EXPANDED POLYSTYRENE (EPS) RECYCLING: AN ENVIRONMENT FRIENDLY PROCESS TO RECOVERY OF DISSOLVED POLYSTYRENE (PS) USING WASTE COOKING SOYBEAN OIL

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Resumo

The current work presents a simple method to recycle waste expanded polystyrene (EPS), using waste cocked soybean oil (WCSO). The process of polystyrene recovery combines the dissolving of EPS in D-limonene with its further separation and mixture with WCSO. The chemical, thermal, and morphological properties, of the obtained material, were characterized by the techniques, Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), X Ray diffraction (XRD) and Scanning Electron Microscopy (SEM). The characteristics of the recovered polystyrene presented high similarity with the standard polystyrene (PS), which suggests low contamination by other materials. The great advantage of this recycling method is that it allow us to obtain PS in solid state at room temperature, without heating needs, instead of other EPS recycle reported methods. The results are promising and point to a viable and simple process of recycling EPS, which offers several environmental benefits.

Introduction

Expanded polystyrene (EPS) is a polymer used worldwide, and its extensive use has become a disposal problem due to the high volume occupied and the time that it takes to decompose naturally. In fact, managing this waste involves certain difficulties; the low mass/volume ratio of EPS waste leads to a high cost of transport and storage, which discourages recyclers from recycling it. Landfill and incineration of EPS are the two common ways to dispose this waste material (Pol, 2010). However, landfills has disadvantages to being high cost, and the incineration process is high-energy consumption and releases potentially toxic gas. Faced with this, alternative methods for recycling EPS waste are needed.

Several techniques to recycle EPS waste are already being propose. For example, the depolymerization, named chemical recycling, is based on EPS chemical decomposition to produce low molecular weight products (Manrich and Santos, 2009). This method can reduce the volume of plastic that ends up in landfills and can produce high-quality raw materials, decreasing the demand for fossil fuels and other natural resources. Unfortunately, in the chemical recycling process there is a large emission of greenhouse gases, which poses a climate risk (Brandrup, 2006). In the mechanical recycling, polymer waste is mechanically crushed into granules that can be used to make new products. This method keeps the molecular structure, which is less energy intensive than chemical recycling and doesn't use toxic chemicals. The main disadvantage of this method is that plastics are not infinitely recyclable mechanically (Brandrup, 2006). The energetic recycling is when polymer waste is incinerated to produce heat, which is used in useful ways, like in producing electricity. However, installing an incineration plant is an expensive process and the smoke produced during polymer incineration includes acid gases, carcinogenic dioxins, particulates, heavy metals and nitrogen oxide (Brandrup, 2006). Additionally, EPS waste has been usually recycled and added to other materials, such as: wood plastic composites made from post-used polystyrene foam (Koay et al., 2018); wood plastic composites made from post-used dissolved polystyrene packing (Agoua et al., 2013); wood plastic composites using recycled expanded polystyrene and wood flour (Poletto, 2011); development of rice husks–plastics composites for building materials (Choi et al., 2006); composites made from cement dust and polystyrene waste that can be used as building construction materials (Asaad and Tawfik, 2011) ; crumbled recycled foam polystyrene waste is used to produce the filler in concrete (Laukautis et al., 2005); use of expanded polystyrene waste in the creation of waterproofing paint (Bellon et al., 2019); recovery and incorporation of expanded polystyrene solid waste in lightweight concrete (Espinoza-Merchan et al., 2020); producing multi-strength grade lightweight concrete containing expanded polyethylene beads (Sadrmomtazi et al., 2012); and composite of polystyrene/concrete as the structural thermal insulating material (Makhmud and Alexander, 2017). However, none of them are used as widely, mainly because they are not economically profitable.

In order to make the recycling process more attractive, EPS waste has been dissolved by organic solvents to reduce its volume, saving transport and storage costs. The commonly used solvents are: ethanol (Bellon et al., 2019); limonene (Bellon et

al., 2019; Jimenez-Francisco et al., 2020); (Garcia et al., 2009; Gutiérrez et al., 2012; Gutiérrez et al., 2014; Yi-Bo Zhao et al., 2018; Bellon et al., 2019; Jimenez-Francisco et al., 2020), acetone (Yi-Bo Zhao et al., 2018; Bellon et al., 2019), methanol (Yi-Bo Zhao et al., 2018), ethyl acetate (Yi-Bo Zhao et al., 2018), benzene (Yi-Bo Zhao et al., 2018), chloroform (Yi-Bo Zhao et al., 2018), and cyclohexane (Yi-Bo Zhao et al., 2018), Cymene (Garcia et al., 2009), Phellandrene (Garcia et al., 2009), styrene (Mumbach et al., 2020), eucalyptus (Garcia et al., 2009; Gil-Jasso et al., 2019), star anise (Gil-Jasso et al., 2019), thyme (Gil-Jasso et al., 2019), chamomile (Gil-Jasso et al., 2019), and toluene (Singhal et al., 2019). Unfortunately, most of these processes- are environmentally harmful because the solvents most commonly used are toxic, such as toluene, acetone, ethyl acetate, benzene, chloroform, and cyclohexane (Yi-Bo Zhao et al., 2018; Bellon et al., 2019; Singhal et al., 2019). In contrast, dissolution processes using natural and nontoxic solvents are often reported (Garcia et al., 2009; Bellon et al., 2019; Gil-Jasso et al., 2019; Jimenez-Francisco et al., 2020). One of them, D-limonene (hydrocarbon classed as a terpene) has been reported as an attractive natural solvent for waste EPS dissolution(Garcia et al., 2009; Bellon et al., 2019; Jimenez-Francisco et al., 2020). This solvent is an essential oil, obtained from orange or lemon peel as a byproduct of juice manufacture. D-Limonene is generally recognized as a non-toxic substance, and some advantages in using it to dissolve EPS waste include: (i) This process does not produce any degradation of polymeric chains (Gil-Jasso et al., 2019),; (ii) After the dissolution process, the polystyrene can be recovered (Gutiérrez et al., 2012; Gutiérrez et al., 2014; Gil-Jasso et al., 2019); (iii) The solvent can also be recovered and reused to dissolve a new waste EPS batch (Gil-Jasso et al., 2019). Based on these, the process of dissolving EPS in D-limonene has been used as a primary step in polymer recycling. The current work is an example where dissolved EPS and waste cooking soybean Oil (WCSO), when combined, give rise to a new material that can be used for several applications. Sarmiento et al. also show that the expanded polystyrene and waste cooking oil can be used to produce a recycled EPS with potential use as construction material (Sarmiento et al., 2016).

In particular, soybean oil is an important commercial product, represents approximately 30% of the total vegetable oils produced in the world, and it is currently used for the preparation of food (Gunstone, 2002). In this context, waste cooking soybean oil (WCSO) refers to an oil, which has been used for frying and can no longer be used for the same purposes. The incorrect disposal of WCSO can cause several environmental problems(Gunstone, 2002). When the WCSO is discharged, directly in the water tributaries, can forming a superficial layer of oil that reduces oxygen diffusion into the water source, causing several damages to the aquatic ecosystems. It is well known that only 1 L of oil can contaminate 1000 L of drinking water, and for this reason, applications that include the use of this waste are welcome to minimize the WCSO pollution.

Corroborating, this work stems from the idea that the environmental problems caused by the incorrect disposal of EPS and WCSO can be mitigated when both are recycled and combined to recovery the polystyrene (PS).

Materials and methods

In this study, commercial waste of EPS packages was used. The raw material was crushed into small pellets in order to produce a homogeneous material (the average particle size was approximately 4 mm). The solvent D-limonene, 99% purity, was purchased from a local store, Óleos Essenciais ®, and used without further purification.

For dissolution experiments, 50 ml of D-limonene and 15 g of EPS were placed in a Becker, and then the mixture was shacked (~ 15 minutes) until its complete dissolution. This proportion (EPS/D-limonene) was determined in preliminary dissolution experiments. All experiments were carried out at room temperature. After the complete dissolution, the mixture was carefully transferred to a separating funnel where the dissolved polystyrene was separated from the D-limonene, after allowing the mixture to precipitate overnight. Therefore, the lower layer (D-limonene) was drained out. The pasty (upper layer - polyestyrene) was then combine to waste cooking soybean oil (WCSO) to produce a solid polymeric material.

Waste cooking soybean oil was collected from households and street vendors located in Uberaba City, Brazil. After collection, the WCSO was only filtered by filter paper (Whatsman 41 sized 20-25 micron) to remove bits of food residues. The polystyrene was recovered simply by mixing equal volumes of reagents (dissolved polystyrene and WCSO) at room temperature. After agitating for one minute, a solid precipitated. Finally, this solid was squeezed between layers of hand paper towel, to remove the maximum oil excess, and let dry at room temperature.

The chemical, thermal, and morphological properties, of the obtained material, were characterized by the techniques: Fourier Transform Infrared Spectroscopy (FTIR, Cary 660 by Agilent Instruments), Thermogravimetric Analysis (TGA), X-Ray diffraction with Cu K□ radiation with steps of 1 degree (Theta)/min (Shimadzu model 6100) and Scanning Electron Microscopy using the backscattered electron mode (SEM, model LEO 1450VP).

Results and Discussion

As mentioned before, dissolution assays were carried out in Becker, where crushed waste EPS was immersed in D-limonene at room temperature, see figure 1(a). Figure 1(b) shows the separation process between dissolved polystyrene (upper layer) and D-limonene (lower layer), after 48h. This procedure allowed the recovery both D-limonene, which was drained out, and the dissolved polystyrene. All components resulting from the separation were analyzed using FTIR spectroscopy (Fig. 1(c): Polystyrene and Fig. 1(d): D-limonene).

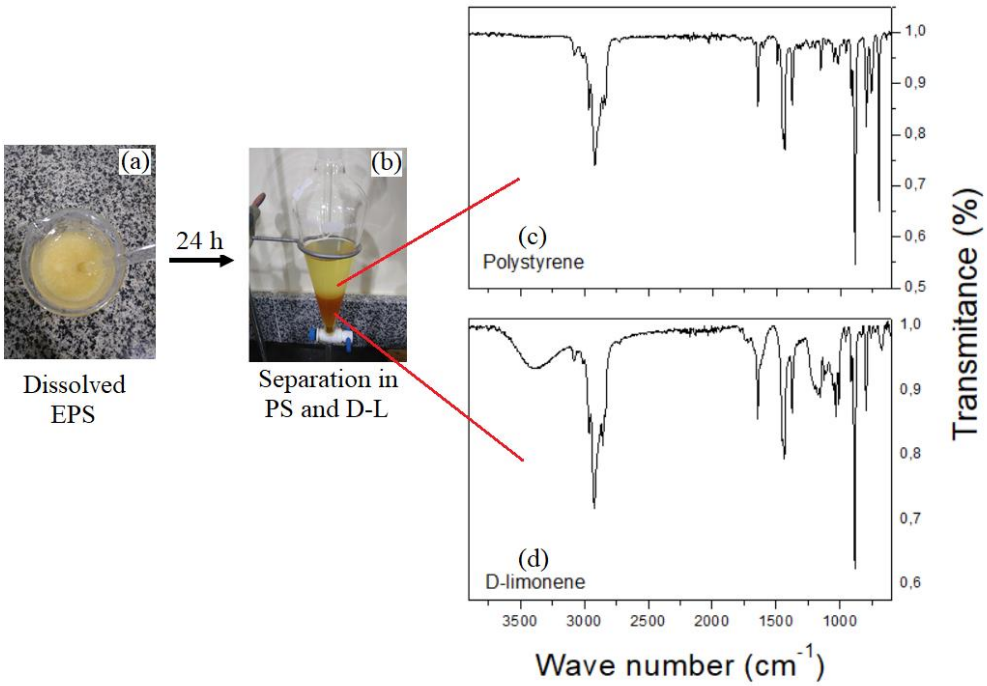


Fig. 1. a) Dissolution of EPS at room temperature by D-limonene, b) Layer separation in dissolved polystyrene and D-limonene, after 48 h, c) FTIR spectra of the dissolved polystyrene and, c) FTIR spectra of the D-limonene.

Figure 1(c) shows the infrared absorption spectra of the dissolved polystyrene. There are several absorption peaks within the involved wavenumber range, similar to the standard polystyrene reported before (Al-Kadhemy et al., 2016; Vardhan and Shukla, 2018). The peak at 3082 cm−1 corresponds to ring hydrogen's C–H stretching vibrations. The absorption bands around 2800 and 3000 are due to C-H stretch aliphatic vibrations. In special, the peak at 2854 cm-1 corresponds to an aromatic C–H. The absorption at 1643 cm-1 is related to C-C stretching. The two peaks at 1492 and 1600 cm−1 are assigned to aromatic C=C stretching. In the region from 1300 to 1380 cm−1, the peak observed at 1376 cm−1 corresponds to aromatic CH2 bending. In addition, the peaks located around 650 to 1250 cm−1 correspond to C–H deformation.

Figure 1(d) shows the infrared absorption spectra of the recovery D-limonene, which is similar to the spectrum for the raw D-limonene, before the dissolution process. The broad absorption band at approximately 3300 cm-1 is related to hydroxyl group stretching. The intense absorptions that appear around 3000–2850 cm−1 are attributed to the C–H, methylene bond, vibration. The peak at 1645 cm−1 is related to the stretching of the double bonds, C=C, which is present in the endocyclic and exocyclic positions of the D-limonene structure. The peaks between 1480–1350 cm−1 are associated with the deformation of the C–H bond belonging to the alkyl groups. The peak at 1050 cm−1 is attributed to the bending of =C-H bonds, and at 887 cm−1 is attributed to =C−H. In the region between 3200 and 3500 cm-1, the broadening peak is attributed to O-H vibration, probably due to water in the D-limonene solution. It is important to mention that, based on our experiments and reported before (Gil-Jasso et al., 2019), the recovered D-limonene can be used again in other dissolution processes, with the same efficiency as the raw D-limonene.

When dissolved polystyrene and WCSO are mixed, in equal proportions of volume, a polymer solid precipitated. The mass formed agglomerated at the bottom of the Becker was easily removed and cleaned. Figure 2 illustrates the reaction sequence, showing the light amber colored polymer mass and the reaction residue material. Sarmiento and co-workers have also been using waste cocking oil in recycling EPS. However, in this case, the oil is used as a solvent where the EPS is dissolved, at 165o C, and then recovered (Sarmiento et al., 2016). Contrary to this, the current work presents a process carried out at room temperature. In this context, recycling dissolved EPS with WCSO may reduce the energy requirement and is cost-effective.

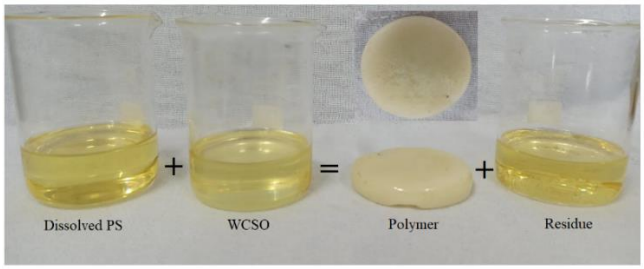


Fig. 2. Schematic representation of the PS recovery, showing the light amber colored polymer mass and the reaction residue or unreacted material.

After the precipitation of PS (Figure 1), it is in the pasty form because a small amount of D-limonene continues to dissolve PS agglomerates. When this mixture is placed in contact with WCSO, it dissolves the residual D-limonene, releasing the PS, which becomes a solid aggregate. Therefore, the WCSO acts as an antisolvent when it is added to the D-limonene/PS mixture. We observed that the D-limonene exhibits an acceptable solubility in WSCO at moderated working conditions, while solid PS are generally insoluble in WSCO. Thus, the addition of frying soybean oil induces the supersaturation and precipitation of the polymer due to the dissolution of limonene in WSCO. In fact, the use for polymer phase separation has been extensively reported in the literature, especially using carbon dioxide as a non-solvent and numerous techniques has been developed in the last years (Campardelli et al., 2014; Gutiérrez et al., 2016). Based on this, WCSO stands out as a potential material to be used as a liquid antisolvent in other polymer production processes.

To confirm the composition of the recycled polymer and the residue, Fourier transform infrared spectroscopy measurements were performed (Fig. 3). The FTIR spectrum for the recovered polymer, figure 3(a), is identical to that showed in the Figure 2(c), indicating that the product is polystyrene. An extra peak can be observed at approximately 1745 cm-1 (see the red arrow) which is related to the polymerization residue contamination. Contamination is basically a mixture of with a small fraction of dissolved D-limonene. There is approximately 2.0 %w of superficial WCSO+D-limonene in the final material. Contamination can be removed by simply squeezing the material between sheets of paper. These results indicate that the final polymer can be further applied, as there is no significant surface fat content. However, to do so, it is necessary to measure several properties in the final PS, such as mechanics, chemical and physics. As expected, the FTIR spectrum illustrated in Figure 3(b) is quite similar to reported for the waste cooking soybean oil (WCSO) (Laskar et al., 2018; Rafati et al., 2018). The absorption peaks at 2921, 2854, 1745, and 1155 cm-1 are due to triglycerides, which are basic components in cooking oils (Laskar et al., 2018; Rafati et al., 2018). The absorptions at 2921 and 2854 cm-1 are related to asymmetrical stretching vibrations of C-H and the absorption at 1745, and 1155 cm-1 are due to C=O bond stretching and -CH2-, respectively. As can be seen, this spectrum contains a significant amount of peaks, mainly in the region between 1750 and 700 cm-1. For a better understanding, the functional groups and modes of vibration associated with the absorptions are illustrated in the Fig 3(b) (Laskar et al., 2018; Rafati et al., 2018). Back to figure 2, it is possible to observe that the soybean oil change its color after the polymer solidification process, which suggest an oil oxidation during this process (Komoda et al., 1966).

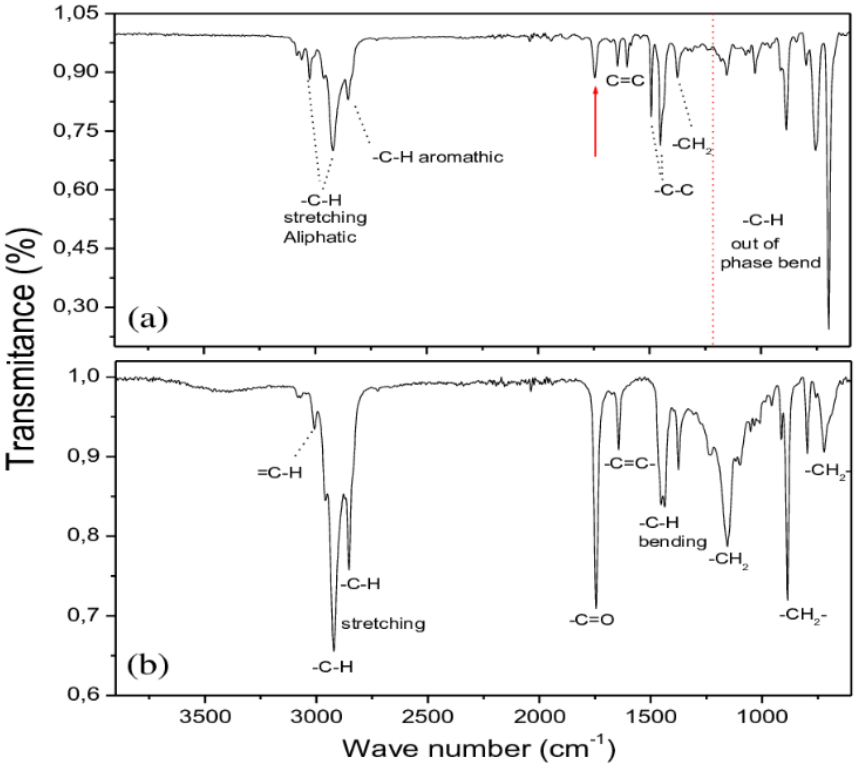


Fig. 3. a) FTIR spectra of the obtained polymer and, b) FTIR spectra of the polymerization residue.

Focus on the polymeric material, Figure 4 shows the X-ray diffraction (XRD) for the recovered EPS. The XRD pattern shows a prominent peak observed at 2θ = 19.65°, suggesting that the polystyrene has certain crystallinity (JunFei et al., 2010; Wankasi and Dikio, 2014). The recovered polystyrene has a roughness surface as shown by the SEM image in the inset of Figure 4. This surface morphology can be the feature of non-controlled solidification processes. As well know, when a polymer solidifies, it may retain an amorphous structure, crystallize completely, or become semi crystalline.

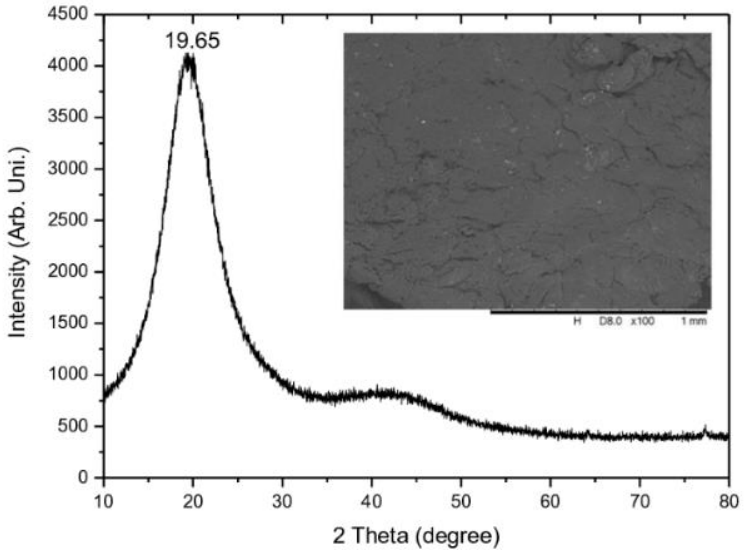


Fig. 4. XRD pattern of recovery polystyrene. The inset shows SEM image of the polymer.

Figure 5 shows the TGA curves of recovery EPS. Recovered EPS shows Ti ~ 163oC, the temperature corresponding to initial weight loss (volatiles components loss). Further, it shows a decomposition temperature at 50% weight loss of T50 ~ 412 oC (related to degradation of PS chains). Finally, recovered EPS shows maximum decomposition temperatures of Tmax ~ 576 oC (total degradation). The loss weight before Ti is due to the residual decomposition. The incomplete degradation of the solid PS after ~ 450oC suggests the existence impurities, probably of WCSO impurities comes from the frying processes.

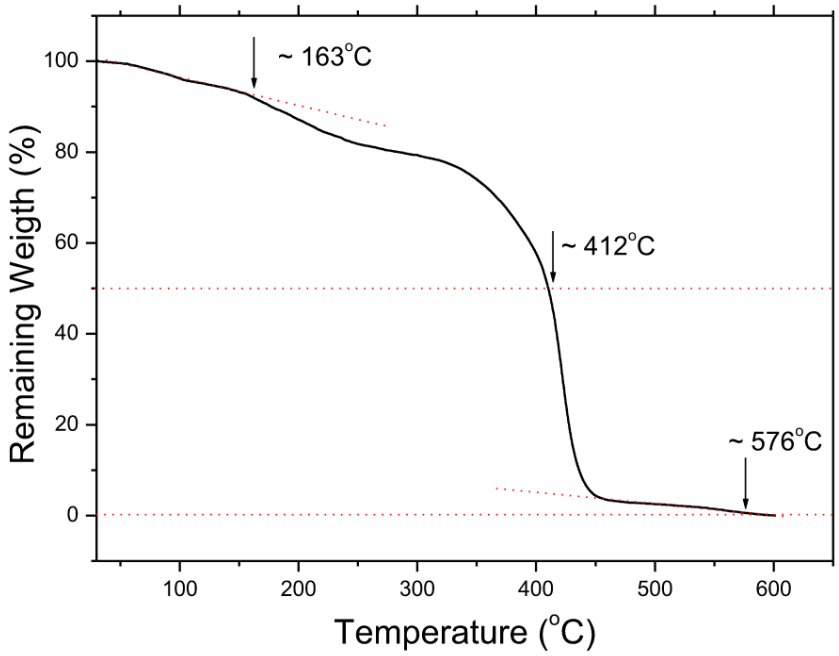


Fig. 5. TGA of the recovered polystyrene.

The results, showed in this section, shows that it is possible to recovery PS from waste EPS but further works are necessary objective to define the properties and applications of the recycle polymer. Additionally, WCSO stands out as a potential material to be used as a liquid antisolvent in other polymer production processes.

Conclusion

This work stems from the idea that waste expanded polystyrene can dissolve in D-limonene and be recovered, using waste cooking soybean Oil (WCSO), to produces a new polystyrene-based product. The obtained polymer was soft and malleable, making it a possible candidate of renewable additive in diverse applications. Additionally, solvent recovery was

carried out to make the process more sustainable. The environmentally friendly process, carried out entirely at room temperature, opens a new sustainable and green approach toward the recycling and recovery of polystyrene waste. Avoiding any heating step, which can be high cost, this process also meets the financial challenges inherent in any industry applications.

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