A Recycling Alternative for Expanded Polystyrene Residues Using Natural Esters

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Research Article

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Abstract

The objective of this research is to provide a new recycling method for one of the most consumed plastics today, since it is used for the manufacture of a wide variety of industrial products, which leads to an environmental problem caused by incorrect handling and final disposal.

The dissolution of expanded polystyrene waste (WEP) was evaluated by using natural esters for its post treatment and recovery. The use of omega-3 as a natural solvent creates an opportunity to take advantage of natural biomass, since it can be obtained from the residues from the fishing activity, this being an economic advantage for obtaining raw material and also friendly with the environment.

For the development of this research, expanded polystyrene containers were used, as well as omega-3 and glyceryl tributyrate as natural solvents and ethyl butyrate as synthetic solvent, methanol and isopropanol for recovery and cleaning of the polystyrene. The characterization of the recovered material was carried out with thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM) and infrared spectroscopy (FTIR) techniques. The experimental data obtained indicated that the use of these esters is a good alternative for the recycling of expanded polystyrene.

1. Introduction

In the last century, petroleum-based polymers gained importance in humans' lives to cover different needs of end-consumer products that are essential to modern life [ha et al., 2021]. The use of plastic is growing rapidly, in 1950 were produced 1.65 million tons worldwide and in 2018 global plastics production almost reached 360 million tons [25]. At current growth rates, the accumulation of plastics waste in landfills and/or in the natural environment is projected to reach nearly 12,000 Mt globally by 2050 [10]. Plastic has many promising characteristics such as low density, durability, low cost and easily moldable [29]. The plastic demand is mainly in packing (39.9%), building/construction (19.8%), automotive industry (9.9%) and the rest includes electronics, furniture, etc [25]. The excessive use of plastic products has generated enormous quantities of waste, which has become a global issue [28].

Current methods of plastic waste management include landfilling, incineration, mechanical recycling, dissolution, and chemical recycling. Globally, only 18% of plastics waste is recycled, and 24% are incinerated. The remaining 58% is either landfilled or enters the natural environment, where plastics accumulate and persist for a long period [10]. Plastic solid wastes in landfills contaminate the groundwater with toxic chemicals [29]. This landfilling of plastic waste enters the marine environment, approximately 12 million tons of plastic waste was found to move into the seas in 2010 [12]. The incineration of plastic waste contributing to amounts of toxic pollutants such as CO, NO_x, SO_x, particles and polycyclic aromatic hydrocarbons [31]. Mechanical recycling is used to reuse the plastic solid waste to form a product with the same inherent characteristics, this method has a smaller environmental impact. Chemical recycling converts plastic waste into fuels and chemical feedstocks.

Dissolution methods consist in three steps, removal of impurities, dissolution of the polymer, and reprecipitation, this method is the most environmentally friendly and profitable [33].

Expanded polystyrene is made from small spheres of polystyrene (from crude oil) containing an expansion agent (e.g., pentane C_5H_{12}) that expands by heating with water vapor [24]. Expanded polystyrene is made of 98% air and 2% polystyrene. Due to chemical inertness, thermal resistance and insulation, mechanical strength and low-cost manufacturing, it is one of the most widespread polymers around the world. However, its non-degradability and low-density lead to huge white pollution. In 2015, 6200 kt of expanded polystyrene was produced, and just around 40% was reused. WEP waste recycled is managed by two main approaches incinerated and mechanical recycling. Incineration of polystyrene can produce hazardous chemicals. Mechanical recycling is more environmentally friendly but the large volume of WEP results in transportation problems and higher costs [33], a solution to this problem is dissolving the WEP residues in organic solvent. The reduction of the volume is 100 times approximately [18]. The WEP materials are dissolved in organic solvents and then recovered by reprecipitation. Organic solvents are toxic and produce additional noxious waste, some examples that have been used in previous works are toluene, xylene, benzene, chloroform, acetone, cyclohexane, butyl acetate, ethyl acetate, and methyl ethyl ketone. [Cella et al., 2018]. An environmentally friendly alternative consists in the dissolution of WEP waste in d-limonene. D-limonene (4-isopropenyl-1-methylcyclohexene) is a natural terpene found in citrus peels and is bio-degradable. Limonene dissolves the same amounts of WEP as some organic solvents but has the problems of low yield and high cost of extraction. Among of the best natural essential oils are star anise, chamomile, thyme and eucalyptus stand out for having better dissolution times and recovery performance of WEP residues [11]. Fatty acids or also called "omega" is a hydrocarbon chain with a carboxylic group and a methyl group at each end of the chain". [De Berrazueta and De Berrazueta, 2007]. The omega-3 fatty acids are polyunsaturated and they can be found in food in three main compounds as eicosapentaenoic acid ($C_{20}:5n-3$; EPA), docosapentaenoic acid ($C_{22}:5n-3$, DPA) and docosahexaenoic acid ($C_{22}:6n-3$, DHA) [Rubio-Rodríguez et al., 2010]. EPA and DHA are in marine fish that lives in cold water such as tuna, salmon, sardines among other varieties. The α -linolenic acid (ALA) can be obtained from vegetable oils, chia, walnuts, and olives.

It was observed that some commercial fish oils dissolved polystyrene, this is because the fatty acids were in ethyl esters form. In this work, fish oil from a local drug store was tested, which contained ethyl ester. This method is low cost and environmentally friendly. Also was used glyceryl tributyrates and ethyl butyrate to observe and compare synthetic and natural esters in the WEP waste dissolution performance.

2. Experimental

2.1 Materials

Ethyl ester fish oil commercial brand PharmaLife Natura ® Fish Oil 1000mg, was used without additional treatment. Methanol, isopropanol, glyceryl tributyrates and ethyl butyrate were purchased from Sigma

Aldrich Company and all of them were used without further purification. The WEP waste collected was waste packaging.

2.2 Dissolution process

Weight percent solutions of 10–100% of WEP waste with respect to 1g of ester were used. As soon as the ester was poured into the WEP waste, it was constantly stirred. Until complete dissolution, a clear solution was obtained, and the dissolution time was counted. The experiments were carried out at room temperature.

2.3 Recovery of polystyrene

The clear solution was dissolved in methanol and the polystyrene precipitated. Then the precipitate was filtered and dried on a filter paper. The precipitate was washed four times with isopropanol. To remove ester residues from the recovered WEP, it was left in isopropanol overnight on a mechanical shaker. Finally, the WEP was allowed to dry and stored.

2.4 Calculation of apparent activation energy E_a

The apparent activation energy of degradation can be determined by methods or kinetic models based on the analysis of the data obtained from the thermograms from the TGA analysis. In this case, the Friedman differential method was applied to calculate the apparent activation energies of degradation of untreated WEP and recovered WEP. The calculations were carried out with the help of Origin 8 software.

2.4.1 Friedman's kinetic model

The results obtained from the TGA analysis can be expressed as a function of the conversion or degree of advance α , according to the equation:

$$\alpha = (W_0 - W) / (W_0 - W_a) \quad (1)$$

Where W_0 corresponds to the initial mass of the sample, W is the mass of the sample at time t and W_a is the final mass of the sample.

Kinetic studies use the basic equation of velocity:

$$d\alpha/dt = k f(\alpha) \quad (2)$$

The independent temperature conversion function $f(\alpha)$ is represented by equation (3):

$$f(\alpha) = (1 - \alpha)\exp(n) \quad (3)$$

The Arrhenius equation is expressed in equation (4):

$$k = A\exp(-E_a/RT) \quad (4)$$

Substituting equations (3) and (4) in (2):

$$d\alpha/dt = A \exp(-E_a/RT) ((1-\alpha) \exp(n)) \quad (5)$$

The heating rate $\beta = T/t$ (K/min) is introduced to equation (5), obtaining equation (6):

$$dt = dT/\beta \quad (6)$$

Substituting in equation (5), the following separable differential equation is obtained:

$$d\alpha/dt = (A/\beta) \exp(-E_a/RT) ((1-\alpha) \exp(n)) \quad (7)$$

Applying a linearization to equation (7) we obtain the equation that allows us to calculate the kinetic parameters based on the data from the thermogravimetric analysis (see equation 8). Its graphical representation is shown in equation (9) (Arrieta et al., 2013).

$$\ln(d\alpha/dt) = \ln(A/\beta) + n \ln(1-\alpha) - (E_a/RT) \quad (8)$$

$$\ln(d\alpha/dt) = f(1/T) \quad (9)$$

The $\ln(d\alpha/dT)$ was plotted against the inverse of the temperature ($1/T$), with the slope $m = -E_a/RT$, where m is the slope, R is the constant of ideal gases ($R = 8.314 \text{ J/Kmol}$), T is the temperature and E_a is the apparent activation energy of degradation. To obtain a good linear fit, it is necessary to select an appropriate range of advancement α , for this is considered $d\alpha/dT$ is a function of α , that is, the graph of $(d\alpha/dT)$ vs α is analyzed. The analysis of these curves will show a maximum in the range of $\alpha = 0.4 - 0.6$ (maximum point of the hood where the behavior is linear) (García and Fernando, 2014). This range coincides with the beginning of the polymer degradation reaction.

2.5 Calculation of the glass transition temperature T_g

The calculation of the glass transition temperature was obtained by means of differential scanning calorimetry (DSC) curves of untreated polystyrene and recovered polystyrene, this with the help of Proteus software version 5.2.1.

2.6 Characterization

Thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) was carried out with a simultaneous analysis equipment (Netzsch STA 449 F3 Jupiter) and was carried out in the range of 25–540°C with a ramp of 20°C/min, a nitrogen atmosphere of 99.999% purity for WEP and WEP recovery. Natural and synthetic esters were analyzed by infrared (IR). Data were collected using a Shimadzu IRTacer-100, with attenuated total reflection (ATR) accessory (35 scans, 4 resolutions, 4000-400 cm^{-1}). Scanning electron microscopy (SEM, JEOL JSM-6510IV) was used to analyze the surface morphology.

3. Results And Discussion

3.1 Dissolution Curves

The methodology proposed in this work is a two-step process: the dissolution of WEP (Fig.1) and its recovery by precipitation. The curves of dissolution time show how long it takes for WEP to dissolve, starting from an initial concentration of 10% to a concentration of 100% WEP (%w/w) using 1g of ester (Fig.2). The results indicate that glyceryl tributyrate takes 130 min to dissolve 100% by weight of WEP (1g of WEP), while omega-3 and ethyl butyrate take 31 min and 7 min, respectively, indicating that the best solvent is ethyl butyrate, since it dissolves WEP in a shorter time.

In his research, Noguchi, in the process of dissolving expanded polystyrene, what he calls "WEP shrinkage" found that to dissolve 30% wt of WEP it took him 53 min using d-limonene essential oil [Noguchi et al., 1998]. On the other hand, in a more recent work, Gil used essential oils for the dissolution of WEP, reporting that the essential oil of star anise is the most efficient in its work and for 30% wt of WEP it requires 5 min, respectively [11]. In this work, the use of omega-3 for the same percentage by weight, there is a dissolution time of 6 min, for glyceryl tributyrate of 11 min and for ethyl butyrate 2 min, approximately, so the use of the proposed esters is competitive when dissolving the expanded polystyrene.

In all three cases, the curves of the times required to dissolve a certain amount of expanded polystyrene increase exponentially as the concentration of expanded polystyrene Co (%w/w) in relation to the ester increases.

The recovery of the expanded polystyrene consisted of washing with sufficient methanol and stirring until a white solid was obtained, then rinsing with isopropyl alcohol to remove any remaining residue. The WEP was allowed to dry at room temperature and characterized by SEM.

3.2 SEM

The WEP before treatment and after treatment was analyzed by Scanning Electron Microscopy. Fig.3a shows the WEP prior to treatment, in which a closed cell structure filled with air is observed [6], with cells or holes characteristic of the material [4], this is due to its manufacturing process. After treatment, this feature is no longer present; micrographs show a rough, airless surface with irregular stacked layers or sheets (Fig.3b and Fig.3c), indicating reduced WEP volume.

3.3 FTIR

The characterization of the esters used for the dissolution of WEP by the IR technique is shown in Fig.4 all the characteristic bands of polystyrene are present in each of the samples indicating a good recovery of the WEP. However, in spectrum (b) a band around 1740 cm^{-1} is observed, this band is due to the ester still present in the sample, so the result suggests an extra treatment to be able to eliminate traces of the ester used.

3.4 TGA

In Fig.5 the results of the Thermogravimetric Analysis belong to untreated WEP shown only a 99.29% mass loss, with a degradation temperature of approximately 360 °C [20,27]. For WEP recovered with omega-3, two mass losses are observed; the first of 3.97% in the temperature range 110-285 °C, which corresponds to the omega-3 content still present in the sample. The degradation temperature is close to 414 °C, which is where the second mass loss of 94.6% is observed, typical of the amount of WEP in the recovered material.

In the case of WEP treated in glyceryl tributyrate, two slopes are similarly observed; the first mass loss of 19.29% correspond it is at the temperature of 120 °C is due to the presence of the glyceryl tributyrate remaining in the sample. This can be corroborated since the boiling point of glyceryl tributyrate is approximately 174 °C. The degradation temperature of the recovered material is 408 °C, at which point the second slope corresponding to the loss of mass of 68.39% is shown, which belongs to the amount of recovery of the expanded polystyrene in the sample.

Finally, for the WEP treated in ethyl butyrate, two slopes are observed: the first mass loss of 2.66% in the temperature range 110-225 °C, due to the presence of residual ethyl butyrate in the sample (bp = 120 °C). The degradation temperature is 364 °C, in which the second mass loss of 96.89% is observed, a mass percentage that is related to the amount of WEP recovered.

3.5 DSC y Tg

In Fig.6 the curves of the samples analyzed by Differential Scanning Calorimetry are shown. An exothermic transition of the four materials tested is represented: WEP untreated and treated WEP esters, respectively. Through the thermogram obtained from the DSC analysis, the glass transition temperatures Tg of the four materials were calculated (see Table 1), the value obtained from the Tg of the WEP untreated is 106.7 °C, a temperature similar to that already reported (101-102 °C) [1]. Having a Tg indicates that the amorphous property of the recovered polymer was preserved.

Table 1. Glass Transition.

Sample	Glass Transition (°C)
WEP untreated	106.7
RE-OME3	75.8
RE-GT	56
RE-EB	94.3

3.6 Apparent activation energy of degradation E_a

Activation energy was calculated with Friedman's differential method. This method compares the rates of weight loss (da/dT) for a fractional weight loss with a certain rate of heating β (K/min) [Muñoz et al., 2015].

In this work, the analysis was done with a heating rate β of 20 °C/min (293.15 K/min) for the four samples. According to the results, the value E_a of the WEP without treatment was 268.5 JK/mol, this value enters the range of 240-275 KJ/mol that is reported in other work for experimental conditions under flow nitrogen, with an average of 245 KJ/mol [14]. WEP values recovered show variations to the approximate value of the WEP untreated, this could be due to the remnants of esters employed for each test, as these are still present in the polymer. The results are shown in Table 2.

On the other hand, different ranges and values of the WEP E_a have been reported, such as values of 125-147 KJ/mol with an average of 138.39 KJ/mol [32], 100-107 KJ/mol with an average of 104.31 KJ/mol and 126.52 KJ/mol [Jun et al., 2006], 46-170 KJ/mol with an average of 89.62 KJ/mol [17] and 60-100 KJ/mol [Azimi et al., 2008], these variations are due to the experimental conditions, that is, if the work was done with a nitrogen or air flow and the gas flow, the temperature and the heating rate, as well as the kinetic model with which the values were obtained of the E_a . However, studies show that the degradation reaction of WEP in a nitrogen or air environment is carried out in a single reaction step [14,32].

Table 2. Results of activation energy of WEP and WEP recovered by Friedman's method.

Kinetic model	Sample	Apparent activation energy (KJ/mol)
Friedman	WEP untreated	268.5
	RE-OME3	286.3
	RE-GT	220.2
	RE-EB	294.3

4. Conclusions

In this study it was confirmed that the use of the proposed esters: omega-3 and glyceryl tributyrate and ethyl butyrate can satisfactorily dissolve WEP residues, having good WEP recovery results. The use of omega-3, gives new opportunities to take advantage of the residual mass of fishing activities. Omega-3 dissolved WEP faster followed by glyceryl tributyrate, with 31 min and 130 min, with a recovery of 94.6% and 68.4%, respectively.

The proposed method creates a range of opportunities for caring for the environment, as it is a green and environmentally friendly recycling method. Likewise, reducing the volume of WEP through dissolution provides an advantage for its transport, since being a bulky waste it turns out to be a complication in the

recycling process. Recovered WEP resulted of the dissolution of expanded polystyrene with organic solvents had been used to obtain coatings, [7] paints, [Myint et al., 2010; Vázquez and Ortega, 2020] resins [Jimenez-Francisco et al., 2018; Salazar and Guerra, 2015], etc. this can be the next step for this investigation.

Declarations

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Figures

Figure 1

Process of dissolving expanded polystyrene waste in omega-3: (a) WEP before dissolution, (b) WEP in contact with omega-3 for dissolution (3 min) and (c) final dissolution of WEP (31 min)

Figure 2
Dissolution time curves for omega-3, glyceryl tributyrates and ethyl butyrate

Figure 3
(a) WEP untreated, (b) WEP recovered with omega-3 treatment and (c) WEP recovered with treatment in glyceryl tributyrates. The scales shown in the micrographs are for a better appreciation

Figure 4

Comparison of FTIR of (a) WEP untreated, (b) recovered WEP treated with glyceryl tributyrates, (c) recovered WEP treated in omega-3 and (d) recovered WEP treated in ethyl butyrate

Figure 5

TGA curves of untreated WEP, treated in ethyl butyrate, omega-3 and glyceryl tributyrates

Figure 6

Thermogram obtained from DSC analysis for WEP untreated and post-treatment

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