple process due to difficulties in sorting, which results in significantly downgraded material properties. This hands-on lab experiment showcases the important knowledge steps in a typical recycling process, including waste collection, plastic separation and sorting methods, processing techniques, and characterization methods including scanning electron microscopy (SEM) and tensile testing. Additionally, this lab demonstrates the importance of miscibility between materials and provides evidence that small amounts of block copolymers (BCPs) can compatibilize immiscible mixed plastic waste, leading to enhanced material properties of recycled products. Through these engaging hands-on lessons, students can not only obtain a better understanding about the challenge and industrial solution of mixed plastic waste recycling, but also realize the importance for protecting environments through waste management.



KEYWORDS: *Upper-Division Undergraduate/General, Demonstrations, Hands-on Learning, Materials Science*

INTRODUCTION

The ever-increasing amount of plastic waste imposes significant stress on the sustainable development of our environment and society. Over the past few years, strong government initiatives and policies have been implemented across the globe to fight against plastic waste, including establishing a circular plastics economy, banning the use of plastic bags, and extensive investments in developing the technology for plastic recycling. In 2021, over 500 million tons (MMT) of plastic were consumed, and less than 10% was successfully recycled.[1,2] The vast majority of plastic was incinerated, landfilled, and/or leaked into the environment, resulting in adverse impacts on human health, climate, and marine ecosystems.[3,4] To directly combat these challenges, the need for recycling and efficient reuse of plastic waste becomes ever more apparent, which has now become an integrated part of promoting a circular economy in our society. To demonstrate the important concept and societal and technical needs of plastic recycling to students, several excellent works have been published focusing on the topics of plastic recycling and waste management. For example, Donahue et al. demonstrated the synthesis of dibenzyl terephthalate to undergraduate students,[5] which can be used for chemical recycling of plastic bottles. Leri et al. performed analysis of plastic waste for sorting in recycling plants, using an infrared spectroscopy-based method in an organic chemistry lab.[6] Furthermore, McNeil et al. reported a critical short course on the topic of sustainable polymers for high school students.[7] These examples demonstrate the collective efforts in academia for teaching future generations about many important aspects of plastic recycling.

There are two major approaches for recycling plastic materials, each having different advantages and limitations. Mechanical recycling is the most established and commonly used method,[8] which directly reprocesses and/or re-engineers plastic waste materials into new products while maintaining a constant chemical identity throughout the process. This method has advantages of low energy consumption, wide availability of equipment, and broad applicability for different plastic types. However, it requires careful materials sorting prior to them entering the waste stream, and the recycled products can exhibit downgraded mechanical properties due to possible chain degradation, branching, or cross-linking from thermal conduction and a high shear environment. Alternatively, chemical recycling can convert plastic materials to a monomer state,[9,10] fuels,[11,12] carbon,[13,14] and/or other chemicals via processes including hydrolysis, pyrolysis, and

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glycolysis. While these approaches may be more energy intensive than mechanical recycling, this method can yield new materials with identical or improved properties as virgin plastics. However, chemical recycling processes are challenging to scale up, requiring system optimization and technoeconomic analysis.

Sorting/separation of different plastics from waste stream is a key step in the recycling process. For example, sink−float sorting is a very common sorting method. Through submerging the cleaned and granulated plastics in a liquid, typically water, the float fraction can be separated from the sink fraction due to their different material densities. When water is used as the reference liquid, the float fraction is typically composed of different polyolefins (POs) and foamed (or porous) materials, while the sink fraction is the rest of the plastics which have a higher density than water, such as polyethylene terephthalate (PET). While there are a wide variety of ways to collect and sort recycled plastics (including Fourier transfer-near-infrared (FT-NIR) spectroscopy, optical color recognition, wind sifting, melt filtration, and manual sorting), they can often be expensive, time-consuming, labor intensive, and can be inaccurate when separating materials with similar properties and/or chemical compositions. As a result, even after multiple steps of separation process, the waste can still be composed of different plastic types. For example, polyethylene (PE) and polypropylene (PP) are difficult to separate through conventional methods due to their very similar densities as well as other physical properties. Additionally, many practical uses of plastics require the addition of fillers and compatibilizers for adjusting their processability and/or materials performance. The introduction of additional components would greatly influence the material density, imparting further challenges in efficient plastic separation based on sink−float sorting methods. One major challenge associated with mixed plastic waste is that polymer blends can be thermodynamically immiscible, especially commodity plastics which often have high molecular weights (e.g., 50,000−1,000,000 g/mol).[15,16] The immiscibility between the different components in mixed plastic waste creates significant phase separation, leading to very limited mechanical properties due to poor adhesion of domain interfaces.

Since complete separation of the mixed plastic waste seems impractical, introduction of blend compatibilizers and fillers have become common routes for increasing blend miscibility and ultimately improving the properties of recycled materials from plastic waste. Specifically, blend compatibilizers can promote miscibility of different plastics by reducing the interfacial tension and stress across the polymer−polymer interface, which leads to stabilization of blend interfaces and enhanced adhesion.[17] This approach is considered to be cost- and energy-efficient to recycle mixed plastic waste since it is based on mechanical recycling methods. However, the introduction of blend compatibilizers can lead to increased complexity in the waste stream. One proposed concept for addressing this challenge is to combine the mechanical and chemical recycling methods, in which after multiple reprocessing cycles, materials can be pyrolyzed or depolymerized to monomer states.

The most widely used blend compatibilizers are block and graft copolymer additives,[18] which can favorably interact with distinct components in the mixed plastic waste through hydrogen bonding and/or $\pi-\pi$ interactions. These additives can be primarily localized in the interfacial regions of the blends for stabilizing the resulting morphology and reducing interfacial tension, which can lead to improved physical properties of the blend. Furthermore, the properties of recycled polymer blends are also dependent on the concentration and chemical structures of blend compatibilizer additives. To demonstrate the application of blend compatibilizers in mixed plastic waste recycling and their impact on final product properties, in this lab demonstration we used polystyrene-*block*-poly(ethylene-*ran*-butylene)-*block* polystyrene (SEBS) as a block copolymer (BCP) additive for recycling mixed plastic waste, which includes polystyrene (PS), polyethylene (PE), polypropylene (PP), and PET (also see Supporting Information for details). From this lab experiment, it is expected that students will have a deeper understanding about (1) plastic waste recycling challenges, (2) the critical need of plastic sorting and separation, (3) an industrial approach to address mixed plastic waste, and (4) the impact of morphology on materials performance, while harnessing their ability to perform mechanical property tests and data analysis, which is broadly applicable to the field of materials science and engineering. Additionally, these lab experiments can be video recorded, allowing their potential further and broader use as a part of virtual lab components for institutions who may have limited resources.

LEARNING OBJECTIVES

1. Identify the critical needs of plastic recycling for the sustainable development of the environment and society.
2. Describe the conventional process for recycling mixed waste plastics and identify the principles and limitations of density-based sorting methods.
3. Describe the operation principles of conventional polymer processing techniques on a lab scale, including compounding/extrusion and compression molding.
4. Obtain the ability to perform tensile tests to measure and analyze the mechanical properties of plastic materials.
5. Identify the impact of BCPs on polymer miscibility and characterize the mechanical properties of mixed plastics.

EXPERIMENTAL OVERVIEW

Plastic Sorting

Sink/Float Test. Waste plastic, including PE, PP, PS in the form of Styrofoam, and PET, from various sources were collected and then cleaned to remove any residual food or adhesive. Prior to cutting the waste plastic, each material was weighed out in order to determine their relative amount (see Table 1).

Plastic waste was cut into small granules of similar sizes (<2 mm in length). A beaker filled with tap water was employed for the separation of plastic. After adding the plastic pieces into the filled beaker and mixing, the materials self-sorted due to their

Table 1. Mass of Each Starting Waste Plastic, Prior to Cutting

material	mass (g)
PE	25.5
PP	33.5
PS	6.7
PET	27.4

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density differences compared with water. The floating plastic was skimmed from the water and set aside and vacuum-dried overnight for the rest of the demonstration. The plastic which sank to the bottom was collected and dried for further use.

Sample Preparation

Compounding/Extruding. The granulated, sorted, and dried plastic from the float fraction was melt processed together via a microcompounder. Prior to compounding, the melting and degradation temperatures were determined for each of the plastics to find an appropriate processing window for the blends. These materials were fed into the compounder (Xplore MC5) set to 200 °C with a screw speed of 80 rpm. After mixing for 5 min, the materials were extruded and collected. After compounding the neat mixed waste plastics, two other batches were compounded under the same processing parameters with the addition of SEBS (see Figure 1), at 1 and 5 wt % loading content, which can act as a blend compatibilizer.

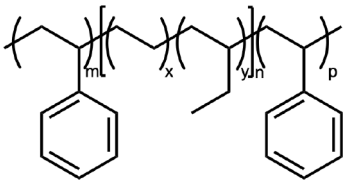

Figure 1. Chemical structure of SEBS.

Compression Molding. After the compounding process, the extruded samples were converted into tensile bars for subsequent mechanical testing. This was accomplished by pressing the compounded material into a tensile bar mold under high compressive forces at an elevated temperature. Each tensile bar was molded at 210 °C under 7 tons. After compression molding, the freshly made samples were removed from the molds and labeled.

Characterization

Scanning electron microscopy (SEM) was used to characterize the morphology of different mixed plastic waste samples after compounding, which was performed by teaching assistants who are graduate students. The backscatter detector (BSD) with an energy of 20 kV was used to acquire images. Tensile testing and analysis were performed by participating undergraduate students, using an MTS Insight frame with a 1.5 kN load cell at 1 mm/min following a modified ASTM D638 standard. Data analysis was performed using Igor Pro 9, where the ultimate tensile strength (UTS) was the maximum tensile stress achieved before fracture, Young's modulus was calculated as the slope of the linear region of the curve prior to yield, and the toughness was the area under the curves, determined by integrating the stress−strain curves for each sample.

SAFETY HAZARDS

Personal protective equipment must be worn during these experiments, including safety glasses, a lab coat, gloves, and closed toed shoes. During melt processing, including compounding and compression molding, extra attention should be taken due to elevated temperatures and high compressive forces; as such, heat gloves should also be worn.

RESULTS AND DISCUSSION

Plastic Sorting

Plastic waste for this lab demonstration was collected from different products after their use, including disposal cups and pipettes (Figure 2a,b). After washing with water for several times, these materials were cut into small pieces for performing sink−float separation experiments (Figure S1). This demonstration helps students gain an understanding of different types of plastics used for different applications and their prevalence in daily life. The hands-on process of cutting the plastics and sink−float sorting of the materials provided students with a better understanding about waste granulation and sorting processes, which are commonly employed in plastic recycling industry.

Sink−float sorting relies on density differences compared with a standard solution. In this case tap water was used as the standard with the plastic granules separating into two fractions: (1) the sink fraction, materials which have density greater than water, and (2) the float fraction, materials which have density less than water. It was determined that the sink fraction was made up of PET, while the float fraction contained PS foam, PE, and PP. Notably, while bulk PS density is higher than water, their use as the foam materials leads to a significantly reduced material density, enabling them to float on the water surface. Therefore, it is important to note that the complete separation of mixed plastic waste is very challenging, due to the presence of additional components which can alter the material density. While this test successfully separated the PET from the rest of the materials, the float fraction contained commingled PS, PE, and PP, which further illustrated the difficulties of separating many similar plastics.

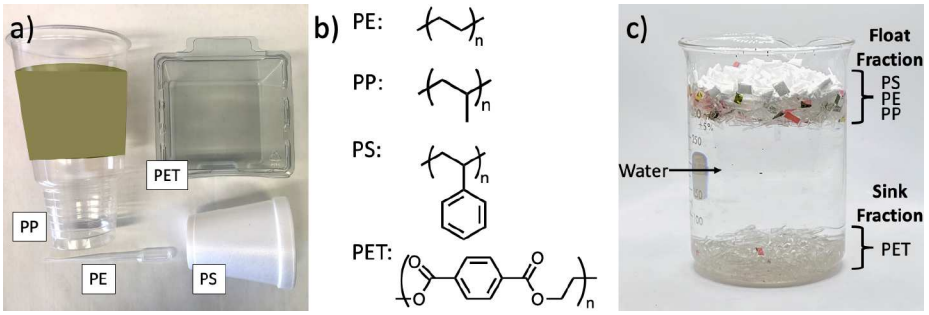

Figure 2. (a) Starting plastic waste materials for experiment, including PP, PE, PET, and PS, and (b) their corresponding chemical structures. (c) Sink−float test performed on chopped up materials from (a), where PET made up the sink fraction, and PS, PE, and PP were the float fraction.

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Sample Preparation

After sorting and separation, dried materials from the float fraction were reprocessed into a new product. Through this process, students are able to get hands-on experience with melt processing techniques including (micro)compounding and compression molding. After determining an appropriate processing temperature for the mixed plastics, which is approximately 200 °C for this system, the plastics should be loaded into the compounder and mixed for approximately 5 min prior to extrusion. In order to determine how the addition of BCPs impact the material properties of mixed plastic waste, SEBS was added in small amounts, 1 and 5 wt % (relative to the total mass), in following compounding batches. This provided the students practice in mass percent calculations and measuring in smaller scales than they may be used to. Due to contaminated dyes in the PP used for this demonstration, the resulting extrudate of the blended materials exhibited a light yellow-green (Figure 3 and S2). This color change provided additional insight into other reasons that mixed plastic is not recycled, in addition to potential poor physical properties.

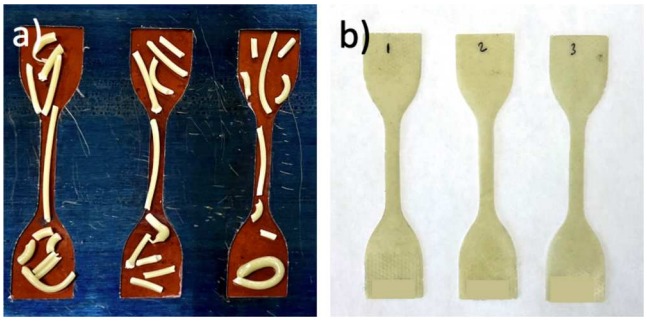

Figure 3. (a) Tensile bar mold with extrudate of neat PS/PE/PP blend from the compounder prior to melt pressing, and (b) three neat PS/PE/PP blends were melt pressed and removed from the mold for mechanical tests.

In order to characterize the mechanical properties of the blends, samples with a dog bone shape were prepared through a melt press (see Figure 3). The extrudate was carefully cut and placed into the molds, making sure there was enough material to fully fill the molds, then pressed with 7 tons at 210 °C for approximately 15 min before being cooled on the benchtop for 15 min. Alternatively, solution blending and drying can be used to prepare mixed plastic waste samples, for which xylene is a known good solvent for dissolving polyolefin and polystyrene materials. After solvent evaporation, the plastic blends can be cut into desired shapes/geometries for following characterization tests.

Morphology Characterization and Mechanical Testing

To determine how the addition of BCPs impact the microstructures of mixed plastic waste, we performed SEM imaging on different mixed plastic waste samples with and without the inclusion of the BCP additive (SEBS). Tensile testing was also performed to determine the ultimate strength, elastic modulus, and toughness of the reprocessed materials.

The SEM images showed a significant decrease in PS droplet size through the addition of SEBS acting as a blend compatibilizer. As shown in Figure 4(a), the PS domain in the mixed plastic exhibits spherical morphology, with an averaged diameter in the range of 7−8 μm. Upon the inclusion of SEBS at 1 wt %, the domain size of PS components

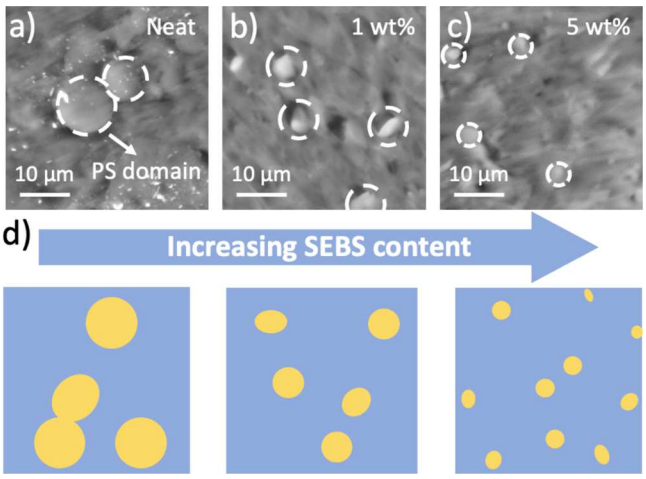

Figure 4. (a−c) SEM images of mixed plastic waste without and with the inclusion of SEBS. (d) A schematic illustration showing that as the SEBS content increases, the domain size of PS is anticipated to decrease due to improved miscibility.

significantly reduced to approximately 2 μm (Figure 4(b)), suggesting the improved miscibility between PS and polyolefin components (including PE and PP). This result confirms that even a very small amount of blend compatibilizers can make significant differences in the morphology of resulting mixed plastics.[19] Further increasing SEBS content from 1 to 5 wt % leads to smaller PS domains (Figure 4(c)), showing that there is decreased phase separation in the blends and the polymer−polymer interfaces have less interfacial stress with the added BCP (Figure 4(d)). The domain size of phase-separated plastic waste, which is in the range of 2−10 μm, is feasible to be characterized using a conventional optical microscope, which provides an alternative method when SEM is not available/accessible for students and institutions.

Imaging the samples via SEM provided the students with a clear visual of phase separation between different plastic types and how inclusion of small amounts of BCPs can have a large impact on the sizes of phase separated domains, especially when students know that the volume fraction of each polymer being compounded together did not change significantly throughout the process.

Finally, tensile tests were performed on each sample, and the Young's modulus, UTS, and toughness were calculated. Students were taught about the basic concepts of tensile tests and relevant stress−strain curves, how to load samples into the grips, as well as how to use the software starting and stopping the test. Additionally, a recent article provides great details about mechanical testing, which can be combined with virtual experiments and used a reference for student learning background knowledge.[20]

As shown in the stress−strain plot for different samples (Figure 5a), both the maximum stress and strain achieved for each sample increased with increasing SEBS content, suggesting improved mechanical properties of mixed plastic waste through the addition of blend compatibilizers.[21] Additionally, while the modulus was similar for all three loading contents, the neat blend modulus was equal to 4.2 MPa, while 5 wt % SEBS had a lower modulus of 3.4 MPa (Figure 5b, all error bars are calculated from three measurements). This decrease in modulus directly translates a slightly stiffer blend without SEBS. While the modulus was similar

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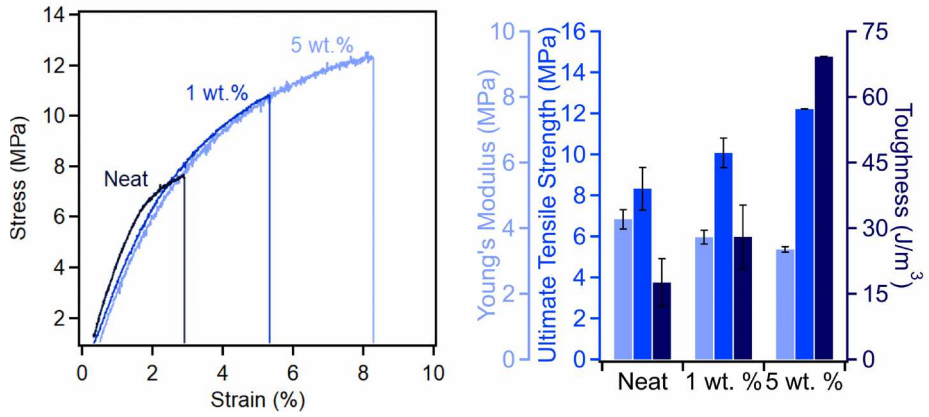

Figure 5. (a) Stress−strain curves for different mixed plastic waste after recycling, including neat, and with 1 and 5 wt % SEBS, and (b) their respective Young's modulus, UTS, and toughness.

between each sample, the UTS was nearly doubled between samples with and without SEBS, increasing from 7.4 to 10.6 MPa and 12.2 MPa for 1 and 5 wt % loading content, respectively. Furthermore, the toughness of recycled products also increased dramatically from 14.2 Jm^{-3} for the samples without SEBS to 69.2 Jm^{-3} with 5 wt % SEBS content. These mechanical testing results clearly showed that the addition of a blend compatibilizer, in this case using SEBS as an example, can substantially increase the UTS and toughness of recycled plastics, which enable the enhanced materials performance for their reuse in many practical applications, such as packaging, filament, and fibers.[22−24] Overall, this lab experiment, used as a REU project and demonstrated in a polymer mechanics course, provides an excellent opportunity for students to obtain hands-on training experiences for learning mechanical testing, while obtaining knowledge about plastic recycling and the need of plastic separation, and how to address mixed plastic waste recycling using blend compatibilizers. Using lab reports, we have comprehensively assessed student learning outcomes on all objectives. The details about specific measures, student performance, and findings are included in the Supporting Information. To provide a brief summary, we found that through this hands-on lab activity, students can describe the operation principles of instruments (compounding and compression molding), identify challenges in using density-based sorting methods, explain the impact of blend compatibilizers on mechanical properties of mixed plastic waste, as well as quantitatively analyze mechanical properties. We found two areas where an opportunity exists for additional efforts in future lab/lecture classes. First, it is important for instructors to emphasize the opportunity of reducing greenhouse gas emission and energy consumption for plastic industry through recycling. Second, the instructor should explain the fundamental mechanisms that are responsible for reducing the size of macrophase separation domains using blend compatibilizers (hydrophobic−hydrophobic associations), which is associated with polymer thermodynamics.[25] It is anticipated that this lab experiment can be employed for materials science related courses as well as within the polymer/plastics science and engineering undergraduate programs. These classes may involve a large student population, which would require instructors to divide them into small groups (e.g., 5−8 students per group) for performing lab activities. In such case, opportunities may exist to allow students to teamwork and practice their statistical analysis skills through analyzing a large data set of mechanical properties.

CONCLUSIONS

This work shows a hands-on lab experiment aiming to demonstrate to students a common recycling process for mixed plastic waste, including separation, processing, and mechanical testing. We also provided examples showing the application of blend compatibilizers to improve the mechanical properties of recycled products. Students collect, cut-up, and sort single-use plastics. These plastic wastes are compounded with varying amounts of BCPs and compression molded. Samples are then imaged via SEM to characterize their microstructures, followed by tensile tests where students then calculate mechanical properties including UTS, elastic modulus, and toughness of each sample. Through these interactive lab activities, students are actively involved in each step and able to learn about the entire recycling process in addition to gaining hands on experience with many instruments in plastic industry. These skills are transferrable to many other applications. Furthermore, by discussing methods for the recycling process, this experiment makes students aware of plastic recycling and its significance in society.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.2c00702.

Notes for instructors; Learning outcome assessment plans; Images of mixed plastic waste and after extrusion; SEM images of mixed plastic waste after reprocessing (PDF) (DOCX)

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Notes

The authors declare no competing financial interest.

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