



Article Solid-State Shear Milling for Recycling Aluminum–Plastic Packaging Waste: A Sustainable Solution for Mixed Plastic Waste

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Abstract: The application of paper-aluminum-plastic packaging has been widely adopted in various fields such as the food and medical industries, owing to its exceptional preservation and obstruction properties. Nonetheless, the recycling process for paper and aluminum from this packaging type typically involves water pulping and solvent separation. The resulting residual waste, commonly known as multi-plastic waste (PMW), poses significant challenges in terms of separation and recycling. In this research article, we propose a solution for the recycling of PMW using solid-state shear milling (S³M). This process utilizes powerful three-dimensional shear force to achieve pulverization and excellent dispersion of multicomponent polymers, all while maintaining ambient temperature conditions. The thermoplastic processability of milled PMW powder was improved. The results indicate that a significant reduction in the the average particle size of PMW from 700 µm to 226 µm after 10 milling cycles, as evidenced by both a particle size analyzer and SEM. Furthermore, S³M processing leads to a good dispersion of PMW domains, as confirmed by the reduction in domain size from 9.64 µm to 2.65 µm. DSC and DMA reveal excellent compatibility between the components of the composite, resulting in improved mechanical properties such as tensile stress (from 14.03 MPa to 22.02 MPa) and unnotched impact strength (from 3.26 KJ/m² to 4.82 KJ/m²). The findings suggest that S³M technology could be an effective and sustainable method for recycling PMW without any separation process, with promising industrial application.

Keywords: solid-state shear milling; aluminum-plastic packaging waste; mixed plastic waste; recycling

1. Introduction

Aluminum–plastic packaging waste (APPW) is a type of multi-layered composite material, such as Tetra Pak, which comprises around 75% kraft paper, 20% low-density polyethylene (LDPE), and 5% aluminum (Al) foil [1]. The utilization of this material has greatly increased due to the barrier property of Al foil, the flexibility of plastic film, and the folding and impact resistance of paper. Such paper–aluminum–plastic is widely used for liquid packaging, as it significantly prolongs the shelf life of perishable liquid foods, including milk and fruit juice. Despite the increasing global output of Tetra Pak, which was estimated at 190 billion bags, its recovery rate is merely 26%. Most abandoned Tetra Pak is either burned or dumped in landfills, leading to secondary environmental pollution and resource wastage in numerous countries [2]. Therefore, the development of recycling technology is essential to address the mounting environmental concerns associated with the disposal of APPW. The paper component of paper–aluminum–plastic can be recycled through hydro-mechanical pulping [3], whereas recycling the remaining aluminum–plastic is challenging. Various methods have been employed for



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). aluminum-plastic separation, such as solvent separation, dry separation, and plasma separation [4,5]. Diop et al. [3] utilized hydro-mechanical pulping and chemical treatment to recover individual components, including cellulose fibers, polyethylene, and Al, from recycled laminated packaging. The composite obtained by combining the recycled plastic and isolated microcrystalline cellulose exhibited improved tensile strength and modulus. Zhang et al. [6] studied wet separation conditions for a laminated sheet containing LDPE and Al foil. Hidalgo-Salazar et al. [7] analyzed the creep response and mechanical properties of composites made from fique mats and LDPE-Al separated from recycled long-life Tetra Pak. These studies focused on the separated components of Al, cellulose, and plastic from recycled APPW, while some other research has also investigated pyrolysis recovery. Korkmaz et al. [8] conducted a study on the pyrolysis of Tetra Pak waste in the temperature range of 400-600 °C. This process produced gas and wax, along with carbon residue and pure Al. Yin et al. [9] investigated the thermal transformations of metallized plastic waste into valuable $Al/Al_4C_3/AlN$ resources and valuable gaseous fuel. Generally, the recycling of APPW has been focused on the recovery of high-value Al and cellulose [10–12]. However, there is limited research available on the recycling of residual multi-plastic waste (PMW). PMW is a complex mixture of various plastic materials, differing in chemical structures, densities, and melting points, which pose a significant challenge for recycling. Moreover, PMW may also contain other types of waste, including sawdust and sand, further complicating the recycling process. Due to the high economic cost and the complicated nature of the process, there is no efficient industrial technology available for effectively recycling mixed plastic waste, resulting in a negative impact on the environment, such as landfill capacity overload and air pollution caused by incineration [13,14]. Therefore, it is of utmost importance to develop a cost-effective and viable recovery method for recycling PMW.

The high economic cost and the complexity associated with recycling residual PMW have resulted in a lack of available industrial technology for its effective recycling. In recent years, micronization technology has been used for the recycling of polyurethane, ethylene–propylene–diene monomer, and polyamide [15–17]. In this current study, PMW from aluminum–plastic packaging waste was recycled using a novel and eco-friendly pulverizing instrument called solid-state shear milling (S³M) technology. S³M technology was developed by our laboratory based on the traditional Chinese stone-mill [18]. Our study investigated the particle size distribution, processability, and mechanical properties of PMW during the S³M process, and the results indicate that this method provides a simple and effective means for recycling PMW with potential for practical application.

2. Materials and Methods

2.1. Materials

In this study, post-consumer multi-plastic waste (PMW) was collected from XiaMen-ShenhaiNewmaterial Co. Ltd., China, and separated in factories. Most of the valuable Al and paper had been recycled, leaving behind the plastic and its impurities, which are difficult to separate and recycle.

2.2. Preparation of Samples

Figure 1a shows a schematic illustration of the practical processing of PMW. Before the preparation of the powder, PMW was first crushed to millimeter scale by a powerful plastic shredder (Taizhou HuangyanJiacheng plastic machinery Co., Ltd., China), and then the crushed sample was further pulverized by the S3M. Figure 1b shows the schematic drawing of the self-designed pan-mill-type equipment. The left part of Figure 1b shows the inlaid pans of the equipment, and its main structural parameters are radius *R*, the division number of milling pan *n*, their corresponding slot number *m*, slot-top width δ , and bevel angle α . Two pans with similar structure are placed in opposite directions, and one of the pans is stationary while the other is movable. In one milling cycle, the angle between two pans gradually decreases to 0, so that the material between them is crushed by the strong extrusion and shearing. This shear force is three-dimensional. During sample preparation,

the crushed sample was uniformly fed into the feeding funnel of this equipment. The material was milled along the spiral track in the milling disc and discharged from the edge of the milling disc. The milling powder was collected from the discharging port and recorded as finishing one cycle of milling. The equipment was running at ambient temperature with cooling water, and the speed of the milling disc was controlled at 30 r/min. The whole milling process is carried out under dry conditions. The materials could be milled repeatedly by continuous operation. After 10 cycles of milling, the powder was extruded into filaments by a Hapro rheometer at 190 °C (RM-200C, HarbinHapro electric technology Co., Ltd., China) and then formed into granular shape by a Plastic Grain Cutting Machine (KQCEC of Chenguang Research Institute of Chemical Industry Co., Ltd., China).



Figure 1. Schematic illustration of preparation of samples (a) and the structure of pan-mill reactor (b).

2.3. Characterization

Particle size after grinding was determined by a particle size analyzer (S3500-SI, Microtrac Instrument Co., Ltd., USA). A small number of milled particles was dispersed in deionized (DI) water, and the particle size was measured after ultrasonic dispersion. Fourier-transform infrared spectroscopy (FTIR) was obtained on a Nicolet 6700 FTIR spectrometer (Thermo Nicolet Ltd., Vernon Hills, IL, USA), and the sample was analyzed at the range of 400–4000 cm⁻¹, using 32 scan summations and 4 cm⁻¹ resolution. The DSC curve of milled particles was conducted on a Netzsch differential scanning calorimeter (204 F1, Netzsch instrument Co., Ltd., Germany) in a nitrogen atmosphere at a heating and cooling rate of 10 °C/min. Thermogravimetric analyses (TGA) were carried out using a Q50 system (TA instrument Co., Ltd., USA). The temperature ranged from 40 °C to 600 °C at a heating rate of 10 °C/min. The thermodynamic properties of samples were studied by

a dynamic mechanical thermal analyzer (DMA) (Q800, TA instrument Co., Ltd., USA). The experiments were conducted at a fixed frequency of 1 Hz and at a heating rate of 10 °C/min. The tests were carried out in the temperature range of -130 °C to 100 °C, using the single bending mode.

The samples were prepared by a flat vulcanizing press (YJ63 \times 2, ChengduRichtechnology Co., Ltd., China) at 190 °C. The microstructure and dispersion of samples were observed by optical microscope (VHX-1000, Keyence Co., Ltd., Japan) with a VH-Z100R zoom lens (\times 100 magnification) at ambient temperature. A scanning electron microscope (SEM) (FEI Instrument, USA) with an accelerating voltage of 20 kV was used to study the morphology of samples. Before SEM observation, the samples were quenched in liquid nitrogen, and the fractured surfaces of the samples were coated with a thin layer of gold by ion sputtering prior to visualization. Dispersion of elements was obtained by Mapping (OXFORD Instruments Co., Ltd., UK).

Rheological properties were measured by a capillary rheometer (Rosand RH7, Malvern Instruments Ltd., UK). Shear rates ranged from 100 to 4000 s⁻¹, and samples were maintained at 190 °C.

The samples for mechanical properties testing were prepared by a HAAKETM minijet pro (Thermo Fisher Scientific Inc., Germany) at 190 °C. The tensile strength and flexural strength of samples were tested using a universal testing machine (5567, Instron, USA) at ambient temperature with a crosshead speed of 10 mm/min, according to ISO 527 and ISO 178 standards. In addition, the unnotched impact strength was evaluated with specimen dimensions of $4 \times 10 \times 100$ mm³, according to ISO 179, using a pendulum impact testing machine (Shenzhen Wance Testing Machine Co., Ltd., China).

3. Results and Discussion

3.1. Characterization of Raw Material

As part of our study, we conducted a thorough examination of the materials acquired. Our approach involved a selection and classification process, followed by a component determination. Given the complexity of the usage scenario, the raw materials consisted mainly of a diverse range of components. Based on our analysis, as depicted in Figure 2, the components could be broadly categorized into several groups. We used DSC and FTIR measurements to investigate the materials, as shown in Figure 2b–g. Our findings revealed the presence of various polymers, including PE, PET, PVC, inevitable sandstones resulting from the recycling process, incompletely recycled sawdust, and other impurities. Additionally, a small amount of Al was detected, which had not been fully recycled. Table 1 provides a breakdown of the weight percentage of each component. The dimensions of the various components ranged from 2 to 8 mm, with some reaching 4–5 cm.

3.2. Morphology of Treated PMW

The present study investigated the impact of S³M treatment on the morphology of PMW via the utilization of SEM and a particle size analyzer. The milling process was performed on PMW for various milling cycles, and the resultant powders were collected for characterization and determination purposes. The process of ball milling involves gradually reducing materials to a powdered state by means of the impact, friction, and grinding of milling balls. Conversely, air jet milling involves pulverizing materials into small particles by utilizing the impact and shear force of high-speed rotating blades and air flows. However, due to the inherent flexibility and extensibility of plastic films, the generation of sufficient collision and shear forces during ball milling or air jet milling presents a challenge in breaking them into particles of a sufficiently small size [19]. In comparison with other methods of pulverization, S³M technology can provide a strong shear force field that serves multiple functions, including pulverizing, dispersal, and material mixing. As evidenced by Figure 3, common techniques such as ball milling and air-jet comminution prove ineffective in pulverizing ductile polymer materials, whereas PMW can be successfully milled to powder through the use of S³M. Following milling treatment, the average size of PMW

was reduced from its initial value of 700 μ m to 226 μ m, as demonstrated in Figure 4a–d. Figure 4c indicates that the average particle size continued to decrease until the 4th milling cycle, with no further size reduction observed beyond the 6th milling cycle, at which the average particle size reached approximately 250 μ m, a size consistent with the reported value (400 μ m) in previous studies on room-temperature micronization [20]. Additionally, particle size distribution maps from various milling cycles were obtained and presented in Figure 4d, with particle distribution ranges tending to become smaller and narrower as the number of milling cycles increased. These experimental findings suggest that micronization through S³M significantly reduces the particle size of PMW.



Figure 2. Source of raw materials (**a**); Composition of PMW and their FTIR and DSC ((**b**,**e**)-PE, (**c**,**f**)-PET and (**d**,**g**)-PVC).

Table 1.	The weight	percentage	(wt%) of	Tetra	packaging	components.
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Components	PE	РЕТ	PVC	Sawdust	Sandstones	Impurities
wt %	67%	18%	7.5%	1.5%	1%	5%



Figure 3. Comparison of S³M technique with other methods (ball milling and air jet comminution).



Figure 4. SEM of PMW particles without (**a**) and with (**b**) milling treatment; Particle size (**c**) and particle size distribution (**d**) of PMW after different milling cycles.

3.3. Chemical Characterization of Treated PMW

To investigate the alteration in chemical bonds within a polymer's molecular structure and its constituent components' interaction during the S³M process, FTIR spectra were obtained for the samples, as shown in Figure 5a. The oxygen-containing peaks of the milled particles at 1700–1800 cm⁻¹ and 3000–4000 cm⁻¹ displayed heightened narrowness and sharpness. These results revealed that mechanochemistry activated PMW and increased its surface activity. The free radicals created by molecular chain fracture and unsaturated bonding become more susceptible to oxidation [21]. Furthermore, no new functional groups were formed during the milling process, as indicated by the absence of any new absorption peaks in the infrared spectra. The effect of S³M treatment on the crystallization behavior of PMW was studied by DSC. Figure 5b illustrates five distinct melting peaks, namely LDPE, HDPE, PP, and PET. HDPE exhibits distinct melting peaks (129 °C and 134 °C) due to differences in molecular weight. The milling process induces molecular chain breakage and a decrease in molecular weight, thereby decreasing the melting temperature. As the milling cycles increase, the melting enthalpy of the milled PE at 108 °C was reduced from 15.13 J/g to 5.8 J/g in the unmilled sample, indicating the destruction of crystallization. The reduction in crystallinity is mainly attributable to the decreased particle size after the S³M process, which hinders the orderly arrangement of the polymer molecular chain, thereby restricting their movement [22].



Figure 5. FTIR (a), DSC (b), TG (c) and DMA (d) of PMW after different milling cycles.

The thermal degradation temperature of composites is a crucial indicator in the processing process of polymer. The thermogravimetric (TG) curves of PMW were examined in Figure 5c, and two main processes were identified during the thermal decomposition. The first process involved the decomposition of PVC, with a weight loss of 15 % observed between 170 °C to 330 °C. The second process was attributed to the decomposition of other polymers in composites including PE, PET, and impurities, which resulted in a major degradation peak at 450 °C. Additionally, the study revealed that a slight loss in mass, mainly due to water volatilization, occurred at temperatures below 170 °C. The residual carbon content was found to be 28%, and this was mainly attributed to inorganic filler residues. The thermal degradation temperature of milled composites showed little variation, with a decrease in the amount of residual carbon. This was due to the reduction in particle size and the increase in specific surface area, leading to more complete degradation. The mass of composites remained constant after heating them up to 500 °C, with 28 wt.% inorganic filler residues. Additionally, a molecular-level compatibility of the composites was characterized using DMA. Figure 5d shows the loss modulus for samples with varying milled times. The loss modulus is a measure of energy dissipation and viscosity of a material. The loss modulus was found to increase with increasing milling cycles, as the internal friction increased, resulting in more energy loss in the form of heat. The composites exhibited multiple peaks due to the presence of various components. Under the strong three-dimensional shear force field, macromolecular free radicals were generated by the fracture of polymer chains, which provides the possibility for mechanochemical synthesis of block and/or graft copolymers [21,23]. As the milling cycles increased, the molecular chain broke to form free radicals, which enhanced the interaction between the components. The peaks of the loss modulus of the composite gradually closed with each other, indicating that there was a strong interaction between the molecular chains and demonstrating better compatibility.

3.4. Morphology of PMW Samples

The mechanical properties of materials are affected by their microstructure, which can be controlled by appropriate processing techniques. In this study, self-designed pan-milltype equipment was used to alter the morphology of samples. An optical microscope was used to further analyze the homogeneity between the phases of the samples. Six samples obtained by compression-molding at 190 °C with different milling cycles were examined, as shown in Figure 6a–c. The experimental results showed that the untreated sample had a disorderly morphology due to its large size and uneven mixing among components, resulting in a large area of phase separation on the surface. In contrast, the surface of PMW was more uniform with no obvious phase separation phenomena after S³M treatment. Microscopic observations of samples after milling 10 cycles indicated very few micron-size phase-separated particles. This suggests that the milling treatment had a significant effect on the morphology of the samples, leading to improved homogeneity between the phases, particularly for PMW, which showed better dispersion and smaller filler size.

The cross-section morphology of a sample is a critical factor that affects the mechanical properties of multicomponent polymers. Here, the sample was quenched in liquid nitrogen and observed under a microscope. Figure 6d shows that the sample without milling had an uneven morphology, with large particle sizes and gaps between the component. As a result, the phase separation was severe, and the interfacial adhesion was poor. The average size of the dispersed phase was found to be 9.64 μ m, with a wide distribution. However, after 10 milling cycles, the cross-section of the sample appeared smooth, as shown in Figure 6g. Upon magnification, it was observed that the particles' size was smaller, and the accumulation between the particles was closer, with a reduced gap between them. The size of the dispersed phase in PMW decreased to 2.65 μ m, and the distribution became narrower. It is worth noting that the phase domain size plays a crucial role in determining the mechanical properties of multiphase and multicomponent polymers.

Figure 6d–i presents the SEM image and the size of dispersed phases of PMW. The increase in specific surface area caused by the reduction in particle size led to a rapid increase in surface energy, promoting physical adsorption and subsequent aggregates of particles. Laser light scattering method was inadequate to determine the size of such aggregates. To better reflect the size of the dispersed phase, SEM was used. Without milling, the sample's cross-section appeared uneven after being quenched in liquid nitrogen (Figure 6d). This was due to the larger particle size and greater gaps between each component, which resulted in serious phase separation and poor interfacial adhesion. The average size of the dispersed phase was 9.64 μ m, with a wide distribution. In contrast, after 10 milling cycles (Figure 6g), the cross-section of the sample became smoother, with finer particles and closer particle accumulation. The dispersed phase size decreased to 2.65 μ m, with a narrow distribution. It is well-known that the phase domain size of multiphase and multicomponent polymers greatly affects their mechanical properties.



Figure 6. Optical micrographs of PMW with different milling cycles: (**a**–**c**) were samples with 0, 6, and 10 milling cycles. SEM images of samples with different milling cycles ((**d**,**e**) were the samples without milling, (**g**–**h**) were the samples with 10 milling cycles) and their size of dispersed phase (**f**,**i**).

To analyze the distribution of each component in the sample, we conducted elemental surface scanning and obtained the corresponding distribution maps, as depicted in Figure 7. The Ca component in the sample mainly originated from sand and stone, while Cl was derived from PVC, which was consistent with the content of PVC in Table 1. The distribution of elements in the sample without milling was relatively concentrated, whereas after

S³M treatment, the elements were evenly dispersed throughout the sample. As a result, the prepared samples had a more homogeneous dispersion structure. S³M technology can achieve ultrafine grinding and uniform dispersion of PMW at room temperature. The particle size and distribution of the powder determine the size of the phase domain. Moreover, according to previous reports, S³M technology can generate a small amount of block copolymers in situ [22,24], as some of the molecular chains were ruptured during the S³M process, and more polar-functional groups are produced, seen in Figure 5a, which enhances the interaction between the components and reduces the interfacial tension, resulting in better compatibility. This molecular-rearrangement reaction, as shown in Figure 8, promotes closer bonding between the milled particles, thereby improving compatibility. Therefore, S³M technology represents an important breakthrough in realizing the recovery and utilization of multicomponent and multiphase PMW without requiring a compatibilizer, viscosity matching, or a separation and classification process.



Figure 7. EDS of samples without milling (a) and milled for 10 cycles (b).



Figure 8. Schematic diagram of compatibilization mechanism in domain size control (**a**) and molecule structure change (**b**).

3.5. Shear Viscosity of PMW Samples

The processing of PMW without S³M treatment is hindered by the large particle size, which causes blockages even at temperatures as high as 250 °C, making reprocessing a significant challenge. However, after S³M treatment, the particles decrease, allowing for extrusion and injection at lower temperatures than 190 °C. Figure 9 depicts the shear viscosity of the composites obtained by a capillary rheometer. The viscosity of the sample decreased with the increase of milling cycles due to the smaller size of the dispersed phase, which enhances the chain slip, entanglement, and macromolecule chain activity. Additionally, the apparent viscosity of macromolecular chains decreases along the flow direction with an increase in shear rate. Thus, S³M treatment facilitates the transformation of polymer processability.





Figure 9. Rheological properties of PMW after different milling cycles.

3.6. Mechanical Property

The feasibility of recycling PMW was assessed by evaluating the mechanical behavior of the recycled composites. Figure 10 presents the mechanical properties of recycled waste PMW after undergoing different milling cycles. The dispersion and compatibilization of composites were greatly influenced by S³M technology (as evidenced by Figures 5d and 7), which in turn affected their mechanical properties. The experimental results revealed that test slabs underwent brittle fracture when strain was less than 10%. However, after undergoing S³M treatment, the mechanical properties of the samples improved. The tensile stress of the unmilled samples was 14.03 MPa, which increased gradually with an increase in milling cycles. After the sample with 10 milling cycles, the tensile stress increased to 22.02 MPa, with a growth rate of 57%. Moreover, the elongation at the breakage of the sample increased from 3.49% of the untreated sample to 4.40% after S³M treatment. The unnotched impact strength test shows that a sample's unnotched impact strength was enhanced from 3.26 KJ/m^2 to 4.82 KJ/m^2 . The mechanical properties of the samples are associated with their structure, as indicated by Figure 7. The cross-section of the samples without milling was rough, and the size of the dispersed phase size was large, which increased the probability of stress concentration. Consequently, under external loading, cracks were generated and propagated quickly, leading to poor mechanical properties of the materials. However, the cross-section of the samples milled for 10 cycles was more uniform, and the interfacial contact area between the particles increased due to the particledispersed phase size reduction and the improved compatibility, which reduced stress defects during the tensile process. Therefore, the mechanical properties of composites after $S^{3}M$ treatment improved. This phenomenon is in line with the Griffith theory of brittle fracture of materials [25].





4. Conclusions

This study investigated the use of S^3M equipment to recycle post-consumer multiplastic waste, with the aim of overcoming the sorting and cleaning challenges posed by the traditional recovery process. Results show that the S^3M process can reduce the size of the recovered multicomponent from 700 µm to 226 µm, with a smaller and narrower particle distribution range observed with increased milling cycles. The S^3M process also results in decreased crystallinity of PMW after the S^3M process, good dispersity of domains, and reduced phase domain, which improved compatibility between components in composite and increased mechanical properties. The tensile stress increased from 14.03 MPa to 22.02 MPa, and the unnotched impact strength was also enhanced. Moreover, the recycled material can be extruded without any blockage of processing instruments. These findings demonstrate the great potential of the S^3M process for the processing of APPW, which can be transformed into a useful product without any separation. The recycling of multilayer plastic films has significant environmental and resource utilization benefits; thus its future prospects are very promising.

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