



Polymer mortars prepared using a polymeric resin and particles obtained from waste pet bottle



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HIGHLIGHTS

- Mechanical and chemical methods are employed for recycle waste PET.
- PET recycled resin with similar properties to commercial resin is obtained.
- PET recycled resin is used as joint material between PET particles and sand.
- Polymer mortars with acceptable physical and mechanics proprieties are obtained.

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ABSTRACT

Recycling of polyethylene-terephthalate (PET) to produce materials like concrete or mortar is one of the best solutions for disposal of such waste plastics. In this work, waste PET bottles were used to produce an unsaturated-polyester-resin (UPER) by a glycolysis process. This UPER was used as a binding agent to produce mortars polymers. Two mortar groups were produced: the first group varying the ratio sand/UPER and the second group varying the ratio sand/UPER/PET particles. The best mechanical and physical properties were showed by mortars with ratio 80/20 sand/UPER and 78/20/2 sand/UPER/PET particles. Results of this study demonstrate feasibility of reusing PET waste as particles and UPER to produce polymer mortars.

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1. Introduction

Polyethylene terephthalate (PET) is one of the most commonly used polymers, it is widely employed as a raw material to obtain products such as blown bottles for soft-drink and it is also used for food packaging containers and other consumer goods. According to the BMC (Beverage Marketing Directory), Mexican people drank 248 l of bottled water per capita in 2011 while American people only consumed 110 l of bottled water per capita [1]. Due to the immoderate increase on use of PET bottles, solid waste problem has grown significantly. In order to find a solution for this problem, in recent years some researches on the recycling of PET have been conducted. PET waste can be recycled by using mechanical methods that involve the collection, disintegration and granulation of the waste of this polymer. It may then be reused

in the manufacture of new products whose properties are lower than the original polymer. Other methods for PET recycling include the use of chemical depolymerization processes, such as alcoholysis process using methanol [2], glycolysis with glycols [3], and the hydrolysis under acidic or basic conditions [4]. Glycolysis of PET can produce the bis-hydroxyethyl terephthalate (BHET) monomer, which has widely been used in production of unsaturated polyester and rigid or flexible polyurethane [5]. Previous studies [6–8] have shown that it is possible to use resin obtained from post-consumer PET bottles as a binder, replacing fractions of cement during concrete manufacture. One advantage of the use of recycled PET in the production of polymer concrete is that PET wastes need not be purified, emphasizing the fact that it does not require color removal as other applications of recycled PET (coats, pillows, rugs, etc.) may do, minimizing this way the cost of the resin obtained. PET recycling for polymer concrete applications also saves energy as it decreases the amount of cement used, besides establishing a long-term disposal of waste PET which is an important

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consideration for recycling purposes [8]. In previous research it has been found that resins based on recycled PET can be used to produce good quality precast concrete elements with sufficient mechanical strength to be used in many applications such as pavements, median barriers, and sewer pipes [6]. The polymer concrete is more resistant to failure in both compression and bending, compared to concrete mortar based on Portland cement. Moreover, it has the advantage of achieving more than 80% of its final mechanical strength during the first curing day. However, the polymer concrete shows temperature sensitivity and its production cost from PET waste is high [9,10].

Other works have shown that the particles obtained from plastic bottles can effectively be used to partially or completely replace natural aggregates. Information has also been provided about the properties of concrete and mortars containing PET as aggregate, filler, particles or fibers. In these works authors conclude that the presence of plastic waste particles as aggregate in the concrete may improve various properties such as toughness which allows this type of concrete to absorb large energy amounts. Density in these materials also diminishes which results in a lightweight concrete [11–13]. Nevertheless, water resistance and the low surface energy of polymeric materials result in a weak mechanical bond between particles of polymer and the cement matrix [13]. A poor mechanical bond may cause little internal micro-cracks in the interfacial bonding area between polymeric particles and the cement matrix [14,15].

The aim of this study is to use waste PET bottles to perform mechanical and chemical recycling of this material. PET particles were produced as reinforcement in the polymeric mortars manufacturing. A chemical recycling of waste PET bottles was also carried out to obtain an unsaturated resin to be used in the polymer mortars production. Chemical recycling was performed using a glycolysis reaction from which an unsaturated polyester resin (UPER) was obtained. This resin was characterized and then was used as a binder at the interface between PET particles and sand in polymer mortars. Two types of polymer mortars were prepared and their physical and mechanical properties were evaluated.

2. Experimental

PET particles were obtained from water bottles post-consumer. Bottles were washed and cut into small pieces by hand to produce PET particles. The particles were meshing using two types of Tyler mesh, No. 3½ and mesh No. 4, with sized about 5 mm × 5 mm. PET particles thus obtained were used directly as particulate reinforcement and also to produce unsaturated polyester resin (UPER).

2.1. Production of unsaturated polymer resin

PET waste particles and ethylene glycol at a ratio of 1:1.5 weight/weight (wt./wt.), were charged together with 0.1% of tetrabutyl titanate into a liter round bottom flask with three necks. A reflux condenser, thermometer and stirrer were connected in each neck respectively. The tetrabutyl titanate acted as catalyst of the depolymerization reaction [16,17]. The reaction was conducted under reflux at 230–240 °C for 3 h. During this time several samples were taken in order to check the amount of monomer present. When the resin reached an acid value of 20–30 mg KOH/g, the reaction was stopped [18]. This resulting product of glycolysis was then reacted with maleic anhydride in a fixed ratio of 0.6:1 based on PET content original to produce UPER. Based on previous studies [19] the polyesterification was conducted as following: in a reactor at 180 °C for 4 h; considering weight of PET, 0.5% of hydroquinone was added to the hot resin to prevent curing of resin before use. Finally, the liquid resin was dissolved in styrene monomer to achieve 40% wt./wt. of styrene in the resin, in order to reduce viscosity.

The conversion percentage of PET is defined as $[(W_0 - W_1)/W_0] \times 100$, where W_0 represents PET initial weight and W_1 represents weight of non-depolymerized PET. Viscosity of resin was determined using a Brookfield TPM-LV-DVE viscometer at 25 °C, with shear rate of 20–40 RPM. Due to the shear rate was very close was not observed the Newtonian behavior. The total percent of solid means the non-volatile matter content of a resin, which is not an absolute quantity but depends upon the temperature and period of heating used for the determination. In this work, the percent of solid was determinate according to NMX-E-152-1984 [20].

2.2. Preparation of polymer mortars

Two types of polymer mortars were prepared, the first contains sand and UPER while the second was prepared using sand, recycled PET particles and UPER.

Table 1 shows the composition of each type of mortar prepared. The mixture for resin curing was prepared based on the weight of UPER, using 1% of methyl ethyl ketone peroxide (MEKP) and 5% of cobalt octoate. This was mixed manually for about 5 min and then cubic mortars of 50 mm by side were molded. Mortars were removed from the molds and cured during 7, 14, 21 and 28 days at 20 °C.

2.3. Characterization of resin and polymeric mortars

Changes in the chemical structure of the raw material and the products obtained after glycolysis and curing were characterized using a Thermo Nicolet NEXUS 470 FT-IR E. To determine the structures corresponding to each type of materials, changes in range of mid-infrared frequencies were monitored, in particular, hydroxyl stretching region (3700–3500 cm^{-1}); symmetric stretching of the ester carbonyl (1750–1700 cm^{-1}); stretch C–O of ester (1600–1400 cm^{-1}); the stretching of C=C conjugated into the aromatic ring (1450–1680 cm^{-1}) and stretching of aliphatic C=C (1620–1680 cm^{-1}) [21].

The crystallinity degree of PET particles and the unsaturated polyester resin were determined using X-ray diffraction in the range 2θ from 10° to 90°, using a Rigaku DMAX-2200 diffractometer. The JCPDF card number used to identify the phases present was: 60-989 polyethylene terephthalate ($\text{C}_{10}\text{H}_8\text{O}_4$)_n [22].

In order to determine thermal resistance of PET and cured resin, a thermogravimetric analysis was performed using an automatic Perkin Elmer Thermogravimetric Analyzer TGA7. The operating conditions were: a gas flow mixture of 79% N_2 and 30% O_2 with a rate of 20 ml/min, initial and final temperature of heating from 20 °C to 1000 °C respectively to heating rate of 10 °C/min.

The compressive strength of the polymer mortars was measured after 7, 14, 21 and 28 days [23]. For each period of time, three specimens were used in compressive strength measurement for each composition, and the average measurement is the one presented and discussed in this work.

Water absorption, density and apparent porosity according to ASTM-C-642-06 were determined for mortar samples with 28 days of curing [24].

Morphological observations on polymer mortars were made using a scanning electron microscope (SEM), JEOL JSM-6610LV with secondary electrons (SE) and backscattered electrons (BSE) detectors at 20 kV. The working distance was maintained at 10 mm.

An Olympus SZX16 stereoscopic microscope was used to visualize the adhesion between matrix-aggregate within mortars.

3. Results and discussion

3.1. UPER characterization

PET obtained from waste bottles was depolymerized by a glycolysis process, after which maleic anhydride was added to produce unsaturated polyester resin (UPER). The resin thus obtained was characterized to compare its properties with those of commercially used polyester resin. The parameters considered for this characterization were: % solids, acid value, viscosity of resin. The results are presented in Table 2; these values are in concordance correspond to values from a commercial resin [25]. It is also important to mention that the depolymerization process used in this work, allowed to achieve a 98.8% of efficiency reaction during the conversion of PET particles to UPER.

FTIR spectra obtained from both, PET and UPER, are shown in Figs. 1(A) and 2(A) respectively. In each spectrum, the positions of major bands corresponding to different functional groups of each material were indicated in Figs. 1(B) and 2(B) respectively. The spectrum in Fig. 1(A) shows an evidence of the chemical structure corresponding to commercial PET. In Fig. 2(A) the PET structural changes after carrying out the glycolysis reaction (UPER) are shown.

For the PET, the bands shown in the FT-IR spectrum of Fig. 1(A) were labeled using lowercase letters. These letters correspond with the functional groups present in the structural formula of the polymer that shown in Fig. 1(B). A stretching band for the C=O of ester groups are observed at 1720 cm^{-1} (letter a); two intense bands for stretching of C–O–C in ester groups can be seen at 1250 and 1095 cm^{-1} (letter b), the stretching of C=C aromatic ring is represented here as a low intensity peak at 1410 cm^{-1} (letter c).

Table 1
Polymer-mortars composition.

Mortar type		Composition		
Group	Name	Sand (%)	UPER (%)	PET particles (%)
I	A	95	5	–
	B	90	10	–
	C	85	15	–
	D	80	20	–
II	C2	83	15	2
	C5	80	15	5
	D2	78	20	2
	D5	75	20	5

Table 2
Physical properties of the UPER obtained.

Solids (%)	Conversion percentage of PET (%)	Acid value (mg KOH/g)	Viscosity average (cP)
81.5	98.9	35	483

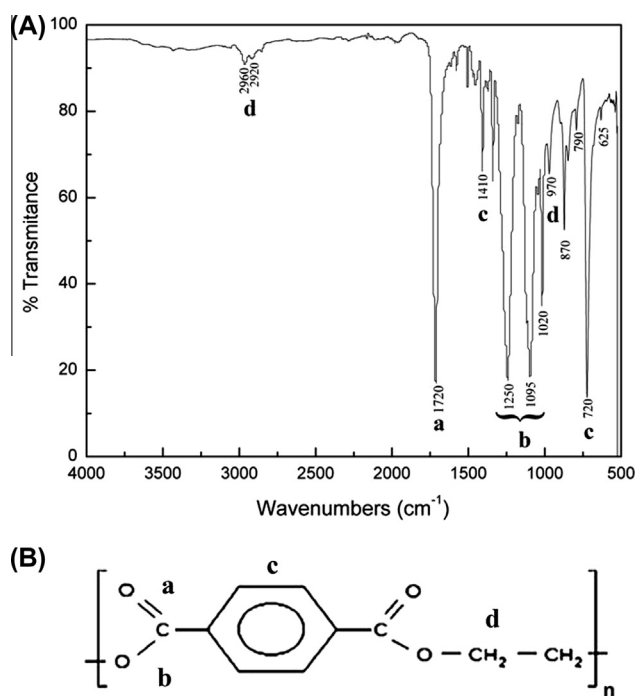


Fig. 1. (A) FTIR spectra and (B) chemical structural formula of the PET.

The stretching of C–H aliphatic has small peaks at 2960 and 2920 cm^{-1} and the bend out of plane of the same group is observed at 970 cm^{-1} (letter d). These absorption positions of the bands are in accordance with the standard spectroscopy of PET [26]. All the functional groups and chemical bonds do not show an evidence of cross linking between the polymeric chains. Therefore, this figure confirms the lineal structure of the PET.

FTIR spectrum for UPER, Fig. 2(A), shows that the major functional groups in the PET are retained after its reaction with maleic anhydride to form the UPER. These functional groups also are indicated in Fig. 2(B); the same lowercase letters are used to facilitate interpretation (letters a–d). The UPER spectrum also shows a band at 1640 cm^{-1} corresponding to stretching of C=C aliphatic (letter e), these unsaturated bonds are formed during the reaction between PET and the maleic anhydride [27]. Besides can be seen a broad band at 3500 cm^{-1} corresponding to the stretching of

O–H bond formed during the glycolysis reaction (letter f). These band positions indicate that the chemical structure of the PET resin was transformed to a polyester resin still preserving a lineal structure (UPER).

Fig. 3(A) shows FT-IR spectrum of preparing resin after curing process, the position of the principal functional groups is very similar to the one observed in UPER spectra (see Fig. 2A). The main difference between FTIR spectra of those materials is the diminishing of the bands intensities in spectrum of cured resin. This could be attributed to heating during the curing reaction that causes cross-linking in the active sites of styrene and UPER [28]. This cross-linking occurs due to the aliphatic double bonds contained in the UPER structure. These double bonds are opened by the styrene presence, so the C–C aliphatic bonds are induced, as shown in Fig. 3(B) with letter e. The band at 1640 cm^{-1} associated with aliphatic C=C, disappears practically. Therefore, this FTIR spectrum justified the three-dimensional structure of the cured resin [28].

The X-ray diffraction patterns are shown in Fig. 4. In this figure it is possible to observe that the PET possesses a semi-crystalline structure because it is a linear polymer that can arrange its chains, while the resin loses the semi-crystalline order due to crosslinking of chains during the curing process. However, in the cured resin its chains cannot bend, as consequence of the cross-linking during the curing process. This phenomenon results in the formation of an amorphous polymer [29].

According to the TGA thermograms for PET and cured resin, Fig. 5, PET is stable up to 350 °C, from this temperature up, its decomposition starts, being completed at approximately 410 °C at which a weight loss of 84% is reached. The cured resin initiates a decomposition process from 200 °C and exhibits a weight loss of 82% at 400 °C. This test was carried out for determining the thermal resistance of the material at high temperature.

Studies of thermal degradation of polymeric materials in polymer mortar composites are important from the practical and scientific point of view. On the practical side, TGA studies not only explain the behavior of polymer exposed to high temperature, but also help to establish criteria for the selection of materials for specific uses [30]. The TGA analysis indicates that the polymer mortar, prepared with the resins obtained after recycling of PET bottles, has a high thermal resistance and can be used in various civil engineering applications such as a binder [6].

3.2. Characterization of mortars polymeric

Water absorbed, density and apparent porosity were determined in both types of mortars and are showed in Table 3. Type I, containing UPER in different compositions and type II containing PET particles and UPER in different proportions.

It was not possible to determine physical properties of mortar with 5% UPER (type IA) due to lack of adhesion between the components. Analyzing the values in Table 3, the density values showed a slight increasing influenced by the resin content. Moreover, while the resin content increased the mortars IC and ID exhibited a considerable reduction in the values of apparent porosity and water absorption from 65% to 70% with respect to values of mortar IB. These results indicate that from 15% UPER the amount of resin was sufficient to wet the sand contained in the mixture, resulting in high-level compacting in mortars.

Under the stereoscope the samples exhibit a good definition of their components; the sand grains are dispersed through the polyester resin as it can be observed in Fig. 6. The image of Fig. 6a shows the mortar with the lower content of resin that has been studied (5% UPER). In this figure it is possible to observe the grains of sand separated from the resin, demonstrating the lack of adhesion between the sand and resin due to the insufficient content of the resin. However, when the content of resin is increased at

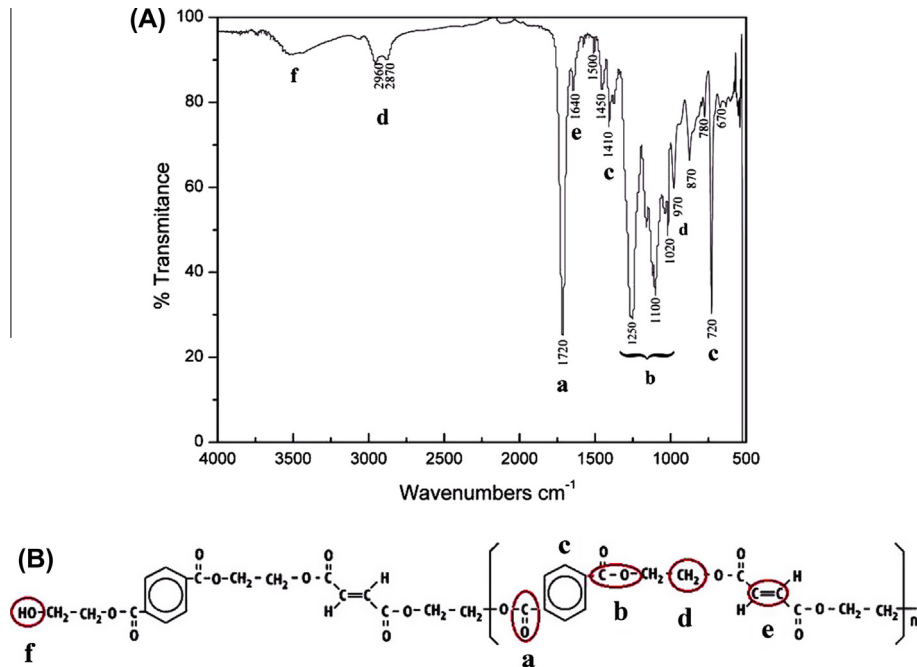


Fig. 2. (A) FTIR spectra and (B) chemical structural formula of the UPER.

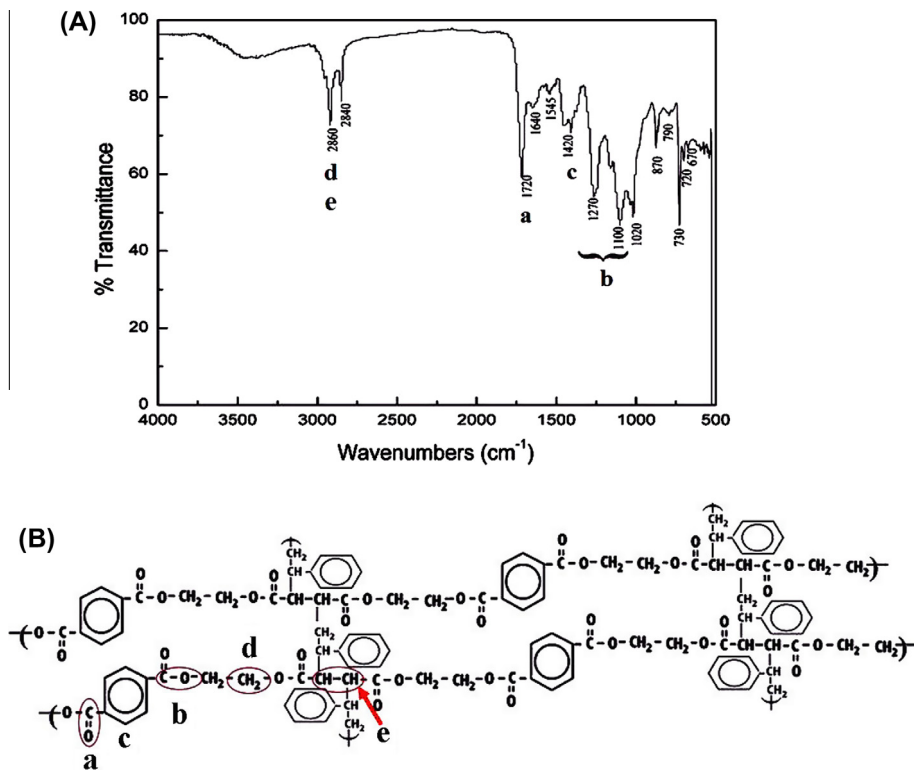


Fig. 3. (A) FTIR spectra and (B) chemical structural formula of the cured resin.

10% and 15%, as shown by the mortars in Fig. 6b and c, where they became more compact and fully covered the sand particles. In Fig. 6d, when the percentage is increased at 20% of resin, the superficial appearance of the mortar is very different with respect to previous figures, in this figure can be seen that the resin cover it fully the sand particles, favoring their wetting.

Compressive strength values of the mortars type I in function of curing time are shown in Fig. 7. It can be observed that mortar IA shows the lowest values of compressive strength (under to 5 MPa). This behavior is attributed to the lack of binding between sand and resin, as seen in Fig. 6a, turning out to be a weak and fragile mortar. The increase in resin content significantly improves compression

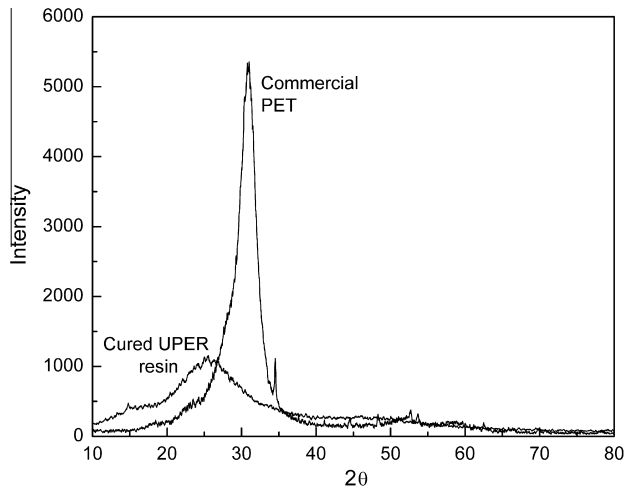


Fig. 4. X ray diffraction patterns of commercial PET particles and cured UPER resin.

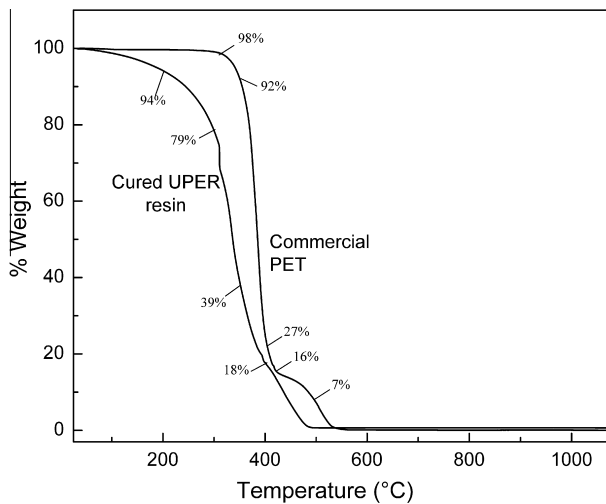


Fig. 5. TGA thermograms of commercial PET and cure UPER resin.

strength since the early days of curing. The values of resistance to compression at seven days of curing were 12, 20 and 22 MPa for the mortars IB, IC and ID, respectively. When increasing the ratio of sand to resin, the compressive strength increased too, due to the improvement in wettability between sand and resin, as shown in Fig. 6b, c and d. These results are in accordance with the data exposed in Table 3, can be seen a decrement in the apparent porosity values of the mortars. The results of Fig. 7, also show a dependency between compressive strength and curing time of the mortar. The values of compressive strength of the mortars after

28 days of curing rise slightly (approx. 7 MPa) compared to the values obtained during the first seven days. This increase is attributable to crosslinking of polymer chains during the curing process of the UPER with the MEKP and cobalt octoate [28]. The polymerization rate is very high in the early hours of cure, but after a while the reaction continues but at a much lower rate of polymerization. As it can be seen the mechanical properties of the resins are time dependent and they seem to reach a steady value after approximately 20 days of curing period [31].

The mortars IC and ID showed the best mechanical behavior in compression, which is why these compositions were selected (15% and 20% of UPER) to prepare mortars with additions of 2% and 5% of PET particles (mortars type II). The values of water absorption, dry and fresh density and the apparent porosity of these mortars type II are presented in Table 3.

The experimental results show that by replacing 2% and 5% (of the sand weight) with PET particles, the values of water absorption and apparent porosity of mortars type II were significantly affected compared with mortars IC and ID. Fresh and dry density values for mortars type II showed a slight decrease that is associated with the content of PET particles. A greater content of PET provides structures that cannot be packaged efficiently due to its flat shape which retains air bubbles at the interface during the PET-sand mixture. In mortar C5 with the lowest percentage of UPER (5%), can be seen some voids which not be filled with the resin (Fig. 8a). In mortar D5, containing a higher percentage of UPER (20%), better packing between the phases could be observed as the content of the resin was sufficient to cover the sand particles and PET, see Fig. 8b.

The quality of the bond between PET particles, sand and UPER in mortars type II was analyzed.

Backscattered electron micrographs of SEM shown in Figs. 9 and 10 correspond to mortars type II with 15% and 20% UPER, respectively. In mortar with 15% UPER, it seems to be that the amount of resin is not enough to bind sand particles and PET completely; a weak connection between components is observed. This certainly increases porosity of mortars, which may explain the increase in permeability measured in mortars C2 and C5. Mortar with 20% UPER shows a better adhesion between PET particles surface and sand. The higher resin content in mortars allows act as a binder, as shown in Fig. 10. A good bonding between PET and UPER is favored due to its similar chemical nature. Other researches on polymeric concrete have been reported that the presence of UPER improves the poor adhesion between PET particles and the cement matrix [14,32].

The compressive strength vs function of curing time for mortars type II is shown in Fig. 11. It is observed that addition of PET particles contributes to decreasing the compressive strength of these mortars. Comparing the results of compressive strength for mortars type IC and ID, with those type II containing 15% UPER, reductions of 12.6% and 18.2% in the values of compressive strength were observed when substituting 2% and 5% of sand with PET particles in mortars C2 and C5, respectively. By increasing the resin

Table 3
Physical properties of the different mortars.

Mortar		Water absorption (%)	Dry density (g/cm ³)	Fresh density (g/cm ³)	Apparent porosity (%)
Type I	B	21.0	1.72	1.93	35.5
	C	7.0	1.87	1.92	12.2
	D	5.4	1.99	2.08	10.7
Type II	C2	16.0	1.75	1.87	27.0
	C5	19.0	1.79	1.95	32.0
	D2	7.7	1.93	2.00	14.9
	D5	9.2	1.85	1.93	17.0

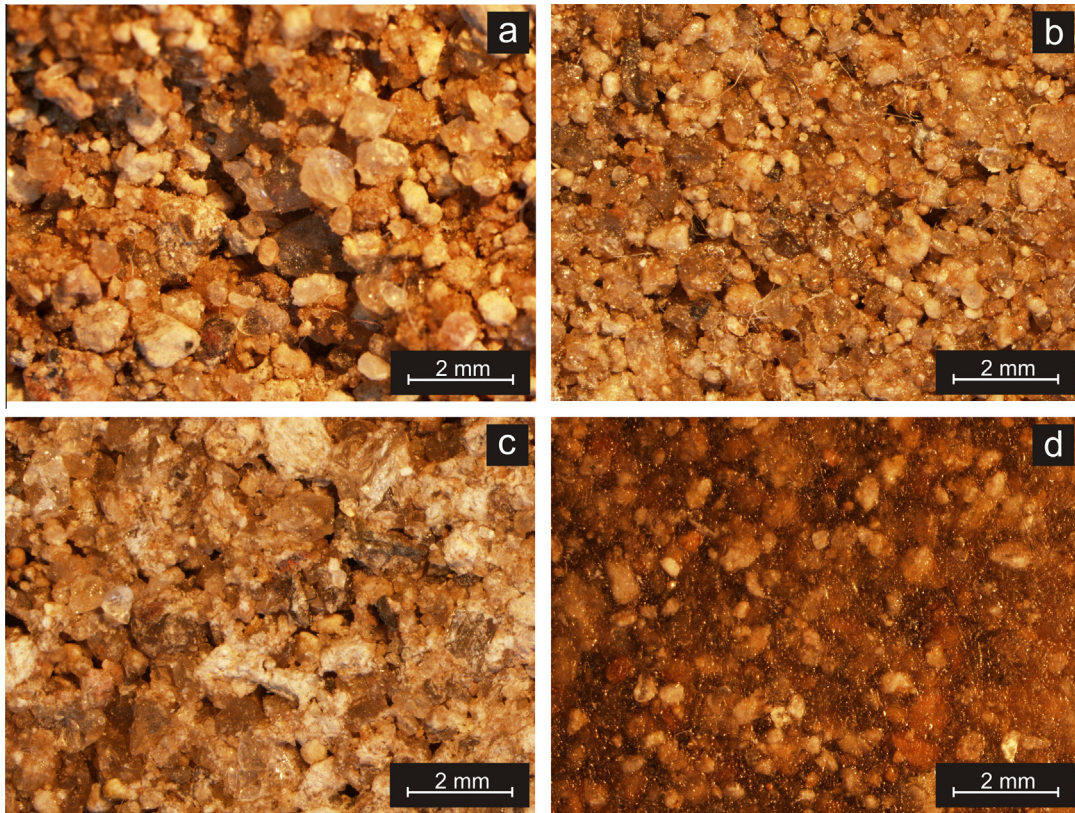


Fig. 6. Surface morphology of the type I mortars: (a) IA with 5% of UPER, (b) IB with 10% of UPER, (c) IC with 15% of UPER and (d) ID with 20% of UPER.

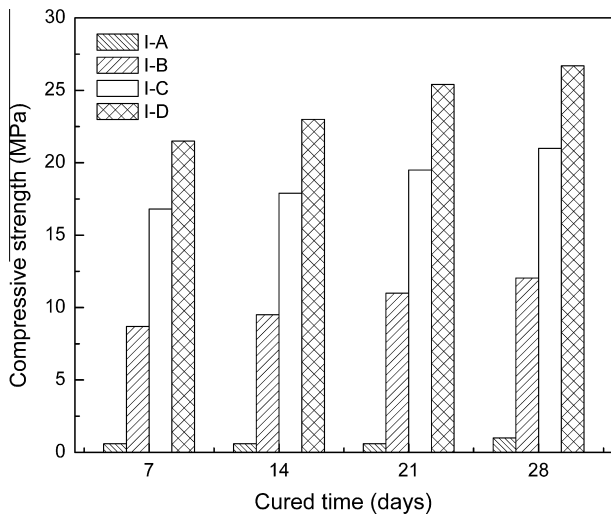


Fig. 7. Compressive strength of polymeric mortars-type I in function of cured time.

content, the compressive strength of specimens improved slightly for mortars type II (D2 and D5). However, compared to the strength obtained for mortars type I, a reduction of 11.2% and 13.8% when adding 2% and 5% of PET particles, was obtained respectively. It has also been reported in previous work that mortars elaborated using concentrations above 5% of shredded PET as aggregate and 12% of epoxy or commercial unsaturated polyester resins, contributes to decreasing the compressive strength between 21% and 70% compared to mortars containing only resin [33].

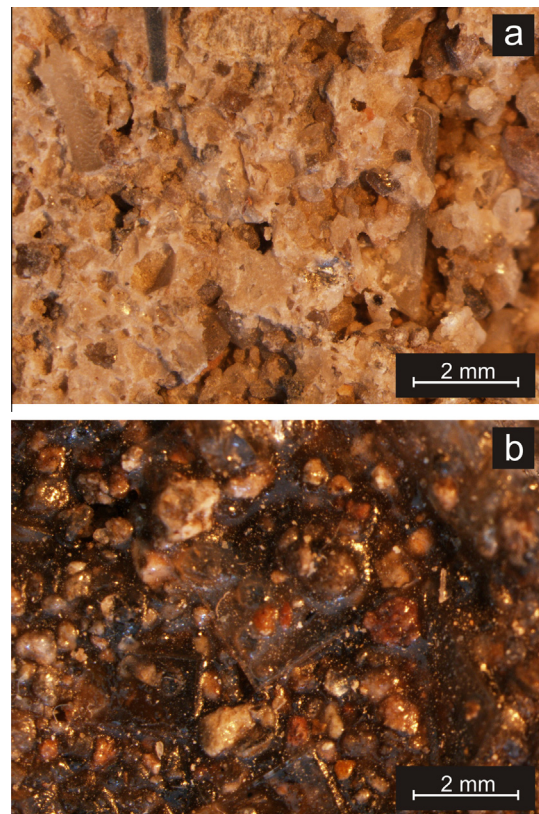


Fig. 8. Surface morphology of the type II mortars: (a) C5 mortar and (b) D5 mortar.



Fig. 9. Surface morphology of the mortar type II C5.

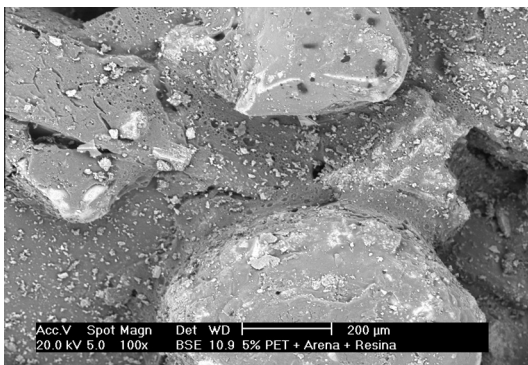


Fig. 10. Surface morphology of the mortar type II D5.

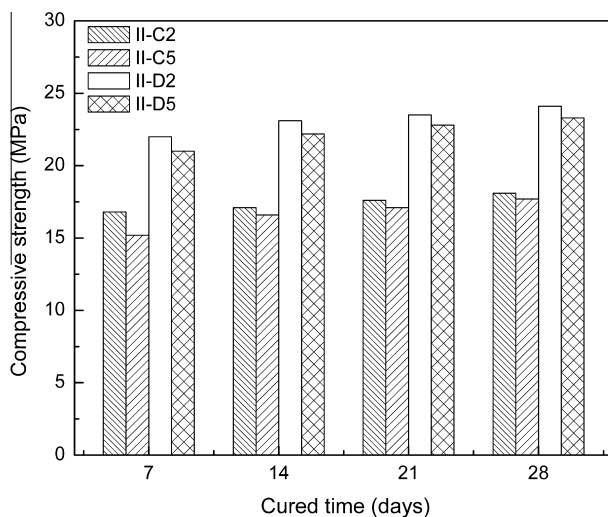


Fig. 11. Compressive strength of polymeric mortars-type II in function of cured time.

4. Conclusions

- In this work two methods to the recycling of PET were used: a mechanical method to produce PET particles and a chemical method to obtain the unsaturated polyester resin (glycolysis). Both products were used in the manufacture of polymer mortars.
- It was possible to synthesize an unsaturated polyester resin from waste PET with optimal physical properties, similar to the commercial polyester resins. Thermogravimetric analysis indicated that after curing process, this resin is stable below

350 °C. X ray diffractogram indicates the structural transformation of the unsaturated polyester resin from crystalline to amorphous phase after curing process.

- Polyethylene terephthalate (PET) post-consumer was added as aggregate or/and as unsaturated polyester resin in the manufacture of polymeric mortars. The importance of this environmentally friendly material can be highlighted.
- The mechanical and physical properties of polymeric mortars prepared in this work are dependent on the resin content. An increase in the resin content produces a sand better wettability and improves junction between phases, thus leading to more compact materials.
- The addition of the PET particles to the polymer mortars slightly reduces the mechanical and structural properties of the new material obtained.

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