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Study of the solubility and stability of polystyrene wastes in a dissolution recycling process

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ABSTRACT

Dissolution with suitable solvents is one of the cheapest and more efficient processes for polystyrene waste management. In this work the solubility of polystyrene foams in several solvents benzene, toluene, xylene, tetrahydrofuran, chloroform, 1,3-butanediol, 2-butanol, linalool, geraniol, *d*-limonene, *p*-cymene, terpinene, phellandrene, terpineol, menthol, eucalyptol, cinnamaldheyde, nitrobenzene, *N*,*N*-dimethylformamide and water has been determined.

Experimental results have shown that to develop a "green process" the constituents of essential oils, *d*-limonene, *p*-cymene, terpinene, phellandrene, are the most appropriate solvents. The action of these solvent does not produce any degradation of polymer chains. The solubility of the polymer in the mentioned solvents at different temperatures has been investigated. The solvent can be easily recycled by distillation.

1. Introduction

The amount of waste of polystyrene (PS) foams is increasing in last years due to the increase of their use in isolation, protecting, storing and serving many different food products. Continuous accumulation of waste plastics leads to serious problems all over the world (Seung-Soo and Seungdo, 2004). Recycling of this waste has recently received significant attention all over the world due to the changes in both regulatory and environmental issues. Increasing landfill costs and decreasing landfill space are forcing consideration of alternative options for the disposal of polystyrene materials.

The two main alternatives for treating polymer wastes are energy recycling, where wastes are incinerated, and mechanical recycling (Garforth et al., 2004). Environmental argumentations such as toxic emissions are building up a public resistance against incineration process (British Plastic Federation). Mechanical recycling (the conversion of "scrap" polymer into new products) is a popular recovery path for manufacturers and is carried out on single-polymer waste streams. Anyway, the market for recycled products can only be found if the quality is close to that of the original. Unfortunately the process of recovery of these wastes are often more expensive than virgin plastic (Brandrup, 1996; Lee, 1995). On the other hand, these recycling processes have other economic problems related to the transportation cost. The low density of polystyrene waste and therefore, the transport of the big volume residues make economically unfeasible these processes.

Considering this fact, other processing schemes should be explored in order to reduce the cost of the two recycling processes mentioned before. It could be accomplished by dissolution with suitable solvents in order to get a volume reduction of more than 100 times (without degradation of polymer chains), since, if dissolution is developed in the source of residue production the transportation is more efficient than in the conventional recycling system.

Thus, PS foams dissolution behaviour in different solvents plays and important role in polystyrene recycling, because it is an attractive alternative to incineration and mechanical recycling since, is the cheapest and one of the most efficient ways for PS foam recycling.

It is well known that some aromatic compounds are good solvents of PS foams (Karaduman et al., 2002). Certain foods items have been shown to be incompatible with the expanded polystyrene used for the manufacture of food containers, since, the dissolution of polystyrene by certain essential oils are produced (Monte and Landau-West, 1982). The treatment of PS with *d*-limonene has been reported as an attractive alternative for PS solubilisation (Nocuchi et al., 1998). However, the earlier works do not show the solubility values or the chain degradation during the dissolution process.

The aim of this work is to develop a process for the recycling of extruded polystyrene in two steps. First, a polystyrene dissolution with suitable solvents in the same place where the residue is produced. Second, a solvent elimination step by vacuum distillation in a recycling plant. Accordingly, in this paper will be evaluated the solubility of polystyrene in several solvents. The degree of polymer chain degradation that the solubilization process produces, as well

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as the degradation produced during the solvent elimination step has been analyzed in order to select the best solvent.

2. Experimental

2.1. Materials

Extruded polystyrene (XPS) waste composition is shown in Table 1. The XPS was supplied by Tecnove Fiberglass and it is used in the insulation of truck bodies for refrigerated truck. The XPS has a polydispersity of 1.7, a number average molecular weight (M_n) of 54024 and its decomposition temperature is 421 °C.

The solvents tested in this study were benzene, toluene, xylene, tetrahydrofuran, chloroform, 1,3-butanediol, 2-butanol, linalool, geraniol, *d*-limonene, terpinene, phellandrene, cinnamaldheyde, menthol, terpineol, eucalyptol, nitrobenzene, *N*,*N*-dimethylformamide, which were supplied by Panreac, *p*-cymene by Aldrich and water used in this investigation was milli-Q (Gradient A10, Elix 5, Millipore).

2.2. Solubility determination

The solubility of XPS foams in the solvents has been determined gravimetrically as the minimum solvent weight necessary to completely dissolve a weighted sample of XPS foam. The solubility was expressed as the mass of XPS and the solvent volume ratio expressed in mL.

The accuracy and reproducibility of the experimental procedure has been determined comparing the results from three independent runs carried out under identical conditions: Solvent: *d*-limonene, Temperature: 25 °C. In these experiments the solubilities were similar (0.26, 0.25 and 0.26 g/mL) indicating that the repeatability of the experimental procedure was good. Nevertheless, to minimize experimental errors each run was replicated twice.

2.3. Molecular weight and Polydispersity index

The number average molecular weight and the polydispersity analyses were performed by GPC on a Waters Associates Liquid Chromatography system (Waters 717, Rydalmere) equipped with a differential refractometer and two μ-Styragel columns (HR1 and HR4). The mobile phase was tetrahydrofuran (THF) at a flow rate of 1 ml/min. Polymer was dissolved in THF and filtered through 0.45 μm filter before analysis. The system was calibrated with narrow disperse polystyrene standards and molecular weights are reported as polystyrene equivalents. The corresponding uncertainties associated with the determination of molecular weight and polydispersity measures are lower than 2%.

2.4. Decomposition temperature

The decomposition temperature was determined by thermogravimetric analysis (TA-DSC Q 100), where the weight loss due to the volatilization of the degradation products is monitored as a function of temperature. The sample weight varies from 10 to 15 mg. Samples are heated from the room temperature to 500 °C at a heating rate of 15 °C/min. The decomposition temperature showed in the experimental section is the temperature of the maximum in the weight loss rate.

Table 1
Extruded polystyrene (XPS) waste.

Composition	% w/w
XPS	90–93
Flame retardant (HBCD)	2–3
Nucleating agent (Talc)	0–1
Ethyl chloride	0–2
Pigments	0.2–0.3

2.5. Vacuum distillation of solvents

Instrument Normalab Analis P 1280 was used to recover the solvent. The pressure above the solution to be distilled is reduced to less than 1 mmHg causing the evaporation of the solvent, the most volatile compound. Vacuum distillation was used with heating the solution (less than 30 °C). Distilling under vacuum allows that solvents with very high boiling point can be distilled at lower temperatures avoiding the thermal degradation of the polymer.

A volume of 100 ml of saturated solution is put into the flask. Under a pressure of 1 mmHg the solution is heated beneath 30 °C in order to avoid thermal degradation of the XPS chains. A mixing of methanol and dry-ice is used like refrigeration system. Vacuum distillation allows the total recovery of the solvent.

Nevertheless, due to the pigments present in XPS the solvent becomes slightly colored. On the other hand, during the recycling process some oxidation of solvent can happen. Therefore, to prevent high level contamination in solvents after a suitable number of cycles the solvent should be purified by distillation

3. Results and discussion

In this work has been proposed a two stage process for the recycling of polystyrene. The fists stage consists in the dissolution of polystyrene with suitable solvents. A good solvent for the recycling of extruded polystyrene should have high dissolution ability and high volatility that will allow its removal with minimum temperature chain degradation. Furthermore, low cost, low toxicity and easy availability are also appreciated properties. In order to select the most suitable solvent, a large number of compounds were tested (with different chemical and physical characteristics). The influence of the solvent on the degradation of the polymer chains during the dissolution process has been also analyzed. The second step consists in the elimination of the solvents selected by vacuum distillation. The effect of this process on the polymer degradation has been also studied.

3.1. Preselection of solvents: Initial screening

The first aspect considered for the selection of the most suitable solvent for XPS recovery by dissolution and further distillation is the solubility ability for XPS. A theoretical initial approach based on Hildebrand theory was done in order to establish initially the best candidates. After that, the values of the experimental test are compared with theory. Table 2 shows the solubility values obtained in the experimental test together with the corresponding Hildebrand parameter of polymer (Brandrup et al., 2003) for each solvent (Barton Allan, 1983; Buckley-Smith, 2006; Miller et al., 1998). It can be seen that several of the solvents tested, some of them with similar Hildebrand solubility parameters to that of XPS, have not the capacity of dissolving polystyrene.

According with the theory of Hildebrand, polymers will be soluble in solvents whose solubility parameters are not too different from their own (Hildebrand, 1924).

For most of the solvents the Hildebrand parameter has been determined and they are reported in previous works (Barton Allan, 1983; Buckley-Smith, 2006; Miller et al., 1998). Nevertheless, for a polymer this parameter (called cohesion parameter (Brandrup, 1996)) depends on the chemical structure of its basic building blocks and in a lower grade of other factors as the molecular

Table 2
Solubility of XPS at 25 °C and Hildebrand and Hansen solubility parameters.

Solvent	XPS solubility (g/ml)	δ_T (MPa)1/2	δ_d (MPa1/2)	δ_p (MPa1/2)	δ_h (MPa1/2)
Polyestyrene	–	22.45[a]	21.3[a]	5.8[a]	4.3[a]
Benzene	0.68	18.51[b]	18.4[b]	0.0[b]	2.0[b]
Toluene	0.60	18.16[b]	18.0[b]	1.4[b]	2.0[b]
Xylene	0.40	18.10[c]	17.8[c]	1.0[c]	3.1[c]
Tetrahydrofuran	0.96	19.46[b]	16.8[b]	5.7[b]	8.0[b]
Chloroform	1.28	18.95[b]	17.8[d]	3.1[d]	5.7[d]
1,3-Butanediol	0.00	37.31[b]	10.0[f]	21.5[f]	28.8[f]
2-Butanol	0.00	23.20[b]	15.8[f]	5.7[f]	14.5[f]
Linalool	0.00	20.26[c]	16.3[c]	4.4[c]	11.2[c]
D-limonene	0.26	16.38[b]	16.4[f]	0.2[f]	0.2[f]
p-Cymene	0.30	16.51[e]	16.5[g]	0.6[g]	0.0[g]
Terpineol	0.00	19.01[e]	13.9[g]	8.0[g]	10.2[g]
Eucalyptol	0.10	16.72[c]	15.9[c]	3.9[c]	3.4[c]
Water	0.00	47.90[b]	15.5[b]	16.0[b]	42.4[b]
Nitrobenzene	0.13	22.15[e]	20.0[e]	8.6[e]	4.1[e]
N,*N*-dimethylformamide	0.31	24.86[e]	17.4[d]	13.7[d]	11.3[d]

[a] Brandrup et al. (2003).
[b] Barton Allan (1983).
[c] Buckley-Smith (2006).
[d] Miller et al. (1998).
[e] Hildebrand (1924).
[f] Hansen (2004a).
[g] Hansen (2004b).

weight and the degree of crosslinking. For this reason, there are some discrepancies between the values of solubility parameter of polystyrene taken from different literature sources (Brandrup et al., 2003; Hansen, 2004a). In this work, we will be considered the values given in Brandrup et al. (2003). This value have been selected since is the value that fits better with experimental solubility data of PS obtained in this work.

According with Hildebrand theory, as expected, the XPS is not dissolved by solvents with parameters very different (1,3-butanediol and water) but is soluble in some of the solvents with similar parameters (benzene, toluene, xylene, THF, chloroform, *d*-limonene, cymene, eucalyptol, nitrobenzene, *N*,*N*-dimethylformamide). Nevertheless, some of the solvents with similar Hildebrand parameters to XPS do not dissolve polystyrene (2-butanol, linalool, terpineol). Finally, there are solvents which similar solubility parameters that nevertheless exhibit quite different solubility capacity (i.e. eucalyptol and limonene). Consequently, this parameter alone is not able to justify entirely the trend behaviour of the XPS in the different solvents.

To explain this fact, it is needed to consider that the solubility parameter represents the total cohesive energy densities which result from additive effects of several types of contributions: non polar bonds, dipole–dipole bonds and hydrogen bonds. Therefore, some solvents with similar total solubility parameter exhibit different behaviour since they have different individual contributions.

Table 3
Interaction radius and RED number.

Solvent	R_a	RED = R_a/R_o
Benzene	8.52	0.67
Toluene	8.26	0.65
Xylene	8.57	0.67
Tetrahydrofuran	9.73	0.77
Chloroform	7.63	0.60
1,3-Butanediol	36.84	2.90
2-Butanol	15.07	1.19
Linalool	12.23	0.96
D-limonene	12.04	0.95
p-Cymene	11.73	0.92
Terpineol	16.08	1.27
Eucalyptol	11.00	0.87
Water	41.11	3.24
Nitrobenzene	3.83	0.30
N,*N*-dimethylformamide	13.12	1.03

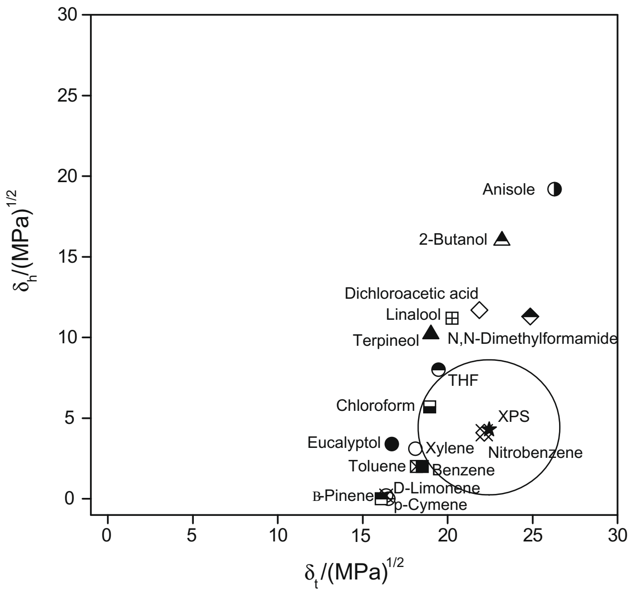

Fig. 1. Solubility of XPS in several solvents. Hoernschemeyer diagram.

Table 4
Number average molecular weight, Polydispersity, and decomposition temperature of XPS in solvents.

Solvent	M_n	Polidispersity	T_C (°C)
Benzene	54112	1.63	419
Toluene	52053	1.75	420
Xylene	53815	1.71	416
Tetrahydrofuran	53990	1.68	418
Chloroform	54052	1.63	418
D-limonene	54129	1.76	421
p-Cymene	50025	1.80	414
Eucalyptol	53723	1.72	419
Nitrobenzene	52965	1.70	414
N,*N*-dimethylformamide	54015	1.69	417

Table 5
Solubility, number average molecular weight, Polydispersity, and decomposition temperature of XPS in in natural solvents.

Solvent	Solubility (g/ml)	M_n	Polydispersity	T_C (°C)
XPS non treated	–	54024	1.70	421
Geraniol	Not soluble	–	–	–
Limonene	0.26	54129	1.76	419
Terpinene	0.25	52180	1.75	415
Cymene	0.31	50025	1.80	415
Phellandrene	0.28	54152	1.70	417
Menthol	Not soluble	–	–	–
Cinnamaldehyde	0.17	52858	1.73	417

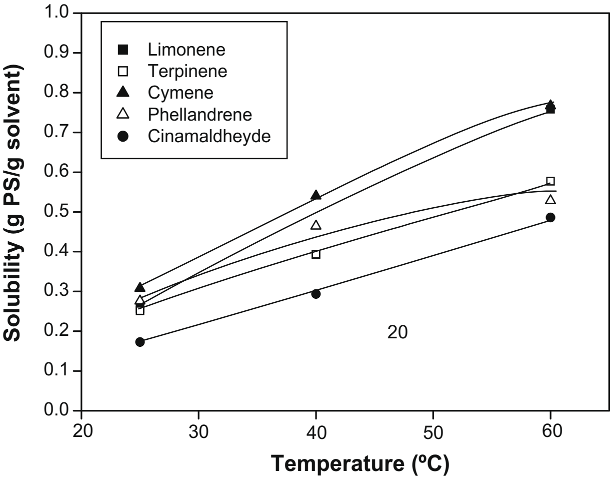

Fig. 2. Effect of temperature on solubility.

Hansen revised the Hildebrand theory and introduced a division of the Hildebrand solubility parameter, spliting the previous unique parameter. δ_d, δ_p and δ_h qualitatively describe the nonpolar interactions (D), the dipolar interactions (P) and the hydrogen bonding interactions (H) (Hansen, 2004a,b). These parameters are summarized in Table 2 for the solvents studied. This theory establishes that a polymer is probably soluble in a solvent if the Hansen parameters for the solvent are within the solubility sphere of the polymer (Hansen, 2004b). To check this hypothesis it is necessary calculate whether the distance of the solvent from the center of the polymer solubility sphere, R_a, is less than the radius of interaction for the polymer, R_o equal at 12.7 in this case (Hildebrand, 1924). R_a is calculated by Eq. (1):

$$R_a = \sqrt{4 \cdot (\delta_d s - \delta_d p)^2 + (\delta_p s - \delta_p p)^2 + (\delta_h s - \delta_h p)^2} \quad (1)$$

where R_a is the distance between the point of the solvent and the center of the solubility sphere, $\delta_x s$ is the Hansen component parameter for solvent and $\delta_x p$ is the Hansen component parameter for polymer. Whit R_a and R_o it can be calculated a parameter widely used to correlate measured solubilities, the RED affinity number (R_a/R_o). If $R_a < R_o$, RED number lower than 1, is a high likelihood of the solvent dissolving the polymer while as the RED number increases over 1 the solvent ability decreases (Hansen, 2004a).

Table 3 shows that, except the linalool, all the solvents fulfil the RED number criteria, although the eucalyptol and nitrobenzene should be good solvent due to a low value of RED number.

To explain these discrepancies, it should be analyzed the Hansen parameters values. As observed, the δ_h value is the main difference between these solvents (linalool, eucalyptol and nitrobenzene) and the rest. These demonstrate that the capacity to form hydrogen bonds is negative in the process of dissolution of XPS foam.

To clarify this fact, the diagram developed by Hoernschemeyer which plot the parameters by individual pairs (δ_h–δ_t) (Hoernschemeyer, 1974) has been used and it is shown in Fig. 1. In this diagram the solubility parameter component of the polymer is placed in the center of the circle of radius δ_h. If the solvent parameter remains inside or near the circle the compound is considered as good solvent. Whereas, if the solvents parameter remains outside the circle may be assumed that the solvents not dissolve the polymer (Güner, 2004). Fig. 1 shows that the solvents that present low δ_h values are the most appropriate.

As rule of thumb aromatic and terpenic solvents with low tendency to form hydrogen bonds are the most suitable solvent for the recycling of XPS.

It has been also analyzed if the type of solvent has some influence on the degradation of the polymer chains during the dissolution process. This degradation generally involves changes into the molecular weight distribution of the polymer and in the decomposition temperature. These changes can give an idea of the degradation level that the treatment with the solvent generates into the initial characteristics of the polymer.

Table 4 shows the polydispersity, number average molecular weight, and decomposition temperature of XPS after being dissolved with the different solvents. It can be observed that in all cases the polydispersity and NAMW remain practically unchanged after treatment with the assayed solvents. The decomposition temperature shows the same trend. Therefore, considering the results obtained, it is possible to affirm that polymer degradation does not occur during the dissolution process.

3.2. Solubility results

The solvents that allow the solubility of higher amounts of XPS are benzene, toluene, xylene, chloroform and tetrahydrofuran. Nevertheless, these solvents are not in good relation with "green chemistry" and therefore, they should not be used for the recycling process. In this work, in order to propose an environmental friendly technology would be more appropriate to propose a natural solvent as the terpenic solvents (like limonene and cymene) that also show high capacity to dissolve the polystyrene. These compounds are an enormous class of natural products spanning well over 30,000 members. They have used throughout history for a broad variety of purposes including perfume, medicine, and flavouring.

Another set of experiments testing a series of terpenic compounds like geraniol, limonene, terpinene, cymene, phellandrene, menthol, cinnamaldehyde were performed at 25 °C. The values obtained are shown in Table 5. As expected, the solvents with polar groups in their structure do not dissolve the XPS (geraniol and menthol) or exhibit a lower solubility potential (cinnamaldehyde). Limonene, terpinene, cymene and phellandrene exhibit similar solubility values being good alternatives to carry out the recycling process. As the solubility capacity is not very different, the best solvent will be chosen in each region depending on the commercial availability and price of these natural solvents in that part of the world.

The degree of polymer chain degradation of the recovered XPS has been also measured on this case. Table 5 shows the polydispersity index, number average molecular weight and decomposition temperature of XPS dissolved with the different solvents. As in the previous case, any change is observed in the polymer chain.

3.3. Effect of temperature on solubility

This group of experiments was carried out in order to determine the temperature at which the dissolution should be performed. The levels of temperatures studied were 25 °C, 40 °C and 60 °C. Higher

Table 6
Effect of temperature on Number average molecular weight, Polydispersity, and decomposition temperature.

Temperature	25 °C			40 °C			60 °C		
Solvent	M_n	P^a	T_C (°C)	M_n	P^a	T_C (°C)	M_n	P^a	T_C (°C)
Limonene	54129	1.76	419	50010	1.82	418	50021	1.81	419
Terpinene	52180	1.75	415	52180	1.75	417	53958	1.75	417
Cymene	50025	1.80	415	46695	1.90	415	45893	1.95	416
Phellandrene	54152	1.70	414	53283	1.72	414	48049	1.84	420
Cinnamaldehyde	52858	1.73	417	51582	1.76	421	48190	1.84	420

[a] Polydispersity.

Table 7
Number average molecular weight, Polydispersity and decomposition temperature of recycled XPS.

Solvent	M_n	Polydispersity	T_C (°C)
Limonene	52674	1.74	421
Terpinene	52935	1.75	421
Cymene	51069	1.79	421
Phellandrene	53805	1.72	421
Cinnamaldehyde	51423	1.77	424

temperatures were not tested to prevent polymer chain degradation. Results obtained are shown in Fig. 2. It can be observed that, for all solvents, the solubility increase with increasing temperature.

Table 6 shows the results of polydispersity, number average molecular weight, and decomposition temperature at different temperatures. As consequence of the soft heating an incipient polymer thermal degradation could be produced. This fact lead to that the polymer decomposition can occur at temperatures much lower than those at which initially occurs. It can be observed that, although the decomposition temperature practically does not vary, the polydispersity slightly increase with increasing temperatures and number average molecular weight decrease, probably due to the beginning of degradation chains. Considering these results it may be stated that temperatures higher than 60 °C are not interesting because, although solubility are higher at higher temperature, it is produced a degradation of polymer chains.

3.4. Recovery of solvent

These experiments have been developed with the solvents considered as best candidates to recycling process. In the last sections, it has been shown that the solvents most appropriate to XPS recycling must have two important properties. They should have high capacity to dissolve XPS, and produce minimum degradation of polymer chain during the dissolution process. As regards (Table 5), the solvent that fulfills both properties are limonene, terpinene, cymene, phellandrene, cinnamaldheyde.

Results obtained with these solvents in the vacuum distillation are shown in Table 7. It can be observed that the process does not produce a noticeable degradation of polymer chains, due to distillation was carried out at low temperatures.

4. Conclusions

Several solvents with different characteristic have been tested as dissolution agents for XPS. The solubility of polymer in solvents is affected by the polarity. The polymer tendency is to dissolve better in the non polar solvents which are chemically and physically most similar to the XPS, although the polar solvent also can be used to the recycling process if they do not have pronounced tendency to form hydrogen bonds.

Limonene, terpinene, cymene, phellandrene and cinnamaldheyde exhibit suitable properties for XPS recycling. These compounds are constituent of essential oil and, therefore, natural solvents. The solubility of XPS in these solvents increases as the temperature so does but at the higher temperature used in this study a slightly degradation of polymer chains are produced. In each country or region the best choice will be variable depending on the commercial availability and price of these natural solvents in that part of the world. The solvent can be easily recycled by distillation obtaining a very good quality recycled polymer.

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