

Mineralogical Study of Polymer-Mortar Composites with PET Polymer by Means of Spectroscopic Analyses

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ABSTRACT

The sheer amount of disposable bottles being produced nowadays makes it imperative to identify alternative procedures for recycling them since they are non-biodegradable. Experimental investigation on the effects of polyethylene terephthalate (PET) polymer, which is a waste material obtained by crushing of used PET bottles, on the mineralogical composition of composites after 28 days of casting are presented in this paper. Various weight fractions of cement 2.5%, 5% and 7.5% were replaced by the same weight of PET plastic; they were then moulded into specimens and cured. The fine powder samples obtained from broken specimens were subjected to X-ray diffraction, FT-IR spectroscopy, differential thermal analysis, thermogravimetric analysis and the composites were also observed by optical microscope. Thermogravimetry (TG) and derivative thermogravimetry (DTG) were used to study the interaction between polymers and cements. Differential thermal analysis (DTA), X-ray diffraction and FT-IR were also used to investigate the cement hydration according to the additions. The results showed that an increase in polymer-cement ratio meets with a decrease in the quantity of Ca(OH)₂; in terms of bonding, the rough surface of particle favours greater contact between PET and cement matrix and doesn't seem to have chemical interaction between the mineral species and the organic molecules which could lead to the formation of new compounds. The present study highlights the capabilities of the different methods for the analysis of composites and opened new way for the recycling of PET in polymer-mortars.

Keywords: Composite; PET Polymer; Interaction; TG/DTG; DTA; X-Ray Diffraction; FT-IR

1. Introduction

Polymer-modified mortars have been popular construction materials because of their excellent properties in comparison with ordinary mortars. Polymers have been used for improving mechanical properties, adhesion with substrates, or waterproofing properties of mortars and concretes. The literature agrees that the properties of polymer modified mortar and concrete depend significantly on the polymer content or polymercement ratio, that is, the mass ratio of the amount of polymer solids in a polymer-based admixture to the amount of cement in a polymer-modified mortar or concrete [1-3].

Although extensive research has been done on recycled materials, there have been very few studies concerning lightweight concrete which incorporates waste products as aggregates. The self weight of concrete elements is high and can represent a large proportion of the load on a structure [4-6]. Therefore, using lightweight

concrete with a lower density can result in significant benefits such as superior load-bearing capacity of elements, smaller cross-sections and reduced foundation sizes. A lightweight structure is also desirable in earthquake prone areas.

Polyethylene terephthalate (PET) is one of the most common consumer plastics used and is widely employed as a raw material to realize products such as blown bottles for soft-drink use and containers for the packaging of food and other consumer goods. PET bottles have taken the place of glass bottles as storing vessel of beverage due to its lightweight and easiness of handling and storage.

In 2007, it is reported a world's annual consumption of PET drink covers of approximately 10 million tons, which presents perhaps 250 milliards bottles. This number grows about up to 15% every year [7]. On the other hand, the number of recycled or returned bottles is very low. Generally, the empty PET packaging is discarded by the consumer after use and becomes PET waste (WPET).

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The major problems that this level of waste production generates initially entail storage and elimination [8]. The recycling of PET bottles and the preservation of natural resources are priority items but to date, the recycling of PET bottles as a lightweight aggregate for concrete has not been studied because of the high melting cost [9].

Researches into new and innovative uses of waste materials are continuously advancing. These research efforts try to match society's need for safe and economic disposal of waste materials. The use of recycled aggregates saves natural resources and dumping spaces, and helps to maintain a clean environment [10-16]. Thus, this current study concentrates on the waste materials without any further transformation beyond crushing, in order to minimize final material costs. Specifically plastics waste (PET) is used as substitute for conventional materials, mainly cement, in polymer-mortar composites mixes.

Even though the main effects of some polymeric additives on the mechanical properties of mortars or concrete are well established, the mechanisms responsible for these effects are not yet fully understood.

Furthermore calcium hydroxide, Ca(OH)_2 , being one of the major phases in hydrated Portland cement, occupies about 20% to 25%, by volume, of the total mass [17]. It may affect the physico-mechanical properties of the hydrated cement to a considerable extent [18]. Additionally, the role which Ca(OH)_2 plays towards the engineering properties and durability characteristics of the systems based on hydrated cement is still a matter of debate among researchers. While studying the durability aspects of cement systems, many researchers have found Ca(OH)_2 being much more unstable in resisting the adverse environmental conditions than calcium silicate hydrate (CSH) gel [19]. Ca(OH)_2 is produced by the hydration of tricalcium silicate (C_3S) and dicalcium silicate (C_2S) phases of the cement. In the set Portland cement, Ca(OH)_2 is formed with well-defined crystallinity having a definite stoichiometry, which may be affected by the presence of additives and admixtures [17].

In the previous work, the author studied the effects of PET polymer on the mortar properties, specifically to decrease the chloride ion penetration depth and apparent chloride ion diffusion coefficient of polymer-mortar composites. This may be explained due to the reduced volume of large-sized pores and the improved resistance to the absorption of the test solutions with an increase in polymer-cement ratio [20]. In addition, Benosman *et al.*, [21] showed the improvement of the adherence strength and the resistance to aggressive solutions of composites using such additions. For this reason, aim of this work is to study the influence of such admixtures concerning the hydration of Portland cement by means of the mineralogical study of the composites with the same composition used in the mentioned works [20,21].

The interaction between polymers and cement Portland can be investigated through several techniques such as thermal analysis, X-ray diffraction and FT-IR. Thermogravimetry (TG), derivative thermogravimetry (DTG) and differential thermal analysis (DTA) are considered important tools for evaluating the nature of hydrated products according to different stages of cement hydration, in addition to quantifying the different phases [22-25].

When cement is hydrated, its main components are transformed into hydration products, mainly calcium silicate hydrate (C-S-H gel) and portlandite. The hydration can be evaluated by measuring the mass loss of hydrated compounds up to 900°C. The following peaks and temperature ranges have been studied when hydrated cement is heated in thermobalance and they are interpreted as described below [23,24]:

- ~100°C: dehydration of pore water,
- 100°C - 300°C: different stages of C-S-H dehydration,
- ~500°C: dehydroxylation of Ca(OH)_2 ,
- ~700°C: decarbonation of CaCO_3 .

This study reports the results of investigations in which methods of thermal analysis, TG, DTG and DTA, were applied to investigate the effects of polymer modification on the process of hydration of portland cement by estimating Ca(OH)_2 content and calcium hydrate content. X-ray diffraction and FT-IR were carried out to study the hydrate products of cement. Certain key proportions are also studied, in contrast with what has been undertaken in previous work [16,26] in order to determine feasibility limits. Also, the analysis by the optical microscope was carried to view the arrangement of PET particles in the cement matrix. The present study highlights the capabilities of the different methods for the analysis of composites and has shown quite encouraging results and opened new way for the recycling of PET waste in polymer-mortars.

2. Experimental

The mortar and/or mortar-polymer composites mixtures were prepared in collaboration with two laboratories: the Laboratory of Materials, Civil Engineering Department, ENSET Oran (Algeria) and the Laboratory of Polymer Chemistry, University of Oran Es-Senia, using the following materials:

2.1. Cement

The cement used was a blended Portland cement type CPI-CEM II/A (pouzzolanic cement) delivered from Zahana factory located in the western Algeria, chemical and physical properties of cement are shown in **Tables 1** and **2**, respectively, according to the manufactories. The chemical composition was obtained by using an X-ray

Table 1. The chemical composition of cement.

Chemical Composition	CPJ-CEM III/A (%)
Loss on Ignition	2.09
SiO ₂	21.82
Al ₂ O ₃	6.57
Fe ₂ O ₃	4.01
CaO	63.43
MgO	0.21
SO ₃	1.86
CaO free	0.24

fluorescence spectrometer analysis type OXFORD MDX¹⁰⁰⁰.

2.2. Polymer

PET is a thermoplastic polyester with tensile and flexural modulus of elasticity of about 2.9 and 2.4 GPa, respectively, tensile strength up to 60 MPa and excellent chemical resistance. It is a semi-crystalline polymer, with a melting point of about ~260°C (Figure 1). Its density (specific gravity) is around 1.3 - 1.4 g/cm³. The PET powder is obtained by finely crushing the drink bottles (waste plastics). After preliminary tests, polymer particles of size lower than 1 mm were used in this study. Figure 2 and Table 3 show the infrared spectra of PET in KBr pellet.

The analysis of PET was performed in the Laboratory of Applied Sciences of Cherbourg, University of Caen Basse Normandie, France, using DTA/TGA 92 Setaram

equipment. The experimental conditions were: N₂ gas dynamic atmosphere; heating rate (10 K/min); alumina top-opened crucible. The samples were heated in the range of 20°C - 350°C.

2.3. Sand

Crushed sand obtained from Kristel quarry in Oran, West Algeria was used. The chemical properties shown in Table 4 were obtained by using an X-ray fluorescence spectrometer analysis type OXFORD MDX¹⁰⁰⁰.

2.4. Experimental Program

Four mixtures were prepared as described in Table 5,

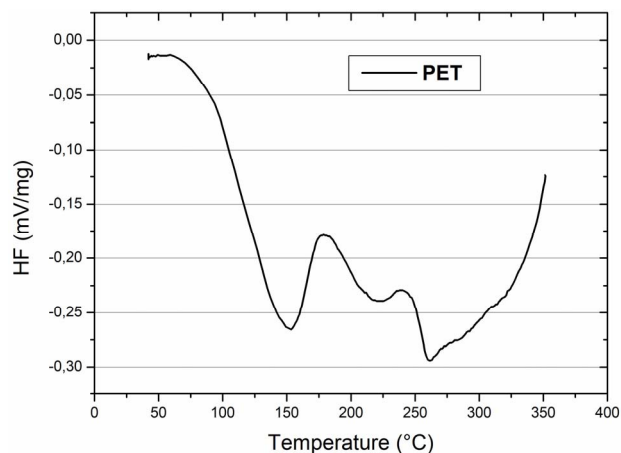


Figure 1. DTA curve at 10 K/min of the polyethylene terephthalate PET.

Table 2. Physical properties of cement.

Setting Time (min)		Blaine Surface Area (cm ² /g)	Absolute Density (g/cm ³)	Compressive Strength (MPa)	
Initial	Final			28-days	
120	200	2987	3.09	32.5	

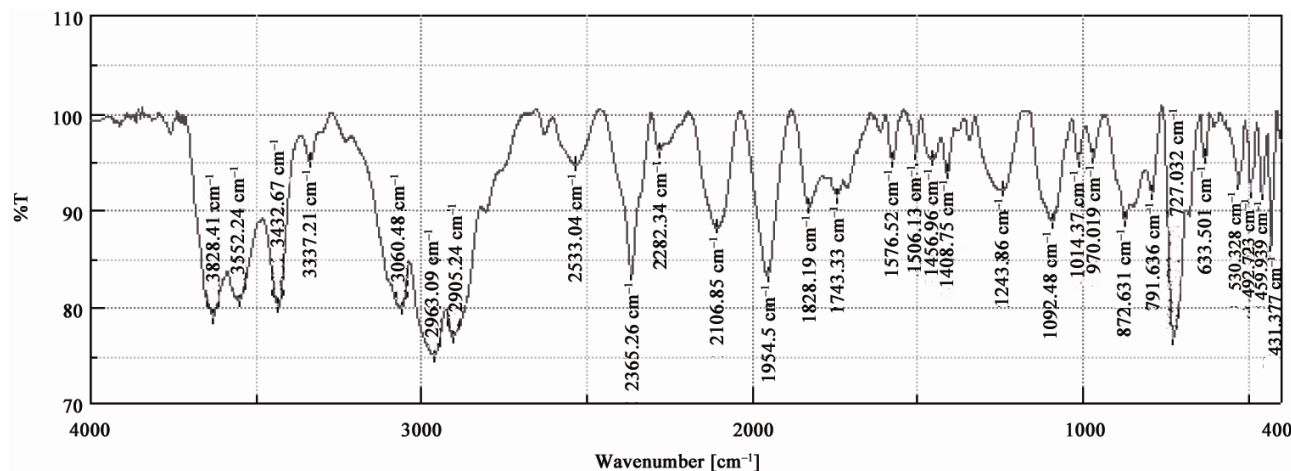


Figure 2. Fourier-transform infrared spectrum of PET polymer in KBr pellet.

Table 3. Fourier-transform infrared table of PET polymer in KBr pellet.

Bands identification	
(cm ⁻¹)	Group
872.63	Alternating deformation of benzene, out of the plane (para-disubstituted)
1092.48	-C-O
1576.52 - 1660.2	C=C, aromatic character
1743.33	-COO-
2963.09 - 2905.24	-CH ₂ -
3060.48	CH, benzene group

Table 4. The chemical composition of sand.

Chemical Composition	Sand (%)
Loss on ignition	43.83
SiO ₂	0.77
Al ₂ O ₃	0.11
Fe ₂ O ₃	0.36
CaO	54.71
MgO	0.21
SO ₃	Nil
Chlorures	Nil

which are the composites with the same proportions used in the previous works [20,21], as explained in the introduction. The materials were weighed and mixed in a planetary type mortar mixer. The mortar mixes had proportions of 1 binder: 3 Sand (by weight). The binder consisted of cement and polyethylene terephthalate. The water to binder ratio was kept constant at 0.5. The physical properties of the pastes of mortars were determined in accordance with EN 196-3 [27].

The preparation for TG/DTG, DTA, X-ray diffraction and FT-IR analysis was carried out using agate crucible, in which the mortar or composite was manually ground until the size of particles was lower than 0.160 mm. For the prevention of carbonation and maintenance of relative humidity, all specimens were stored in the vacuum up to the time when the test started.

The analyses were performed in the Laboratory of Polymer Chemistry, University of Oran, using Labsys TGA SETARAM Thermogravimetric Analyzer equipment, easy to use (ambient to 1600°C). The experimental conditions were: He gas dynamic atmosphere; heating rate (10°C·min⁻¹). The samples were heated in the range of 20°C - 1100°C at a constant rate. The Ca(OH)₂ and calcite were estimated from the weight losses measured in the TG curve between the initial and final temperature of the corresponding TG peak.

The composites were submitted to thermal analysis in a DTA equipment, type Linseis mark DTA L62, in the Laboratory of Materials, ENSET Oran. The experimental

conditions were 1) continuous heating from 25°C to ~1100°C; 2) heating rate: 10°C/min; 3) alumina, top-opened crucible. DTA curves were obtained.

XRD was used to identify the polycrystalline phases of cement and hardened cement paste by means of the recognition of the X-ray patterns that are unique for each of the crystalline phases. The qualitative XRD investigation was performed in a Philips PW 1710 X-ray diffractometer with Cu K α radiation and 2 θ scanning, ranging between 5° and 80° of 2 θ .

The composites were analyzed by an optical microscope, type Keyence VH-5911 commissioned by Microvision software, available at the Laboratory LMDC of INSA, Toulouse, France.

A FT/IR-4200 Type A, Photometrec Fourier-transform infrared spectrometer was used, and the mortar/composite cement were analyzed in KBr pellets. The spectra were traced in the range of 4000 - 400 cm⁻¹ (wave number), and the band intensities were expressed in transmittance (%).

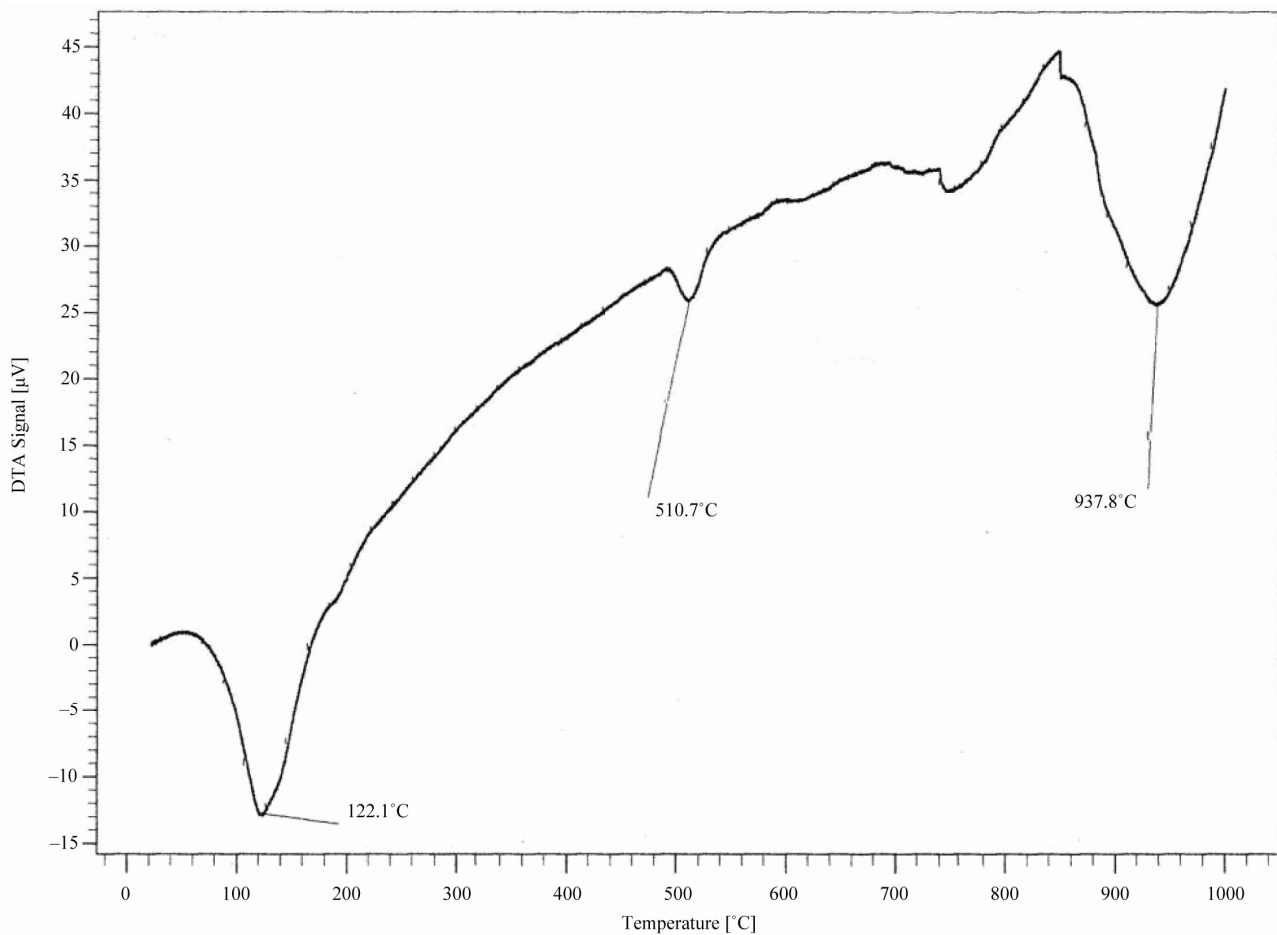
3. Results and Discussion

3.1. Thermal Analysis (DTA, TG/DTG)

The DTA and TG curves obtained in all of the tests are typical of hydrated cement composites containing carbonate phases. Three major endothermic reactions occurred during the heating of the samples: 1) release of the evaporable and part of the adsorbed water (CSH and ettringite) at 110°C approximately; 2) Ca(OH)₂ dehydration, between 450°C and 510°C; 3) decomposition of the carbonate phases at ~850°C - 900°C (**Figure 3**). As shown in **Figure 4**, PET polymer strongly influences the DTA curve, causing 1) enlargement of the exothermic shoulder at ~200°C - 420°C; 2) decreasing of the endothermic peak intensity and weight loss on the dehydration of calcium hydroxide; 3) sharp changes in the curves at temperatures higher than 510°C (in most cases, an endothermic peak at 580°C and three exothermic shoulders at 820°C - 840°C, 620°C - 660°C and 540°C were detected); 4) enlargement of the endothermic peak intensity at 620°C - 780°C and decreasing of the peak temperature

Table 5. Mix proportions and physical properties of polymer-mortar composites.

Mix Design	Polymer-Cement Ratios (%)	Water Demand for Standard Consistency (%)	Setting Time (min)		Density (g/cm ³)
			Initial	Final	
/					
PET 0	0	24.5	120	200	2.28
PET2.5	2.5	25	125	205	2.23
PET5.0	5.0	25.5	130	210	2.22
PET7.5	7.5	26	145	225	2.21

**Figure 3. DTA curve at 10°C/min of an unmodified mortar PET0.**

(from about ~937°C to ~926°C); and e) decreasing of the weight loss in the latter temperature range. These results comply with an early study of Silva *et al.* [28], in which EVA copolymer strongly influences the DTA/TG curves.

Figure 5(a) shows the TG curves of mortar without polymer PET0. It can be seen that TG/dTG curves for this mortar consist of four zones:

- ~40°C - 123.3°C: dehydration of pore water,
- ~123.3°C - 420°C: dehydration of calcium silicate hydrates,
- ~420°C - 510°C: dehydroxylation of calcium hydroxide,
- ~840°C: decarbonation of CaCO₃.

Figure 5(b), Figures 6(a) and 6(b) show TG curves of composites with PET polymer addition of 2.5%, 5% and 7.5% (polymeric solids). The TG curves obtained in these tests are typical of hydrated cement composites containing carbonate phases and PET polymeric admixtures influences. As it is shown, the curves can be divided into five major parts, according to different reactions:

- ~123.3°C: dehydration of pore water,
- ~123.3°C - 345°C: dehydration of calcium silicate hydrates,
- ~490°C - 517°C: dehydroxylation of calcium hydroxide,
- ~650°C - 780°C: Enlargement of the endothermic peak

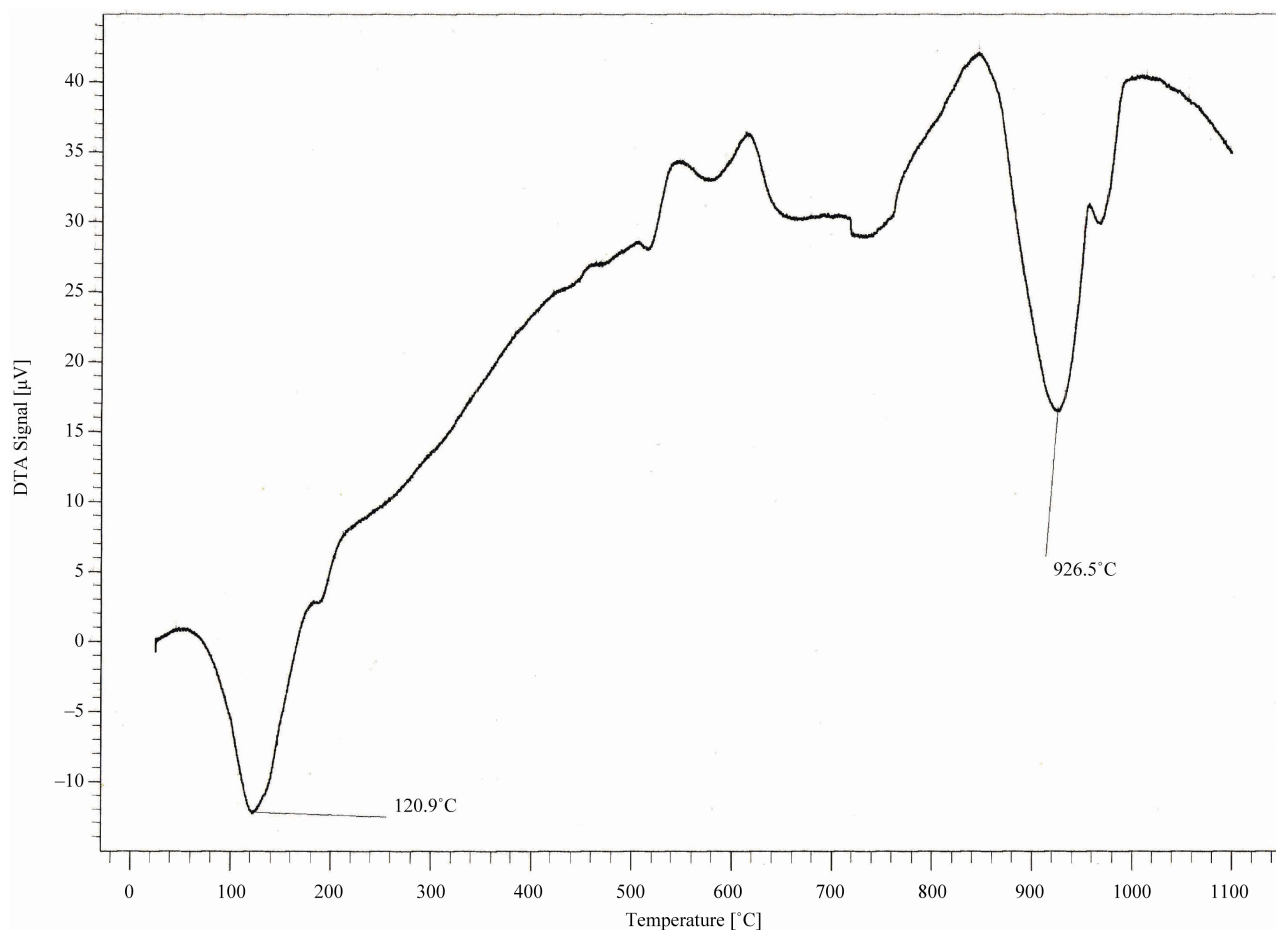


Figure 4. DTA curve at 10°C/min of the composite PET5.

intensity; as it was found from the DTA analyses.

~800°C: decarbonation of CaCO_3 .

All the weight loss data are expressed as a function of the ignited weight of the sample, as suggested by Taylor [29]. The calcium hydroxide content was determined from the following equation:

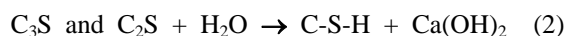
$$\text{CH}(\%) = \text{WL}_{\text{CH}}(\%) \times \frac{\text{MW}_{\text{CH}}}{\text{MW}_{\text{H}}} \quad (1)$$

where $\text{CH}(\%)$ is the content of $\text{Ca}(\text{OH})_2$ (in weight basis), $\text{WL}_{\text{CH}}(\%)$ is the weight loss occurred during the dehydration of calcium hydroxide (in weight basis), MW_{CH} is the molar weight of calcium hydroxide and MW_{H} is the molar weight of water. Since the exact stoichiometry of decomposition reactions of the carbonate phases is not known, the results are expressed in function of the weight of CO_2 gas released during the decomposition, and not as carbonate phase's content.

When one adds the PET to the composite there is a diminution of the calcium hydroxide (CH) content and an increase of the dehydration temperature, as can be seen in **Figures 7(a)** and **(b)**. Also, Silva *et al.* [28] demon-

strated that EVA polymer strongly reduces the CH content. **Figure 8** shows that PET polymer sharply decreases the quantity of carbonate phases in the composites, measured by TG/DTG.

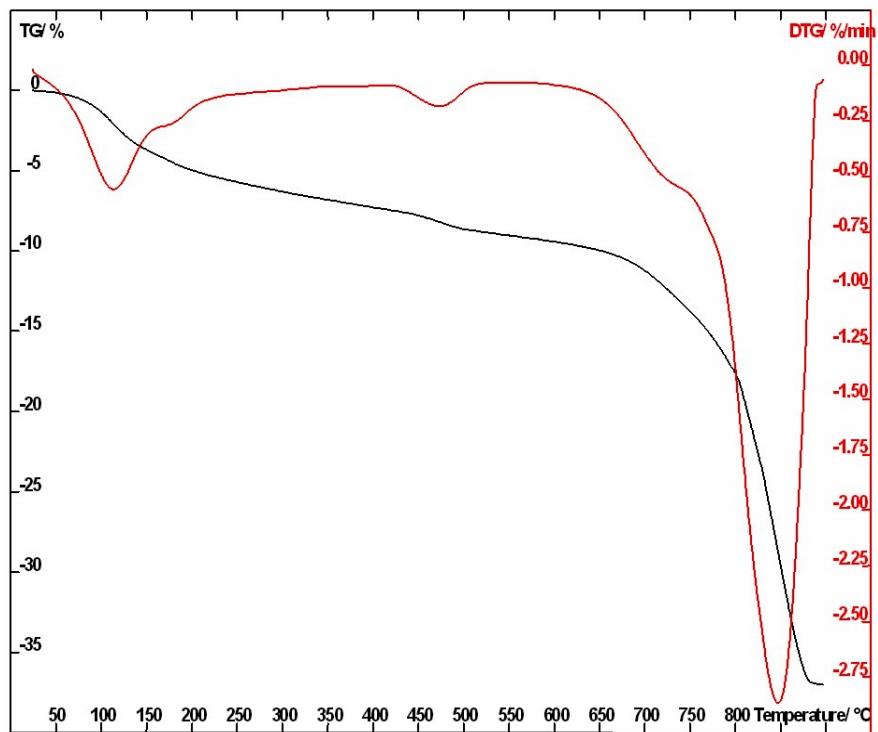
Because when the rate of substitution of PET increases, there is less formation of portlandite $\text{Ca}(\text{OH})_2$, which is mainly the result of the following reaction:



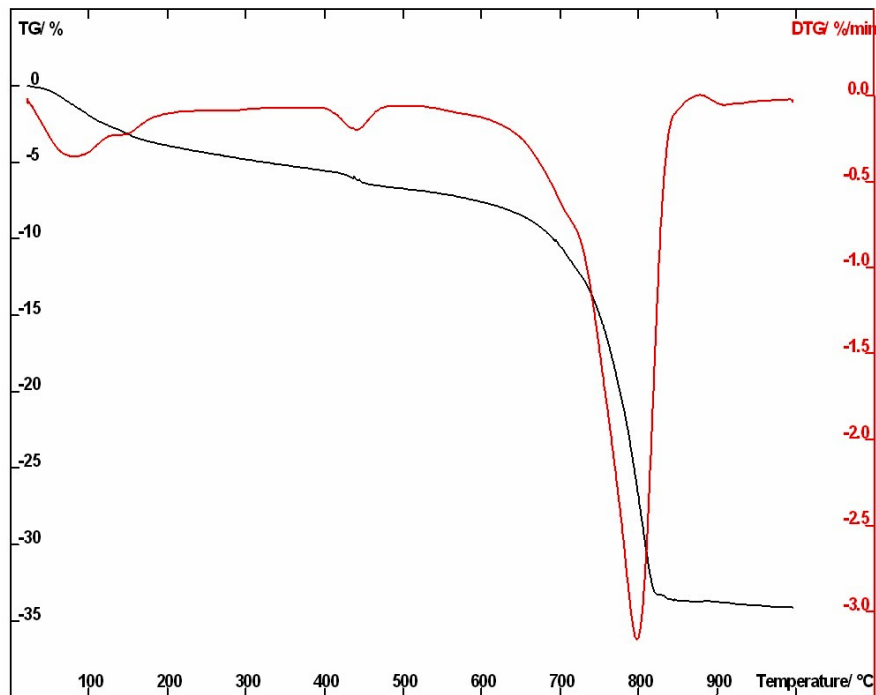
3.2. XRD Analyses

The XRD results show some qualitative differences in the hydration rate due to the incorporation of PET polymer. **Figure 9** shows the X-ray patterns of the composites with 7.5% of polyethylene terephthalate, and composites without polymer PET0. The main compounds observed are $\text{Ca}(\text{OH})_2$ in the form of portlandite, a big amount of CaCO_3 resulting from carbonation of $\text{Ca}(\text{OH})_2$ and calcium silicate anhydrous. The peak intensity in the region $2\theta = 18^\circ - 18.1^\circ$ [30,31] has been considered as a measure of the quantity of $\text{Ca}(\text{OH})_2$ [32].

So, it is also noted here that at a polymer-cement ratio of 7.5% a slight increase in the peak intensity compared



(a)

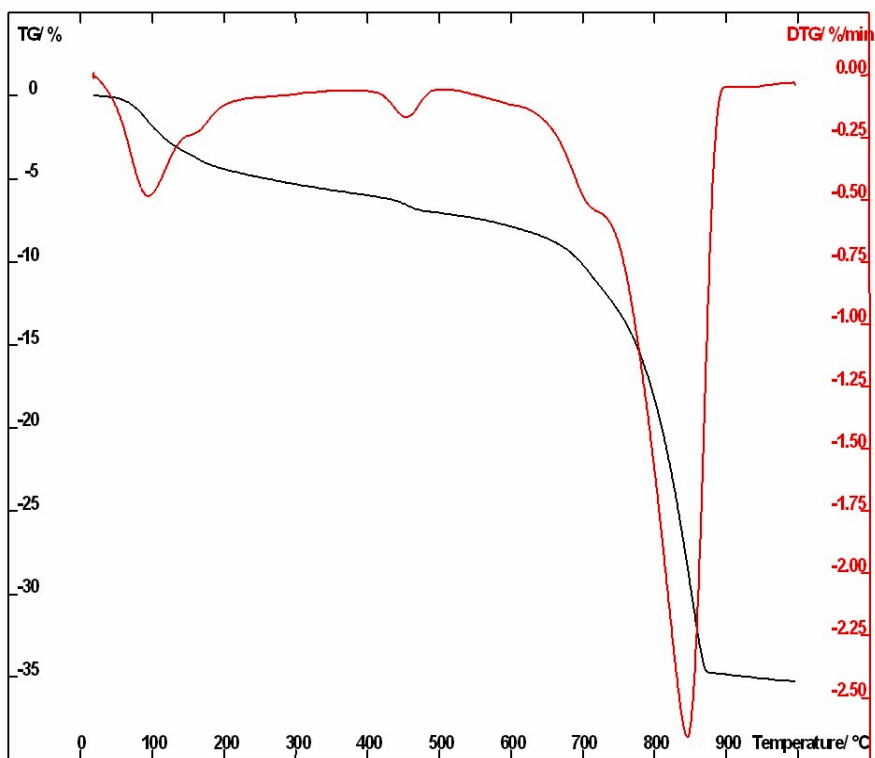


(b)

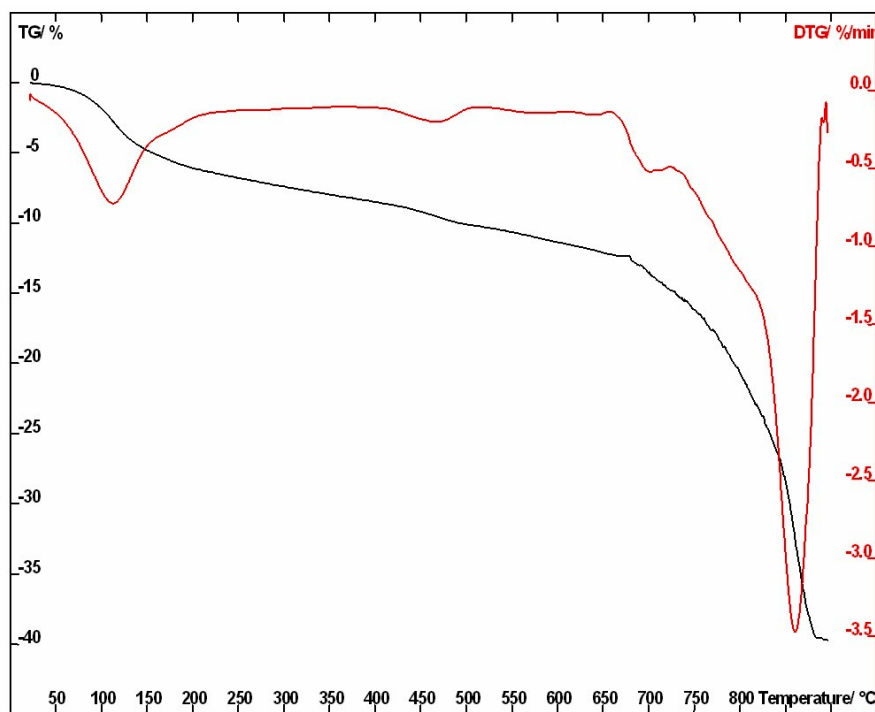
Figure 5. TG/DTG curves at $10^{\circ}\text{C}\cdot\text{min}^{-1}$ of unmodified mortar CPI-PET0 (a) and the composite PET2.5 (b).

to the unmodified mortar is observed. Further, $\text{Ca}(\text{OH})_2$ crystals may possibly produce sharper reflection in the presence of PET polymer due to a change in the orientation pattern of the crystals. This sharper reflection offsets

the effect gained by the lowered quantity of $\text{Ca}(\text{OH})_2$, and so the peak intensity at a polymer-cement ratio of 7.5% appears at an increased level compared to that of the unmodified mortar. These results are in line with



(a)



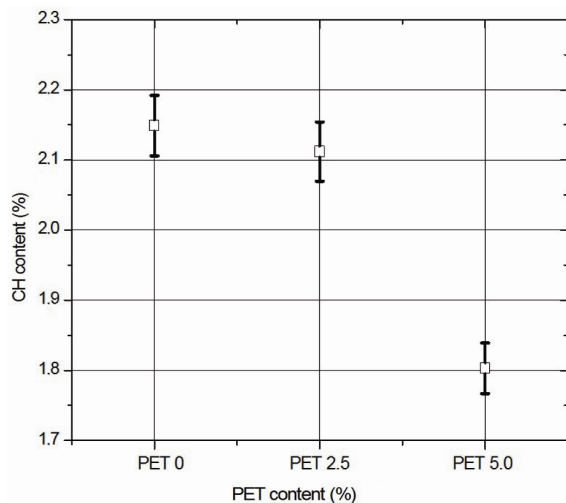
(b)

Figure 6. TG/DTG curves at 10°C·min⁻¹ of the composite PET5 (a) and the composite 7.5 (b).

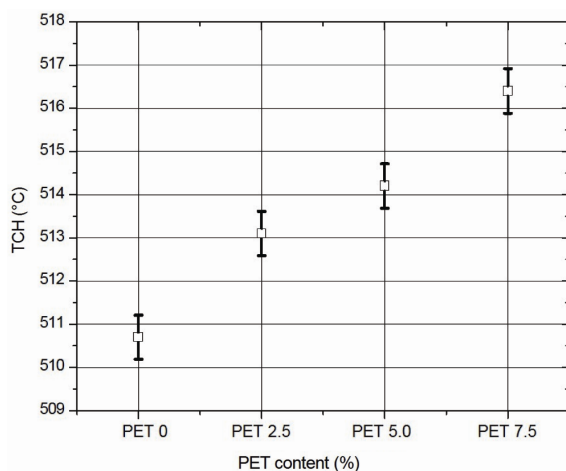
those of some previous research's [19,26,31].

On the basis of the experience gained by DTA/TGA analysis, it is possible to explain this variation in terms of

the fact that the PET addition causes a progressive decrease in the amount of portlandite Ca(OH)₂ in the composites compared to unmodified mortar.



(a)



(b)

Figure 7. Effect of PET content on the calcium hydroxide content (CH) (a) and its dehydration temperature (TCH) (b).

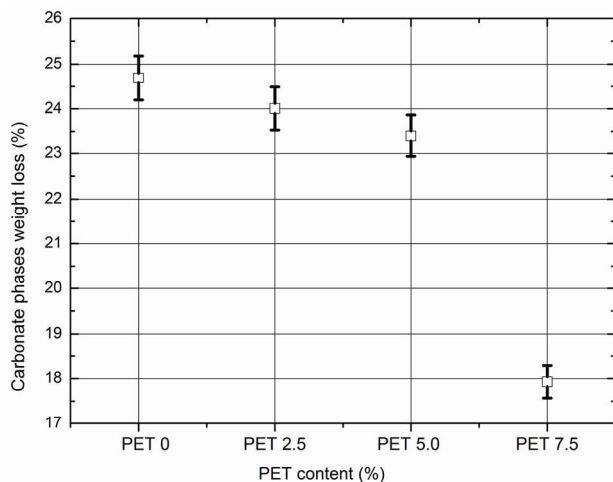


Figure 8. Effect of PET content on the weight loss due to carbonate phases' decomposition.

3.3. The Optical Microscope Results

Figure 10 shows optical micrograph of the composite containing the PET particles. In terms of bonding, the rough surface of particle favours greater contact between PET and cement matrix. The particles appear well covered by cement matrix. Furthermore, Benazzouk *et al.*, [33] found that there is a good adherence between rubber particles and cement matrix. So, the introduction of the PET addition leads to a densification of the cement matrix, which serves to improve material strength and durability [21,34].

3.4. FT-IR Analyses of Hydrated Cement and PET Polymer

The FT-IR spectra of the PET0 and PET7.5 composite cement hydrated up to 28 days and PET polymer are presented in **Figure 11**. The major changes of the FTIR spectra in the hydrated cement are:

Calcium hydroxide bands ($\sim 3638\text{ cm}^{-1}$) and also for the free OH groups, combined and adsorbed water of C-S-H, AFm and AFt phases (3451.96 cm^{-1}), molecular water ($3440 - 3446$ and $1640 - 1654\text{ cm}^{-1}$), carbonate phases (~ 1427 , 874.56 and 713.5 cm^{-1}). The broad band at $\sim 1020 - 1018\text{ cm}^{-1}$ arises from C-S-H vibrations, in agreement with those reported by Martinez-Ramirez [35]. As can be seen in **Figures 2, 11** and **Table 3**, PET polymer doesn't cause any modifications in the spectra profile.

The PET0 and PET7.5 composite have the similar IR spectra (**Figure 11**). So, there isn't any chemical interaction between the mineral species and the organic molecules which could lead to the formation of new compounds, in agreement with XRD analysis.

4. Conclusions

The following conclusion can be drawn from the obtained experimental data:

From the thermogravimetric investigations performed, showed in the TG/DTG and DTA curves, it is possible to conclude that polymeric additions have influenced the cement hydration.

The qualitative XRD investigation revealed that a lower intensity of $\text{Ca}(\text{OH})_2$ (in the region $2\theta = 18^\circ$) was obtained in the presence of PET, compared to mortar without polymer. Similarly, we found a decrease in the $\text{Ca}(\text{OH})_2$ content in the TG analyses for the modified mortar with polymer addition. As it can be seen, composite with 5% polymer content presented the lowest $\text{Ca}(\text{OH})_2$ compared with the other composites.

The FT-IR analyses exhibited that there isn't any chemical interaction between the mineral species and the organic molecules which could lead to the formation of

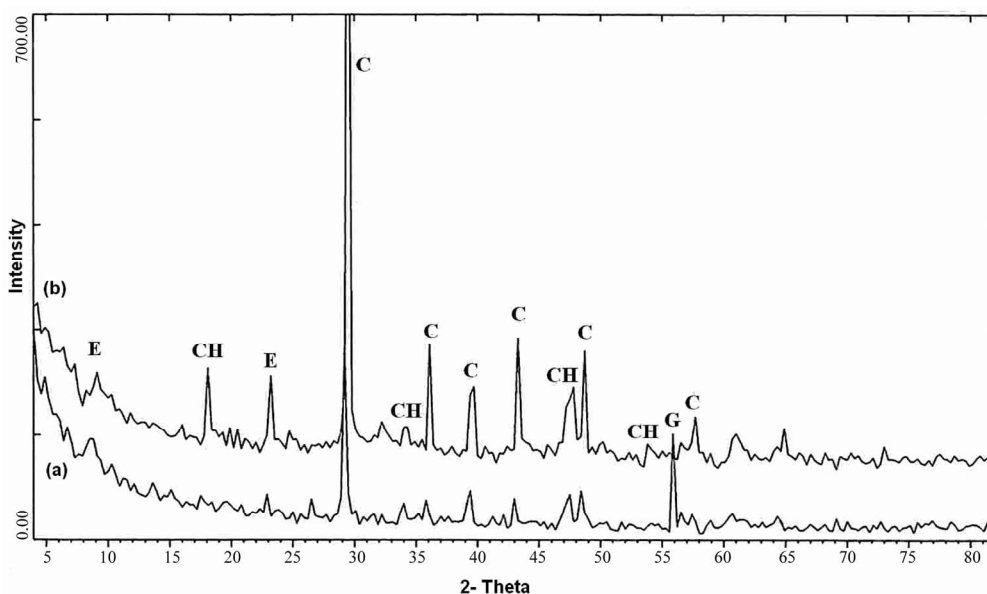


Figure 9. XRD patterns of the PET0 (a) and PET 7.5 (b) composites after 28 days of Hydration. CH, portlandite ($\text{Ca}(\text{OH})_2$); C, calcium carbonate (CaCO_3); E, ettringite ($\text{Ca}_6[\text{Al}(\text{OH})_6]_2(\text{SO}_4)_3 \cdot 26\text{H}_2\text{O}$); G, gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$).

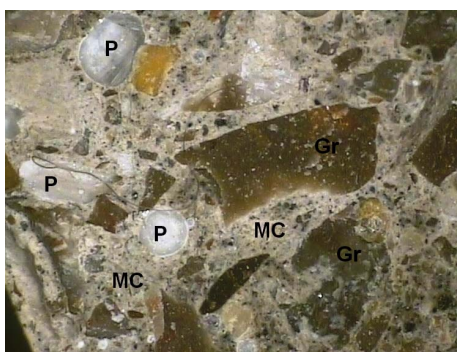


Figure 10. Optical micrograph of composite containing the PET particles at 360 days—adherence of particle additives to the matrix (magnification X25). P-PET, Gr-aggregate, MC-cement matrix.

new compounds, in agreement with XRD analysis.

In terms of bonding, the rough surface of particle favours greater contact between PET and cement matrix. The particles appear well covered by cement matrix. So, the introduction of the PET addition leads to a densification of the cement matrix, which serves to improve material strength and durability.

The application in civil construction of cement composite-based PET wastes appears to be feasible considering the results obtained from analysis of its properties. This study contributes toward the program of PET wastes recycling and pollution reduction. To conclude, studies on the durability of the composite when exposed to aggressive environment, like resistance to acid, sulfate at-

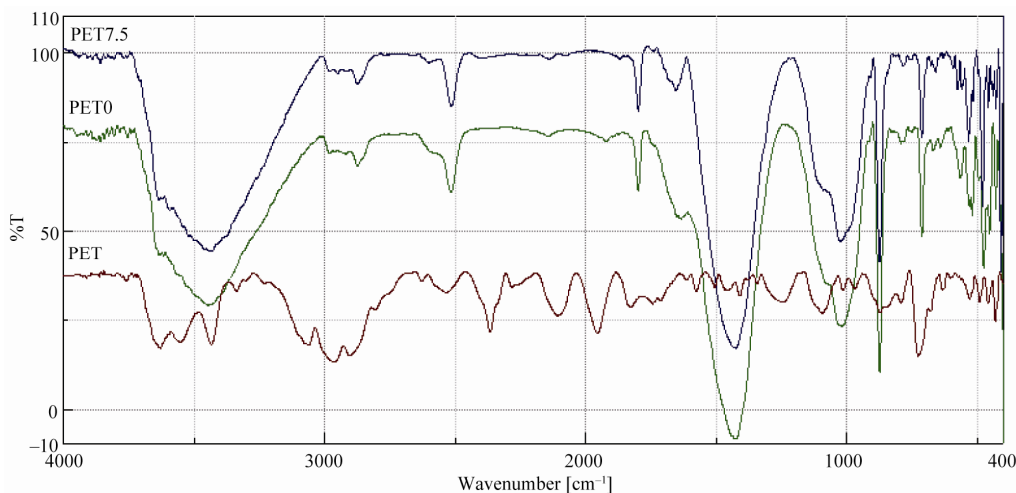


Figure 11. FT-IR spectra of PET0 and PET7.5 composite cured for 28 days and PET polymer.

tack and corrosion have started and displayed encouraging first results.

The durability of the concrete is an important engineering property. It determines the service life of concrete structures very significantly. Some conclusions were stated by the author in his previous study [20,21]:

- The resistance to acids solutions explained by the mass loss of polymer-mortar composites exposed to HCl and CH₃COOH solution were lower than those of unmodified mortar.
- The basic solutions are harmless to composite materials.
- The resistance to chloride ion penetration was improved [20].

Such mortars can be recommended as effective materials for preventing the chloride-induced corrosion of reinforcing steel in various concrete structures and towards the chemical resistance to typical reagents for industrial uses.

5. Acknowledgements

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