Toughening Approaches of Recycled Polystyrene

Subjects: Polymer Science

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Several environmental and techno-economic assessments highlighted the advantage of placing polystyrene-based materials in a circular loop, from production to waste generation to product refabrication, either following the mechanical or thermochemical routes.

Keywords: polystyrene ; recycling ; waste plastics

1. Introduction

Badische Anilin und Soda Fabrik (BASF) introduced the first commercial-grade polystyrene (PS) in the 1930s. Since then, PS has captured its niche market owing to its cost-effectiveness, excellent processability, low density, clear appearance, dimensional stability, and amenability to be sterilized by radiation. Today, the applications of PS are diverse, and it has even managed to outcompete other plastics in various industries. Unfortunately, as with other plastics in the market, the utilization of PS was aimed at single-use. The disposable and lightweight nature of different PS-derived materials led them to accumulate in the environment as wastes with no facile mechanism for degradability.

The impediment to recycling PS is like those of many plastics. However, it is compounded by the negative scrap value of expanded polystyrene (EPS), the preponderant PS-derived litter ordinarily available as food packaging material. In contrast, recycled plastic fractions giving the highest profit are rigid PS packaging (2021 price levels), with a 14% internal rate of return ^[1]. This disparity is due to the product's price and yield, which determines recycling's economic incentives. Moreover, visual segregation of PS from the mixed waste stream is challenging, and the presence of other specific contaminants could significantly affect the recovery ^[2]. Plastics based primarily on high-impact polystyrene (HIPS) and acrylonitrile–butadiene–styrene (ABS) constitute the lion's share of waste electrical and electronic equipment (WEEE), thus garnering increased interest in recycling these products ^[3]. However, the intrinsic immiscibility of the polymer's components and the varied nature of the sourced polymers remain a challenge in advancing the recycling of WEEE as sources of next-generation polymeric materials.

Today, in the age of accelerating industrialization and urbanization, the modern society's solid waste management programs have considered the gradual closing of the circular loop in terms of sustainably recycling and reusing polymers. The main problem in utilizing post-consumer polymers over and over is the consequent degradation of their mechanical strength, often regenerating polymers of low value. This form of recycling is known as downcycling. In contrast, enhancing the mechanical strength of recycled materials or the regeneration of high-value polymers or chemicals in recycling is called upcycling ^[4].

1.1. Polystyrene

Styrene is a petroleum-derived, liquid, aromatic hydrocarbon, and is the precursor monomer of polystyrene (PS). PS is a thermoplastic that is usually solid at room temperature, melts at an elevated temperature during molding or extrusion, and then solidifies. As a thermoplastic, joining or welding PS is feasible, limited only by the brittleness of the employed PS grades. The radio and ultrasonic frequency welding technique or solvent bonding can be utilized with some PS grades ^[5]. As a film, PS possesses high tensile strength and excellent transparency, but with poor gas, moisture, or vapor barrier properties. However, this low gas barrier property enables the fabrication of 'breathable' PS films. PS is generally brittle and exhibits less chemical resistance to organic solvents from aliphatic, aromatic, and chlorinated varieties, including acids and bases, ketones, and cyclic ethers.

Nevertheless, in dilute aqueous solutions of acids or bases and aliphatic alcohols of high molecular weight, PS is moderately resistant. On the other hand, PS exhibits chemical resistance to ethylene oxide (EO), low molecular weight alcohols, and oxidizing and disinfecting agents such as bleach. In fact, EO has been used to sterilize PS-based materials, with no significant effects on physical properties upon exposure ^[I]. Sterilization using steam or an autoclave is not

employed with PS due to the low heat distortion temperature. Exposure to gamma and e-beam radiation is also commonly employed in sterilizing medical-grade PS-based materials without a significant compromise on the properties of PS ^[B]. This stability is due to the high aromatic content of PS, wherein the electronic clouds of the phenyl moieties can neutralize the reactive free radicals generated on exposure ^[9]. Several grades of commercially biocompatible PS copolymers are available in the market, although PS is not normally employed in applications that necessitate biocompatibility.

1.1.1. Chemistry and Synthesis

The polymerization of the styrene monomer via the generation of free radicals is the main route in the synthesis of polystyrene. This free-radical polymerization employs free-radical initiators activated by thermal or radiation sources.

PS can be synthesized in three different forms depending on its tacticity: atactic, syndiotactic PS (sPs), and isotactic PS ^[10]. Tacticity, in this case, refers to the spatial arrangement or order of the phenyl groups within the polymeric chain, and this arrangement influences the polymer's properties. Commercially, atactic PS, a polystyrene having randomly distributed phenyl groups on either side of the polymeric chain, is preponderantly available. At the same time, the sPS was initially jointly introduced in Japan by the Idemitsu Petrochemical Company and Dow in 1988. The phenyl groups in sPs are in alternating positions on the polymeric chain, resulting in the semi-crystallinity of PS. The melting point of this engineering polymer, sPS, lies at ca. 270 °C and is synthesized through a continuous polymerization process similar to polyolefins that employ metallocene single-site catalysts. Although sPS is brittle, it exhibits high chemical resistance and possesses a very low dielectric constant. Glass reinforcement or blending with other polymers are a few ways to circumvent the brittleness issue of sPS. Moreover, the ease of processing and high flow of sPS are suitable material characteristics required for thin-wall applications.

1.1.2. Polystyrene Grades and Applications

Polystyrene generally exists in two chemical grades: crystal polystyrene and HIPS. The crystal PS or general-purpose PS is mostly synthesized via the bulk polymerization technique and considered unmodified polystyrene. Crystal PS-derived resins are glassy and very transparent, supplied principally as granules. Physically, PS can be in solid, foam, or expanded (EPS) forms. The general-purpose or crystal polystyrene, although brittle, is the thermoplastic of choice for PS injection molding and extrusion processes. Out of the extrusion process come many packaging materials and extruded trays. The injection molding of crystal PS yields several medical components and diagnostic labware, which are not limited to syringe hubs, petri dishes, canisters, and trays for tissue culture. Some office accessories and dinnerware such as cups or tumblers, cutleries, and other plastic housewares are manufactured by the injection molding process. Foamed sheets out of extrudable crystal grade PS yield most of the commonly employed materials for meat packaging and the fast-food industry sectors. In contrast, various insulation materials for the building industry come out of foamed boards. The general-purpose PS are characteristically hard and rigid, exhibit high gloss, good electric/dielectric properties, and dimensional stability, with low water absorption and limited chemical resistance, but possess excellent resistance to yradiation. The processability of crystal grade PS is excellent; however, it is prone to stress cracking in specific environmental conditions. In comparison, HIPS is tougher, having improved impact resistance, and thus is mostly utilized in thermoforming. HIPS is characteristically translucent to opague in appearance, exhibits low gloss, and has reduced electrical properties and fair dimensional stability, with reduced water absorption and resistance to chemicals, but fair resistance to y-radiation. HIPS also exhibits excellent processability and is less prone to cracking if stressed in specific environments. Thermoformed medical products that are HIPS-derived include epidural, catheter, and heart pump trays, suction canisters, syringe hubs, and respiratory care equipment. Both chemical forms of PS compete with polypropylene (PP), poly(vinyl chloride) (PVC), or acrylic-based materials, especially for a lot of respiratory and medical products.

• High-impact polystyrene (HIPS):

HIPS is PS with a bright white appearance and is derivatized with rubber-like moieties. During styrene polymerization, the rubber-like polybutadiene is incorporated into the growing polystyrene chains. The in situ grafting and partial cross-linking of the butadiene moieties leads to better adhesion between the rubber-like particles and the PS matrix, resulting in toughness improvement. In turn, this reaction significantly affects the final polymer's properties. HIPS resins have characteristically good dimensional stability, rigidity, and impact strength, and are easily processable. In 3D printing, the HIPS has a wide print temperature range of ca. 210–250 °C.

The elastomeric content of high-impact grade PS ranges from 6–12%, while medium-impact grades contain about 2 to 5% elastomers. HIPS-derived PS are common commodities in the food packaging or consumer product business in the form of yogurt or dairy containers, drinking bottles, cups or tumblers, lids, and dinnerware, where impact resistance is essential. HIPS is also a component of many consumer electronics, refrigerator linings, and high-quality office appliances, and is

used in the manufacturing of toys, audio-visual equipment, and medical devices. HIPS is generally biocompatible and has been used long-term in high-impact prosthetic devices with no adverse effects in contact with human tissues. Several 3Dprinted surgical or dental instruments have been designed using HIPS.

• Oriented polystyrene (OPS)

The crystal polystyrene grade remains brittle except when it is biaxially oriented, becoming flexible and durable. This stretching of the PS sheet in the transverse direction yields oriented polystyrene (OPS), resulting in the toughening of the polymer sheet. Culinary baking and delicatessen trays are fabricated from extruded OPS.

• Expanded polystyrene (EPS)

Expanded polystyrene (EPS) is a plastic consisting of a stiffened cellular structure resulting from molded spheres or little pearls of expandable polystyrene, generally a mixture of approximately 5% PS polymer and 95% air ^[11]. Blowing agents like pentane or chlorofluorocarbons are needed to generate air space. Because the entrapped air fills the voids as the polymer stiffens, the material becomes lightweight with low thermal conductivity. Among the rigid insulation foams, EPS has the lowest average R-value and is the most cost-effective. For these reasons, EPS is aptly used as insulation in extrudable foamed board forms such as insulating concrete foams (ICFs) and structural insulation panels (SIPs) in building construction, and as patterned sheeting in other engineering or architectural applications, although it is fragile to work with. EPS absorbs water; however, it is rated for ground contact and can be rendered as an insect-repellent. EPS is semipermeable, and unable to create a vapor barrier.

• Styrenic copolymers

Styrenic copolymers are PS derivatives that are structurally similar to HIPS. The chemical derivatization of PS has been shown to improve its rigidity, chemical, and scratch resistance, among others. One of these styrenic copolymers is the Acrylonitrile-Butadiene-Styrene (ABS) Copolymer, mostly obtained from emulsion polymerization. In the synthesis of ABS, submicron rubber-like particles are initially produced from an emulsified butadiene before grafting styrene-acrylonitrile copolymer on the shell of rubber particles, which eventually leads to a grafted rubber concentrate (GRC). GRC is then finally compounded with styrene-acrylonitrile copolymers (SAN) as a toughening approach in specific applications. ABS can also be obtained by a mass process like HIPS. However, in comparison with HIPS, ABS polymers are tougher and more chemically resistant but are more challenging to process. ABS-based polymers are utilized in the interior trim of automotive components, luggage, household pipes, housing for power tools and vacuum cleaners, and in the fabrication of plastic toys.

Another styrenic copolymer is SAN. SAN is synthesized by copolymerizing the acrylonitrile (AN) monomer at 15 to 30% by weight with the styrene monomer (SM) ^[12]. SAN-derived materials are for applications necessitating extra attributes such as toughness, excellent chemical resistance, and high heat distortion temperature. These attributes are highly considered in the fabrication of toys, shower doors, and bottles or containers used in the cosmetic industry. SAN resins are a major component in the compounding of ABS resins by emulsion polymerization.

Other styrenic copolymers also exist, including maleic anhydride copolymerized with styrene and transparent impact polystyrene (TIPS). Styrene and maleic anhydride copolymers are used in high-heat applications. TIPS is synthesized by copolymerizing styrene and butadiene via the anionic block copolymerization technique. Because most styrenic copolymers are structurally similar to PS, they could be tailored as phase homogenizers in PS/other plastics blends.

• Brominated polystyrene:

This type of PS is based on HIPS, derivatized with bromine moieties in the polymer chain or modified by incorporating halogenated and analogous additives, rendering it with ignition or flame resistance properties. TV housings and electronic equipment such as computers, printers, and copiers employ brominated PS during fabrication. Some WEEE may contain brominated PS.

2. Use of Compatibilizers

Recycling is a time- and cost-intensive undertaking. These reasons are enough to discourage the already few existing recycling industries. Apart from determining the appropriate recycling methods, the chemical compatibility between the diverse nature of waste streams needs to be considered. Otherwise, phase separation of the different components could lead to low-quality products. To circumvent phase separation, chemical modifiers are introduced during the re-extrusion of

plastic wastes. The modifiers are also called coupling agents, compatibilizers, or performance enhancers. Compatibilization is a viable option to negate the exacerbating costs of upcycling PS waste. Regarding the chemical structure, some compatibilizers are oligomer/polymers synthesized as block (*b*), graft (*g*), and random (r) copolymers. Each compatibilizer's chemistry is tailored to the mixture's thermodynamic interactions between immiscible components. The compatibility improvement in blended polymers involves incorporating another substance (the compatibilizer) which can facilitate the cost-effective conversion of plastics to high-value products by optimizing their miscibility and stability ^[13]; hence, compatibilization is an upcycling technique.

It has been reported that PS fraction in WEEE plastic is the predominant polymeric component, necessitating recovery of this polymer in WEEE ^[15]. Recycling of this fraction without separating it into components was demonstrated by Grigorescu et al. using a melt compounding technique ^[16]. In ^[16], a blend of styrene-butadiene-*b*-copolymer (SBS) and hydrogenated and maleic anhydride-modified SBS (SEBS-*g*-MAH) acted as the compatibilizers or impact modifiers. It was claimed that the PS-derived composites from this process have improved UV and flame resistance (while no such testing was conducted), and the physical and mechanical properties were comparable to HIPS.

It is possible to fabricate ABS/HIPS blends recovered from WEEE. Hirayama et al. further demonstrated that the recovered ABS/HIPS blend could have controllable mechanical properties using compatibilizers such as styrene– butadiene–styrene (SBS) and styrene–ethylene–butylene–styrene/styrene–ethylene–butylene (SEBS/SEB) ^[3]. SBS and SEBS/SEB as compatibilizers had significantly affected the morphology of HIPS/ABS. Adding the compatibilizers changed the size and shape of the dispersed PS-derived phases, resulting in mechanical property changes in the polymer. Incorporating virgin polymer in the blends also affected the morphology.

In Jaidev et al.'s blending work, recycled PVC and ABS and recycled HIPS were recovered from uninterrupted power supply (UPS) housing and television housing, respectively ^[17]. The phase homogeneity in the PVC/ABS and PVC/HIPS blends was inferred from morphological, thermal, and mechanical analyses. At similar composition of r-PVC, the blend with r-ABS, has better mechanical properties than the blend with r-HIPS.

3. Addition of Tyre Crumbs or Rubber

Recycling tires at end-of-life can facilitate the recovery of functional raw materials for future use that is sustainable and environment-friendly. Valuable materials like rubber, carbon black, steel, and fibers can be recovered from discarded tires. Devulcanized rubber tires (DVR) as fillers could be a sustainable way of recycling waste rubber tires and PS. DVRs have been used as fillers for thermoplastic thermal insulators ^[18]. They have been incorporated in powder form into PS from 0– 50 wt.% using a melt extruder. In addition, the compressive strength was in the range of 11.66 to 7.47 MPa, and the flexural strength was ca. 40.4 to 19.26 MPa. Hashin & Shtrikman's conduction models (series and parallel) validated the thermal conductivity results. Furthermore, the DVR filler treatment with a base had a positive compatibilizing effect, significantly improving the mechanical and thermal stability of the composites.

Ground tire rubber (GTR) with particles lower than 200 microns has been blended with several polymers. In fact, six potential low-requirement insulator applications, were determined that meet the rigorous requirements of the Spanish Association for Standardization (UNE) and the International Electrotechnical Commission (IEC) ^[19]. Although PS/GTR blends were part of this entry and found to be sufficiently insulating, with minimal decrease in their mechanical properties (tensile strength and elongation at break), the overall properties exhibited by PS/GTR blends do not conform with the strict requirements set by UNE and IEC for electrical applications, paramount of which was the elongation at break.

4. Ternary Blends

The blending of polymers has the distinct advantage of interfusing the properties of each component polymer. Binary blends of polymers have been commonly employed, and only recently did ternary blends catch the attention of material scientists. Ternary blending leads to a variety of micro and nano morphologies. The structuration at this domain level has been demonstrated to improve the mechanical properties of the employed matrix thermosets ^[20] and thermoplastics ^[21] ^[22]. An exploration of the ternary blending of PS has been carried out by several researchers ^{[23][24][25]}. However, only a few reports exist where PS was upcycled from the waste stream. The straightforward ternary blending approach would encourage PS-based recycling.

Jaidev et al. identified the polymers from keyboard waste aided by the keyboard parts' embossed resin identification code (RIC). They formulated a ternary blend from the recovered polymers ^[15], intending to improve the thermal, mechanical, and morphological characteristics. The recovered polymers PS, ABS, and HIPS from the WEEE have impact strengths of

29 J·m⁻¹, 42 J·m⁻¹, and 20 J·m⁻¹, respectively. When a ternary blend contained PS as the major phase, the impact strength increased to 66 J·m⁻¹. An improvement in the ternary blends' thermal stability from 380 °C to 396 °C was observed, with a negligible effect on the glass transition temperature (*Tg*). The blends' salami morphology enabled a brittle to ductile transition, improving the observed characteristics.

Relying on the RIC and Fourier transform infrared (FTIR) spectroscopy to sort waste keyboards components, in a similar study formulating ternary blends of varying levels of PS, ABS, and HIPS, the optimum tensile strength, flexural strength, and impact strength of 35 ± 3 MPa, 65 ± 3 MPa, 45 ± 3 J·m⁻¹ were determined, respectively, for the ternary blend ^[26]. Morphological and thermal analyses of impact fractured specimens revealed that the blends were homogeneous. A 98% degradation of the blends was also observed at 700 °C using thermogravimetry analysis (TGA). No significant changes in the properties of blends were noted, paving the way for the elimination of multiple sorting of plastic components, thereby reducing the cost aspect with this upcycling approach.

5. Ionic-Crosslinking

The structure of PS consists of bulky and rigid phenyl groups dangling on the polymer backbone, and the segmental interaction along the chain is weak and unpolarized. These characteristics of PS result in its poor impact toughness and fair heat resistance. Cross-linking with ionic moieties has been demonstrated to circumvent these effects. Cai et al. used zinc dimethacrylate (ZDMA) to demonstrate ionic cross-linking into recycled ABS/HIPS blends ^[2Z]. ZDMA could self-polymerize, forming poly-ZDMA (PZDMA) particles. Cross-linking could occur by graft polymerization with butadiene on the recycled HIPS or ABS chains of ZDMA. The ionic interaction between unsaturated carboxylate and Zn²⁺ improved the compatibility between recycled HIPS and ABS. An improvement in the blends' storage and loss modulus, impact strength, and tensile strength were observed. The notched impact strength at four wt.% ZDMA was 133% higher than that of blends without ZDMA. The polybutadiene (PB) phase was well dispersed into the recycled HIPS/ABS blends, as seen by using SEM.

Another use of Zn-based ions for cross-linking PS was demonstrated by Yu et al., although neat PS was employed. It is expected that the methodology could also apply to the PS waste stream. Yu et al. aimed to enhance the heat resistance during the melt blending of PS by incorporating a PS ionomer derivative, the high-ion-content Zn-salt poly(styrene-*ran*-cinnamic-acid) (SCA-Zn) ^[28]. This ionomer derivative needed to be of dense ionic cross-linking but of low molecular weight to enhance the blend's *Tg* and ease of processing during melt blending. Consequently, SCA was synthesized by copolymerizing styrene with 20 wt.% cinnamic acid. SCA was then melt-reacted and neutralized by excess ZnO to ca 21,000 g mol⁻¹, deemed the half-critical weight average molecular weight (Mw) for this purpose. Varying levels of SCA-Zn were incorporated with PS during melt blending with good compatibility. The phase-separated morphology of the 60/40 PS/SCA-Zn blend resulted in higher heat resistance.

6. Addition of Polymers, Organic and Inorganic Fillers

Adding other oligomers or polymers, organic and inorganic, is an effective toughening method for the polymer matrix. Hayeemasae et al. prepared a composite based on recycled PS and eggshell powder (ESP) [29]. As the secondary filler, calcium carbonate (CaCO₃) was added to reinforce the composite's performance. During the extrusion of the ESP/CaCO₃ hybrid PS composites, the torque decreased with the increasing percentage of CaCO₃. Furthermore, increasing the percentage of CaCO₃ in the hybrid composites enhanced Young's modulus, impact strength, tensile strength, and elongation at break. Moreover, CaCO₃ provided better heat resistance than ESP. The agglomerates toughened the PS matrix instead of acting as defects. The amorphous polymer then exhibits mechanical properties close to the semicrystalline polymer, causing higher deformations and lower modulus. The low filler level enabled the organized agglomeration in a stacked way, rendering them with reinforcement and super lubricity effects. Ab Ghani et al. used graphene nanoplatelets as a filler in PS and found that 15 wt.% of styrene butadiene rubber (SBR) as the toughener exhibited the best mechanical properties [30]. Physical reinforcement of the brittle PS has been demonstrated using polyhedral oligomeric silsesquioxane (POSS) [31]. POSS is chemically inert, and only physical interactions are possible with PS; however, different structural types of POSS (lamellar and dumbbell-shape) significantly influenced the overall properties of fabricated composites. Chemical interactions with PS are facilitated by incorporating appropriately functionalized POSS, as in the case of POSS cages with vinyl or vinylidene moieties. The mechanical properties of SiO₂-PS composites with grafted chain conformation were dependent on the P/N ratio [32], where P is the degree of polymerization of the matrix and N is the degree of polymerization of the grafted brush. High composite strength is expected at N ~ P, with swollen grafted chains. The grafted chains at N < P are expected to be collapsed, facilitating particle pull-out. Thus, tuning the P/N ratio could increase strength or strain energy density.

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