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Artículo Original

Estudio de viabilidad de la incorporación de tereftalato de polietileno (PET) micronizado en morteros

Feasibility study of the incorporation of micronized polyethylene terephthalate (PET) in mortars

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Resumen

En el presente estudio se investiga la influencia de la incorporación de residuos de tereftalato de polietileno (PET) micronizado sobre las propiedades físicas y mecánicas de los morteros. Primero, se evaluaron los materiales utilizados mediante pruebas físicas y químicas, y luego se realizó el estudio de la dosificación y moldeado de las probetas en las dimensiones de 5 cm x 10 cm. Las propiedades físicas y mecánicas de los morteros se evaluaron a través de la absorción, índice de vacíos, masa específica, resistencia a la compresión simple y módulo de elasticidad del ensayo. La incorporación de PET al mortero resultó en un aumento en el índice de absorción y vacío y una disminución en la masa específica y resistencia a la compresión en los dos niveles analizados. La incorporación de este material es viable, ya que los valores obtenidos se encuentran dentro de las normas estandarizadas y el uso de este residuo en productos de construcción civil ayuda a reducir el impacto ambiental causado por una disposición inadecuada.

Palabras clave: Morteros, construcción, reciclaje, residuos plásticos, sostenibilidad

Abstract

This study investigates the influence of the incorporation of micronized Polyethylene terephthalate (PET) residues on the physical and mechanical properties of mortars. First, the materials used were evaluated through physical and chemical tests, and then the study of the dosage and molding of the specimens in the dimensions of 5 cm x 10 cm was carried out. The physical and mechanical properties of the mortars were evaluated through the test's absorption, void index, specific mass, resistance to simple compression, and modulus of elasticity. Incorporating PET to the mortar resulted in an increase in absorption and void index and a decrease in specific mass and compressive strength at the two levels analyzed. Incorporating this material is viable, as the values obtained are within the standardized norms and the use of this residue in civil construction products helps reduce the environmental impact caused by inadequate disposal.

Keywords: Mortars, construction, recycling, plastic waste, sustainability

1. Introduction

Recently, due to population growth and rapid global industrialization, a disposal culture has developed and generated large amounts of waste of all types [1]. The recovery of solid waste provides the possibility of taking advantage of the resources contained in them as inputs or raw materials within the scope of the circular economy [2].

During the last decades, plastic waste has been studied as a component of concrete and mortar. They have been used as aggregates, a binder to replace cement, and fiber reinforcement [3]. Given the growing scarcity of spaces for landfills and their increasing costs, waste today poses a serious threat to the environment, which is why recycling can be a promising solution [1].

Polyethylene terephthalate (PET) is the most commonly used polymer globally due to its application engineering properties, a recyclable thermoplastic, naturally transparent, and good resistance to traction and impact [4]. However, the PET bottle, a post-consumer product, has generated great interest in the environmental consequences involved, and a suitable alternative is to incorporate it in mortar and concrete [5].

Recycled PET in Brazil has several applications, being distributed mainly in the textile industry and the manufacture of unsaturated resins. Laminates, metal sheets, food, and non-food packaging represent a significant proportion of reused PET [6]. In recent years, researchers have focused their studies on using plastic waste as an alternative to natural resources as raw material [5, 7].

An alternative for the application of plastic waste is its incorporation in construction works, which use considerable amounts of materials [8]. In the construction sector, PET has been used in various applications, such as asphalt mixtures [9-11], production of concrete with fibers [12-17] or as an aggregate [18-21], plaster [22] and blocks [23]. Considering concrete applications, the most economical is the direct use of PET as an aggregate [8].

Considering the correct disposal of the significant volume of polyethylene terephthalate (PET) waste generated worldwide, the performance of interlocking blocks produced with partial replacement of fine aggregate by micronized PET in the percentages of 2.5, 5.0, 7.5, and 10.0% was evaluated. The results indicated that the values of compressive strength, modulus of elasticity, and flexural strength decreased with the increase of PET percentage [24].

Akaozoglu *et al*. [25] investigated the performance of PET as an aggregate in mortar, comparing conventional mortar to mortar with the addition of PET. The study points to a reduction in the resistance to compression and flexion at 28 days with the incorporation of PET. The literature presents some research already developed with incorporating PET in mortars [3, 6, 25, 26].

Reis and Carneiro [6] investigated the use of PET as a fine aggregate in polymeric mortars at levels of 5, 10, 15, and 20%. The authors point out a reduction in resistance to compression and flexion but an improvement in the modulus of elasticity. Oliveira and Castro-Gomes [3] carried out a study on the physical and mechanical behavior of mortars reinforced with recycled PET fibers in the contents of 0.5% and 1%. They concluded that the incorporation of this additive provided again in flexural strength.

Sposito *et al*. [5] developed research to evaluate plaster mortars based on Portland Cement produced with incorporating PET residues in the contents of 2.5, 5, 10, 15, and 20%. The results were similar to those found in conventional mortars regarding absorption, permeability, and elastic modulus. However, the authors did not evaluate the mechanical strength of the PET-modified mortar with the levels mentioned above. Polyethylene terephthalate (PET) is the most common thermoplastic polymer belonging to the polyester family that consists of $C_{10}H_8O_4$ monomeric units [27]. The micronized PET has a granulometry passing through sieve No. 200 (75µm) and retained in sieve No. 400 (37µm).

Thus, we have two questions: the physical and mechanical evaluation of mortar modified with micronized PET and the viability of this material. Therefore, this work aims to evaluate the influence of micronized PET as a partial substitute for fine aggregate on modified mortars' physical and mechanical properties with contents of 5 and 10% and to verify the viability of this additive.

2. Materials and Methods

This topic describes the materials and procedures performed during the experimental phase of the research, following the standards of the Brazilian Association of Technical Standards (ABNT).

Materials

In the production of the mortar of this research, Portland cement CP II Z – 32 was used. The fine aggregate was Quartzous Sand and the hydrated lime used was Carbomil, type CH-I, manufactured from pure Cretaceous limestone. The particle size distribution was performed by laser diffraction of the hydrated lime, and modal behavior was observed with an average diameter of 9.87µm. For this lime, there are no particles larger than 100µm, with a percentage of 54.30% for the diameter of 5µm. The water used was potable and intended for human consumption.

PET plastic has a density of 1.38 g/cm³ at 20°C, a melting point of 265°C, and a glass transition point of 69°C [27]. Therefore, the source of the PET plastic used in this study was drinking water bottles. Micronized PET is a material that passes through sieve no. 200 (75µm) and is retained in sieve no. 400 (37µm), supplied in these dimensions by the company DEPET, headquartered in the municipality of Campina Grande-PB. Figure 1 shows the particle size distribution of the aggregates, tested according to the NBR NM 248:2003 standard, and Table 1 shows the results of the physical characterization of the materials.

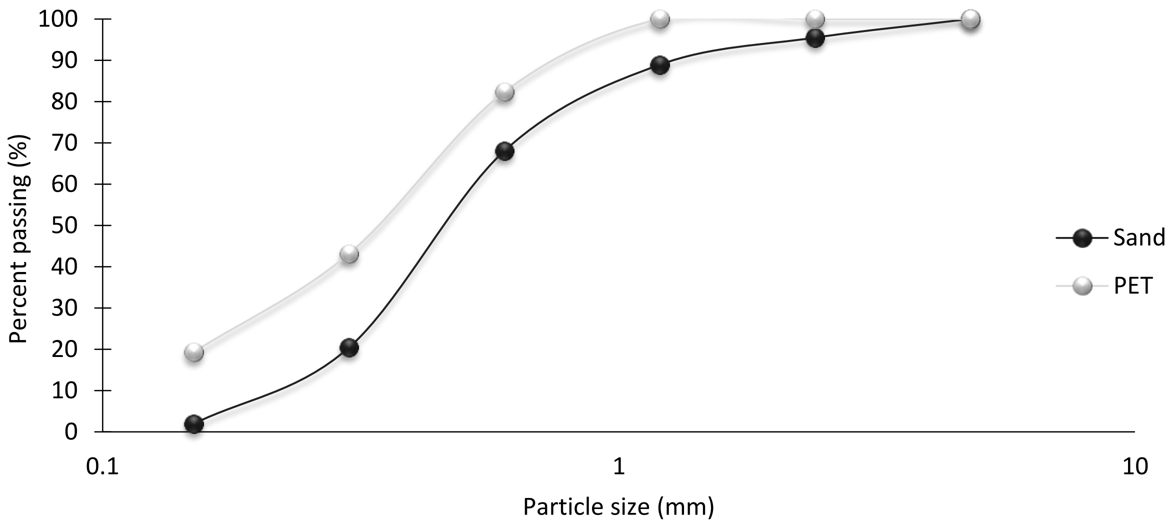


Fig.1 Particle size distribution of sand and PET aggregates

Table 1. Physical characterization of materials

Test	Results				Standard
	Sand	PET	Cement	Lime	
Actual specific mass (g/cm³)	2.56	1.38	2.91	2.63	NBR 16916:2021
Fineness index (%)	2.25	1.55	2.84	-	NBR NM 248:2003
Nominal maximum diameter (mm)	2.36	0.6	-	-	NBR NM 248:2003

According to Metha and Monteiro [28], most natural aggregates have a specific mass between 2.6 and 2.7 g/cm³, which are close to those obtained in this study. Furthermore, it is observed that the granitic aggregates are within the parameters established for use in concrete, according to the standards of ABNT and Neville [29].

It is observed that the result obtained for the sand fineness modulus in the value of 2.25 is within the optimal use zone, in which the fineness modulus can vary from 2.20 to 2.90. The sand used is classified as medium sand. The maximum diameter obtained for this aggregate is 2.36 mm. According to the values obtained, this sand is considered well graded, not presenting a significant deficiency or excess of any size, thus promoting a mortar with better workability and fewer voids between the grains. It is verified that the result of the fineness modulus obtained for PET is 1.55. This value falls within the lower limit established by NBR 7211:2009 for fine natural aggregates, ranging from 1.55 to 2.20. The maximum diameter obtained for this aggregate is 0.6 mm. Therefore, PET waste is classified as acceptable. The granulometry of the fine aggregate plays a fundamental role in the preparation of mortars. The aggregate dimensions directly influence the voids, the water/cement factor, and the workability of the mixtures.

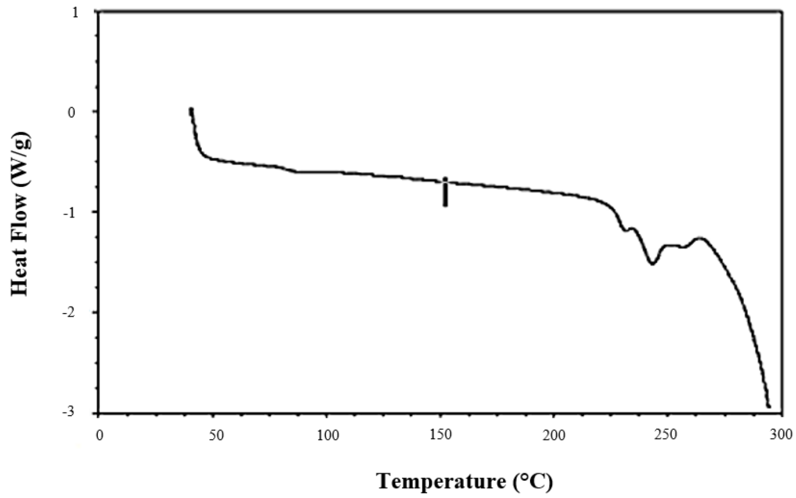
The Portland cement used in the research was CP II – Z 32 MPa cement, which contains Pozzolanic material ranging from 6 to 14% by mass, which gives the cementless permeability. According to Recena [30], cement's natural and unitary specific mass is approximately 2.70 g/cm³ and 1.0 g/cm³, respectively. Thus, the values obtained in this work are close to the values mentioned above. Furthermore, according to NBR 16697:2018, the limit value for cement fineness is 12%, so that the result obtained meets the established limit. Furthermore, Recena [30] obtained the characteristic values of specific mass and unit mass of lime in the order of 2.57 g/cm³ and 0.65 g/cm³, respectively. Therefore, the values obtained for the lime analyzed in this study are very close to those mentioned.

Tests for chemical, physical and mineralogical characterization of PET were carried out. Purity analysis by Differential Scanning Calorimetry (DSC) is a well-established technique. The method assesses the purity of the compound by analyzing the melting peak obtained, applying the Van'tHoff melting point depreciation law - which predicts the depreciation of the melting point of the pure compound due to the presence of impurities.

The occurrence of endothermic peaks is observed from the temperature of 200°C, indicating the occurrence of physical and chemical changes in the composition of Polyethylene Terephthalate. Differential thermal (DTA) and thermogravimetric (TG) analyzes of PET were performed in a BP Engenharia equipment, Model RB 3000, operating at 12.5°C/min. The maximum temperature used in the thermal analysis was 300°C, and the standard used in the DTA tests was calcined aluminum oxide (Al_2O_3). The TG curve of the PET was obtained with a sample mass of 7 mg, under nitrogen atmosphere (flow rate 50 mL/min).

Figure 2 shows the Differential Scanning Calorimetry (DSC) curves and the term differential and thermogravimetric analysis of micronized PET.

a)



b)

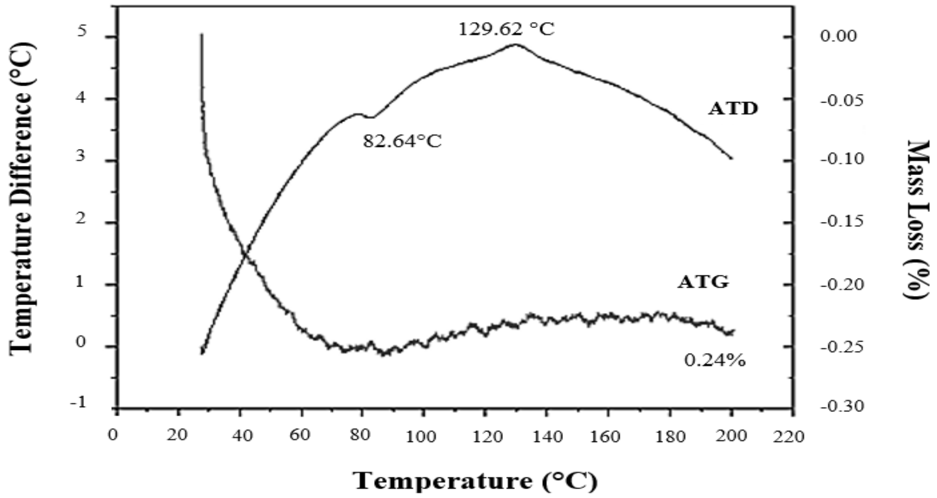


Fig.2 Characterization charts: a) Differential Scanning Calorimetry (DSC) of PET; b) Thermodifferential and thermogravimetric analysis of PET

In Figure 2 (b), it is observed that there is approximately 82.64°C in the occurrence of an endothermic peak, indicating a change in the material's physical state (solid to liquid), with a slight loss of mass. At 129.62°C, an exothermic peak is observed, demonstrating a new change in physical state (liquid to vapor). According to the thermogravimetric curve, it can be pointed out that there was a total mass loss of 0.24%.

Fourier Transform Infrared Spectroscopy (FTIR) is used to obtain infrared absorption, emission, photoconductivity, or Raman diffraction spectra of a solid, liquid, or gas. An FTIR spectrometer simultaneously collects data from a wide spectral range, giving it an advantage over a dispersive spectrometer, which measures intensity over a very narrow range of wavelengths for each measurement. For example, Figure 3 shows the infrared spectroscopy of Polyethylene Terephthalate.

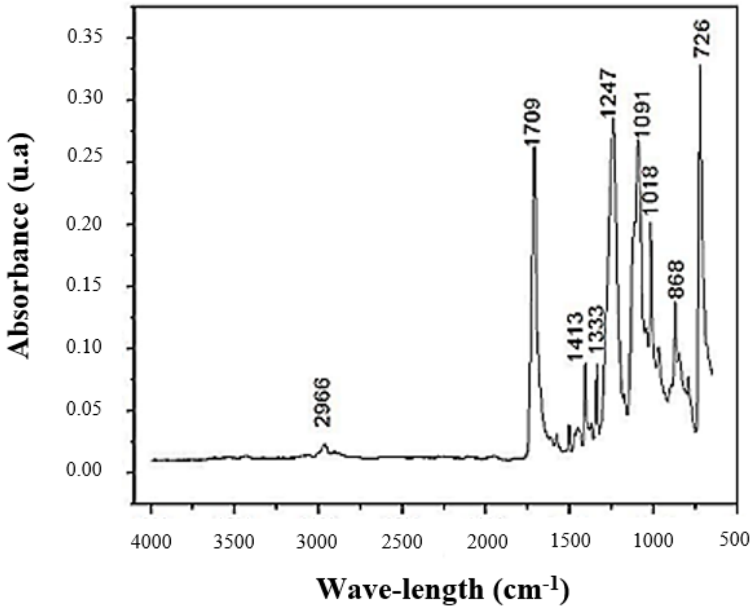


Fig.3 Infrared Spectroscopy of Polyethylene Terephthalate (PET)

The infrared spectrum of Polyethylene Terephthalate indicates the existence of several typical functional structures existing in the Polyethylene Terephthalate chain, highlighting the following characteristic bands: at approximately 3000 cm^{-1}, identified by the axial strain vibration of the group (=C–H), present in aromatic compounds (benzene); at 1709 cm^{-1} C=O stretch of carboxylic acid, indicative of the band; in 1247 cm^{-1} C(O)–O stretch of ester groups; at 1091 cm^{-1} and at 1018 cm^{-1} indicative of stretching of the C–O bond and approximately 726 cm^{-1} angular deformations of the substituted carbons in the aromatic ring.

Methods

The experimental research program was carried out in two stages: the first in the molding of specimens with pre-established levels of micronized PET residue, and the second stage corresponds to the analysis of the properties of the modified mortars.

For mortar dosage, the method used was that of the Brazilian Association of Portland Cement (ABCP), and, after the characterization of the materials, the mass mix of 1:2:9 (cement:lime: sand) and w/c (water/cement factor) varying for each content. The choice of replacement percentages of fine aggregate by micronized PET waste was 5 and 10%, determined from information extracted from previous research [5, 6] about the theme and knowledge material properties. Table 2 shows the trace and dosage used in this research.

Table 2. Mass trace for a mortar specimen

Mortar	Cement (g)	Lime (g)	Sand (g)	Micronized PET (g)	w/c factor	Water (ml)
Reference mortar (REF)	34.5	25.2	377.8	0	1.53	81.25
Mortar with 5% PET (5% PET)	34.5	25.2	358.9	18.9	1.52	75.0
Mortar with 10% PET (10% PET)	34.5	25.2	340.0	37.8	1.52	75.0

After the characterization of all the material and the study of the adopted trait, the molding of the specimens was carried out. To prepare the specimens, cylindrical molds were used, with dimensions 5 cm x 10 cm. For the compressive strength test, three specimens of reference mortar, three specimens of mortar incorporated with 5%, and three more for the content of 10% of Micronized PET. For the water absorption determination test by immersion, two specimens were molded for the reference mortar, two mortar specimens incorporated with 5% PET and two more for the 10% content, thus totaling 45 specimens, which were cured in wet sand, for ages 7, 14 and 28 days.

The molding followed the procedures according to NBR 7215:2019, with the mortar being made in four layers, where 30 uniform blows are applied with a standard socket in each layer. Finally, the leveling is carried out using the ruler. After 24 hours of molding the specimens, they are demolded, identified, and placed in the wet sand, as shown in Figure 4.

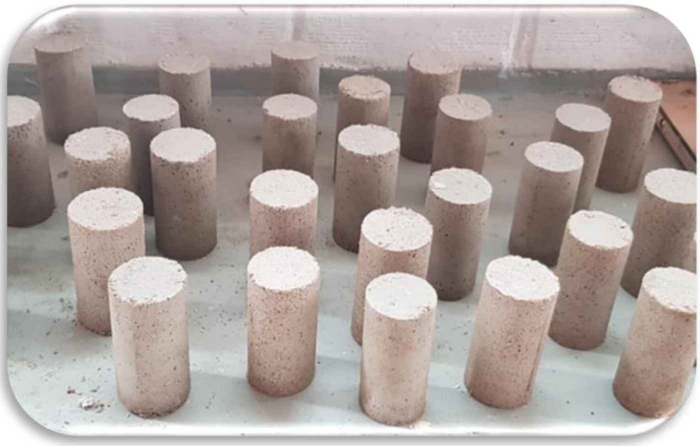


Fig.4 Unmolded specimens

Tests of water absorption by immersion, void index, and specific mass were carried out to determine the mortars' physical properties. These tests are governed by NBR 9778:2009. After being submerged in curing, the specimens were oven-dried for 72 hours (105 ± 5°C) and measured their masses were. Then they were submerged in water at room temperature for another 72 hours and were weighed again.

The simple compressive strength test, a technique used to verify the compressive strength of mortars made of Portland cement in its hardened state, was performed using the NBR 7215:2019 standard. The procedure is carried out through an adaptation that specifies the compressive strength of Portland cement, which was modified to measure this property in mortars. The type of press used to break mortar specimens must act at a speed of 0.25 ± 0.05 MPa/s, as recommended by the standard. The results obtained for the simple compressive strength of the mortar were the average of three specimens.

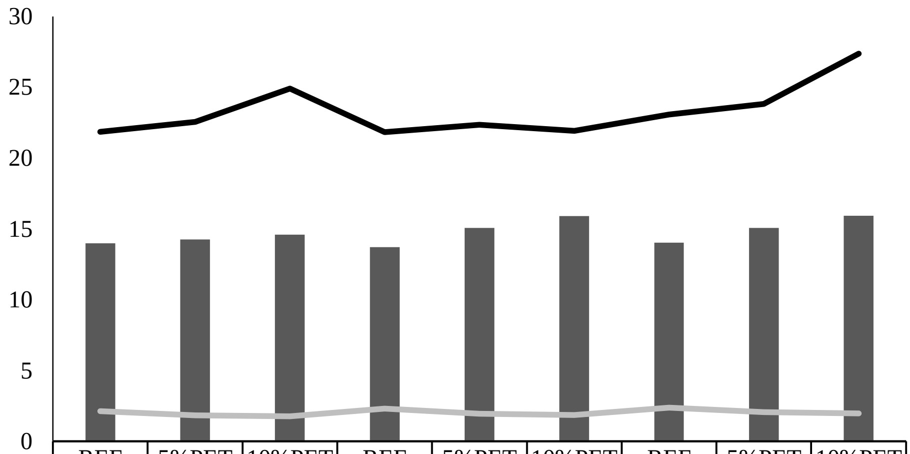
The test for evaluating the modulus of elasticity in mortars with partial replacement of the fine aggregate is an adaptation according to the NBR 8522:2017 standard, which prescribes that the modulus of elasticity or modulus of deformation as the property of concrete whose numerical value is the angular coefficient of the secant line to the specific stress-strain diagram. Knowing the modulus of elasticity of concrete, whose numerical value corresponds to the initial tangent deformation modulus, as proposed in this standard, makes it possible to meet the requirements of structural design, considering the possibility of inferring the modulus of deformation in usual design stresses through approximations provided for in the technical standards. Thus, three specimens were molded for each PET content, for each age. The modulus of elasticity value was the average value between the three specimens of each incorporation content and the age of the mortars. The specimens were broken by simple compression in a press located at the Pavement Engineering Laboratory of the Federal University of Campina Grande. Through the generated stress-strain diagrams, the modulus of elasticity values was determined.

3. Results and Discussion

This section presents and discusses the results obtained in the experimental phase of the specimens of mortars with micronized PET residue.

Determination of the physical properties of mortars

Figure 5 shows the results for water absorption by immersion, void index, and apparent specific mass for the reference mortars and those incorporated with 5 and 10% micronized PET to replace the fine aggregate.



	REF	5%PET	10%PET	REF	5%PET	10%PET	REF	5%PET	10%PET
	7 days			14 days			28 days		
Absorption	13.98	14.25	14.59	13.7	15.07	15.91	14.03	15.07	15.93
Void Index	21.86	22.55	24.9	21.82	22.35	21.93	23.07	23.82	27.38
Apparent Specific Mass	2.13	1.84	1.76	2.32	1.94	1.87	2.37	2.07	1.97

Absorption Void Index Apparent Specific Mass

Fig.5 Water absorption (%), void index (%), and apparent specific mass (g/cm^3) of conventional and modified mortars

It is possible to state an increase in water absorption with the incorporation of micronized PET at both levels, compared to the reference mortar. For 28 days, the mortar incorporated with 5% PET showed an increase of 7.41% more than the reference mortar, and the mortar with 10% PET obtained an increase of 13.54% when compared to the reference mortar. The results corroborate previous studies carried out by Hannawi *et al*. [31] and Spósito *et al*. [5], where they verified an increase in the absorption of mortars after incorporating PET. However, Safi *et al*. [32] point to a decreasing effect of absorption of water by immersion with values below 4% of addition of PET in self-compacting mortar, and this behavior is related to the filling of voids in the cement matrix.

As the PET content in the mortar increased, the absorption also increased. This phenomenon occurs due to the hygroscopic characteristic of this residue – it absorbs water and does not release it. Furthermore, there is no chemical interaction between the polymer and the cementitious matrix. This mechanism generates residual porosity, thus increasing water absorption. The results found to corroborate the research by Modro [33], who evaluated the water absorption of Portland cement concretes incorporated with three types of recycled PET waste to replace mineral aggregates: PET film (or FI), PET sand (or AR) and PET flake (or FL). The author observed that for all concretes incorporated with PET waste, and there was an increase in water absorption as the percentage of replacements increased. Likewise, Almeida [34] observed in his study that by promoting the increase in the percentage of PET in all the concrete mixes studied, there was an increase in the value of water absorption due to the fineness modulus of PET being lower than that of sand and, consequently, PET has a greater surface area, contributing to increased water absorption.

As shown in Figure 5, the mortar with 5% PET, at 28 days, had a slightly higher void ratio, approximately 3.25%, compared to the reference mortar. This result is not very significant, but it is associated with increased water absorption concerning the reference mortar. The mortar with 10% PET incorporation showed similar behavior, but more significant, an increase of 18.68% compared to the reference mortar and 14.95% compared to the mortar with the addition of 5% PET The values were obtained to corroborate the data observed by Spósito *et al*. [5].

According to Modro [33], the increase in porosity or void index is related to the specific surface area of the residue; the more irregular the residue, the larger its specific area. Meneses [35] obtained in his study values of void and porosity indexes for concrete with the addition of PET very close to the reference concrete. The researcher attributed this result to the fact that the addition of fiber had little interference in the water/cement ratio of the mixture.

The incorporation of micronized PET reduced the accurate, specific mass of the mortar. This is because the specific mass of micronized PET is lower than the specific mass of sand, characterized in this research as 2.56 g/cm³. For the 5% replacement, the reduction at 28 days of the specific mass was 12.67%. For 10% replacement, the reduction at 28 days of specific mass was 16.88%. Therefore, a higher percentage of PET replacement causes a more significant reduction in specific mass. The specific mass data corroborate the research developed by Reis and Carneiro [6] and Spósito *et al*. [5], in which they verified a decreasing behavior of the specific mass as the content of PET incorporated into the mortar increases, both in the polymeric mortar and in the Portland cement-based mortar, respectively. Therefore, the higher the substituted content, the lower the specific mass. The same behavior was also verified by Hannawi *et al*. [31] in their study.

Determination of mechanical properties of mortars

Figure 6 shows the results of the simple compression test and modulus of elasticity of conventional and modified PET mortars.

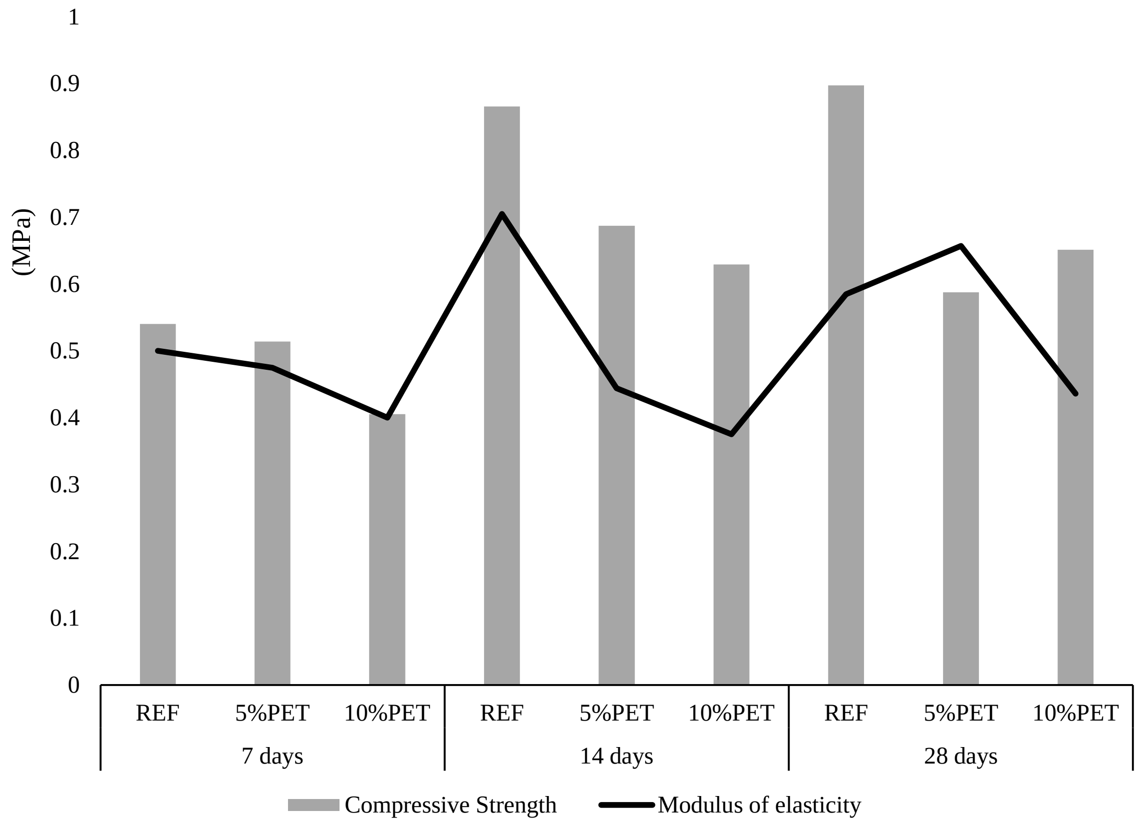


Fig.6 Simple compression strength and modulus of elasticity of the analyzed mortars

It is observed that the compressive strength values, at 28 days, were approximately the same for both contents used, which shows that over time, there is no significant variation between the contents of 5 and 10% when it concerns the compressive strength. According to Figure 6, it is observed that the micronized PET caused a reduction in the compressive strength, for both acceptable aggregate replacement contents, for all ages. For the 5% PET content, the strength was very close to the reference mortar at seven days, but at other ages, the reduction was more significant. At 28 days, there was a reduction of 34.5% for the mortar incorporating 5% of PET and 27.38% for the mortar with the addition of 10% PET compared to conventional mortar.

Spósito *et al*. [5] did not evaluate the mechanical strength of mortars incorporated with PET in the levels of 5 and 10%, and in this research, a reduction in these values was observed. According to Modro [33], the reduction in the compressive strength of Portland cement concretes incorporated with PET waste compared to reference concretes due to less chemical interaction between the polymer and the cement matrix and, mainly, due to lower intrinsic mechanical resistance of polymers concerning mineral aggregates, which have a much more excellent mechanical resistance.

According to Silva *et al*. [26], the reduction in resistance is because plastic aggregates have a smooth surface, are impermeable and less resistant, unlike natural aggregates, and may thus compromise the bond. Table 3, of the NBR 13281:2005 standard, classifies the mortars for laying and coating walls and ceilings according to their resistance to simple compression.

Table 3. Classification of mortars for laying and coating walls and ceilings in terms of compressive strength

Class	**Compressive strength at 28 days (MPa)**	**Method**
P1	≤ 2.0	
P2	1.5 a 3.0	
P3	2.5 a 4.5	NBR 13279:2005
P4	4.0 a 6.5	
P5	5.5 a 9.0	
P6	> 8.0	

Source: NBR 13281:2005

The compressive strengths at 28 days found in the tests were 0.588 and 0.652 MPa, for the incorporations of 5 and 10% of micronized PET, respectively. Therefore, as described in Table 3, both results are classified as mortars for laying and covering walls and ceilings, class P1.

According to Figure 5, it can be concluded that the incorporation of PET in percentages of 5 and 10% resulted in a reduction in the modulus of elasticity of the mortar at 7 and 14 days. On the other hand, it is observed that after 28 days, there was an increase in the modulus of elasticity of the mortar with 5% of PET about the reference mortar and the mortar with 10% of PET, with an increase of 12.3% and 50.7%, respectively.

Spósito *et al*. [5] observed that the dynamic modulus of elasticity decreased with the increase in the percentage of PET. Since the dynamic modulus of elasticity is influenced by the porosity of the transition zone, the modulus of elasticity of the cement matrix, and each material. Due to the poor adhesion between the cement matrix and PET, the greater porosity in the transition zone results in a material with less rigidity. Comparing the values at seven days and 28 days, an increase in the modulus of elasticity was observed in the mortar with 5% PET compared to the reference mortar. This increase may be related to the formation of hydrated cement products (CSH) and calcite from the reaction of lime hydroxides with carbon dioxide. These chemical reactions are formed over time, modifying the transition zone, filling the voids, and thus leading to a more rigid material [28, 36, 37].

4. Conclusion

The incorporation of PET residue caused an increase in the water absorption of the mortar under study. The specimens of mortars modified with micronized PET had a specific mass lower than the reference mortar, and the void ratio increased. This was due to the density of PET being lower than the density of commonly used sand. The PET resulted in a reduction in strength compared to the reference mortar. According to standard parameters, the coating and laying mortars incorporated with 5 and 10% micronized PET were classified as class P1. The modulus of elasticity of the modified mortars was lower than those obtained by the reference mortar, but at 28 days, the mortar with 5% PET had the highest modulus of elasticity. It is believed to be due to chemical reactions formed over time, modifying the transition zone, filling the voids, and thus increasing the rigidity in the material. Thus, the PET content that showed the best performance among the properties evaluated was 5%. Therefore, all the analyses carried out presented values within the specified norms, and even the compressive strength results were inferior to the reference mortar. These products show partial success, indicating the viability of PET in mortars. Furthermore, the use of waste in construction products helps to reduce the environmental impact caused by inadequate disposal since this waste has a high decomposition time in nature.

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Conflict of Interests

No potential competing interest was reported by the authors.

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