

Effect of Polymer Waste Mix Filler on Polymer Concrete Composites

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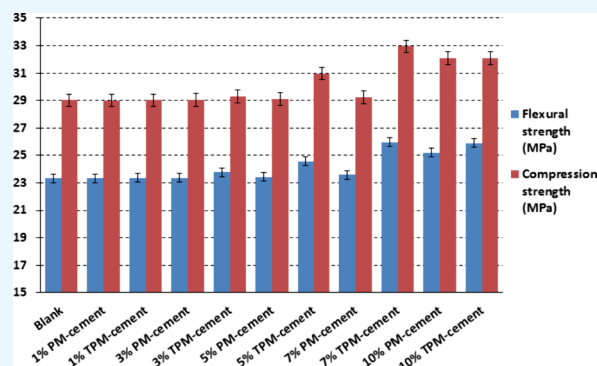
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ABSTRACT: The hazards of polymer waste and emitted gas on the environment pose a global challenge. As a trial to control this, the current work aims to reuse the polymer waste mix (PM) as fillers in calcium silicate to prepare new composites of environmentally friendly polymer concrete. PM was first subjected to treatment to obtain treated PM (TPM) and then was filled in new dicalcium silicate cement with different concentrations. The microstructural characterizations declare the successful preparation of the dicalcium silicate base material. After the curing reaction, the precipitated carbonate main product is responsible for the gained properties. The CO₂ uptake% in the proposed composites reached 16.6%, referring to the successful storage of CO₂ gas during curing. The treatment reaction led to an increase in the flexural and compression strengths due to the strengthening of the polymer waste mix–cement interface; the strengths were increased gradually with more contents of TPM fillers. 7% TPM–cement concentration achieved the highest flexural strength and compression strength of 10.2 and 12.7%, respectively, compared with blank cement. The used polymer improved slightly the pull-off force of the prepared cement, and 7 and 5% TPM–cement composites have the maximum values. All the proposed composites passed the impact testing without failure, where the combination between the polymer waste and silicate cement resulted in a stable composite surface. Compared with the blank, the different concentrations of TPM–cement composites show more stability against water absorption. In addition, the proposed composites and blank cement have a very low carbon dioxide emission. The ability to recycle the polymer waste, form new type of low-energy silicate, improve the mechanical and surface properties, uptake CO₂ gas, and reduce gas emission makes the proposed polymer waste mix–cement composites as environmentally friendly construction products.



1. INTRODUCTION

Plastics are considered as one of the growing industries which produce huge waste of most man-made products. Therefore, recycling of plastics and polymeric materials has had a global interest due to their increased production and widespread uses. There are serious environmental concerns for the disposal of postconsumed plastic waste, either stored in landfills or burnt, that increases ozone depletion due to the emitted toxic gases. Because of the nondegradability of plastics, they increase the environmental drawback in addition to the hazardous effect on soil and waterways. Some ways of recycling are being encouraged and developed to exploit this large volume of waste and also to keep the desired properties of recycled polymers as much as possible.^{1–4} As the usage of plastics has been increased dramatically in daily life in the last decades, it is reported that about 8300 million tons of virgin plastics was manufactured. Therefore, the amount of plastics accumulated in the environment is sharply increased; about 12,000 million tons is expected to be produced by year 2050.^{5–7} Waste valorization, including polymers and other solid waste, is one of the trending research areas which has been drawing

attention in the previous few years. Specifically, the valorization of polymers for producing new sustainable products is developed. Combination of waste, low-carbon, low-energy, and natural byproducts for making polymeric and non-polymeric composites can provide specific advantages.^{8–11} Polymers have numerous superior properties compared with other materials, so the ability to exploit their waste in developing materials and new products was discussed.^{12–14} The thermoplastic polymers serve as promising materials owing to their good mechanical and physical properties. Types of thermoplastics, such as polyethylene terephthalate (PET), polyethylene (PE), polyvinyl chloride (PVC), poly(methyl methacrylate) (PMMA), and others, are recently applied with different forms in the fields of composites, pipelines,

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electronics, aerospace, food and water packaging, microfluidics, optical materials, etc.^{15–18} PVC and PET are widely used thermoplastic polymers and have daily multipurpose applications. Their abundant waste can be taken as effective environmental friendly fillers to produce materials conserving natural resources and reducing greenhouse gas emission.^{19–21}

On the other side, cement and construction composites have great interest due to the different applications and benefits they provide. One of the recycling methods is the use of polymer waste, solid byproducts, natural waste, and bricks in construction materials, such as composite blocks and lightweight partitions, to share with the waste management.^{22–24} Several features, such as lightweight, high strength and elasticity, easy processing, corrosion resistance, low cost, and weathering resistance, make them alternatives to commercial aggregates in modern building and low-density construction applications.^{25–28} Composites are included in different forms and types due to the durability and improved properties, such as reinforced concrete, reinforced plastic, cement, polymer–cement composite, fiber–cement composite, and fiber–polymer composite.^{29,30} The global consumption of plastic in the building and construction markets is expected to be duplicated in 2027 with more intense studies. The influence of adding waste powders of concrete bricks and recycled polyethylene terephthalate on mortar was studied in novel improved construction composites. The compressive and flexural strengths were increased by 79 and 80% after curing the 30% hybrid formula. The powder hybrid showed reduced water absorptivity and chloride permeability coefficient due to the presence of effective fillers in the durable composites.³¹ The thermomechanical processing technique of recycled high-density polyethylene was studied depending on the given extrusion parameters; the mechanical properties and changes in the polymer structure were investigated after processing.³² The recycled ethylene–vinyl acetate was used as a second filler to cement, in addition to silica filler, with different concentrations. The hybrid material resulted in mitigating the mechanical strength reduction and improvement of ductility and waterproofing.³³ Aggregated scraps of PVC pipes were used as a partial additive to concrete. With regard to mechanical properties and unit weight, the 50 and 70% concentrations were found to follow the category of structural lightweight concrete.³⁴ Novel composites, based on high-strength concrete and plastic waste aggregate, were used for desired flanges and webs. The obtained products were applied as T-beams for flexure, shear force, and shear connection between the flange and the web at the section interface.³⁵ In many cases, the difference in the nature of the cement base and polymeric filler may cause unstable polymer–cement interface, so the prepared composites have undesired properties. To avoid this weakness, some minor treatments are performed on the reactants, either recycled or virgin, in different types of polymeric composites. Coupling materials, acids, plasticizers, nanofillers, heating, and radiation are the common methods for the targeted treatment. These treatments are expected to reinforce the attachment between the polymer and cement, after adding the active functional groups to the polymer filler. The mechanical and physical properties of the obtained composite are developed consequently.^{36–38}

It is well known that the preparation of the cement component itself is a high energy-consuming process. We consider a new type of cement base, as a nonhydraulic type. The nonhydraulic cement is considered as a low-energy

cement, compared with the traditional Portland cement. The curing of this concerned type is based on the carbonation mechanism where the concentrated CO₂ gas could diffuse the pores of cement silicate in the presence of a warm humid environment. The carbonation reaction gives carbonates which enrich the mechanical and physical properties.^{39–41} In connection with the polymer cement composites, the main work focuses on the usage of polymers in the form of fibers and aggregates. Research conducted on the exploitation of the polymer waste mix with low-energy silicate is inadequate. With this regard, herein, we investigate the formation of a new environmentally friendly polymer cement composite based on the polymer waste mix and low energy dicalcium silicate as filler and base materials, respectively, for the first time. The reactants were prepared, and then the composites were cured via carbonation. We try to collect the advantages of low-energy cement and the PET/PVC waste mixture, where PET and PVC wastes are difficult to separate. The physical and mechanical properties are studied, and the CO₂ uptake is investigated, maximizing the environmental friendly impact.

2. RESULTS AND DISCUSSION

2.1. Carbonation. The curing reaction of the prepared dicalcium silicate is discussed here. Figure 1 illustrates the XRD, FTIR, and TGA data, as usually performed for proofing the curing. As noted in Figure 1a, the obtained XRD patterns typically characterize the structure of C₂S after the performed sintering reaction. The related peaks show the combination of the γ -C₂S and β -C₂S phases. In Figure 1a, the change in the patterns after the curing reaction and the appearance of the peaks characteristic to the crystalline calcium carbonate and C–S–H (specifically, the broad peak near 30° and small peak at 50°) confirm the curing process and the uptake of CO₂ gas.^{42,43} γ -C₂S itself cannot react with water, where it is carbonated to CaCO₃; however, β -C₂S could have an initial hydration reaction to form C–S–H which might be indicated by the peak near 30°. Similar literature reported the curing of dicalcium silicate and the formation of the CaCO₃/C–S–H products which supported the improved properties.^{44,45}

The FTIR analysis is illustrated in Figure 1b. Generally, the main peak in the range of 800–1000 cm⁻¹ characterizes the vibrations of Si–O bonds in pure silicate. The peaks at 850 and 922 cm⁻¹ indicate the asymmetrical stretching of Si–O bonds in the obtained C₂S cement material.⁴⁶ The higher wavenumbers after the curing reaction are attributed to the curing process. The absorption peaks in the range of 870–1200 cm⁻¹ characterize the asymmetrical stretching of Si–O bonds present in the silicate–calcium carbonate. The overlapping band at 875 cm⁻¹ and the broad one at 1440 cm⁻¹ characterize the bending and stretching of the C–O bond in carbonate, respectively.⁴⁷ Overall, the peaks appear in the range of 870–1500 cm⁻¹, confirming the curing reaction. The observed microstructural data ensure the preparation of the calcium silicate base material and the further conversion to carbonate/C–S–H during the curing reaction. The precipitated products are responsible for the improved properties.

The TGA measurement is indicated in Figure 1c. We notice two distinct peaks. The first is a minor shoulder peak observed at 270 °C. This peak is attributed to the thermal decomposition of C–S–H produced after curing the β -C₂S phase through the hydration reaction. The second peak, as the main sharp peak at the range of 650–770 °C, is due to the decomposition of CaCO₃ produced after curing. Actually, the

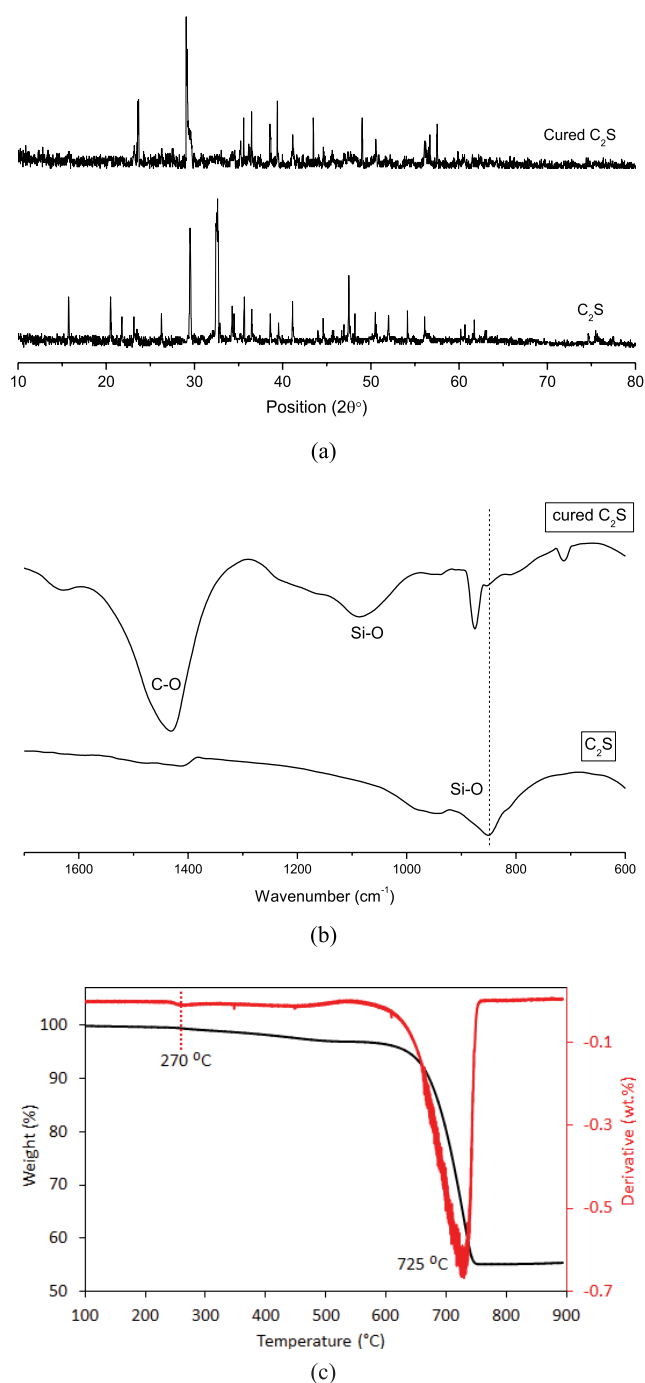
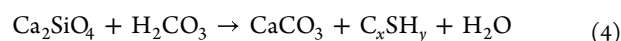
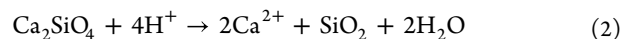
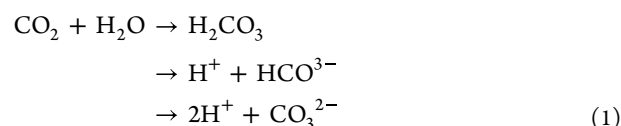


Figure 1. XRD (a), FTIR analysis (b), and TGA (c) of dicalcium silicate.

major carbonate product was precipitated during the curing of the γ -C₂S phase through the carbonation reaction. Some related literatures have reported a similar mechanism.^{48,49} Overall, the prepared γ -C₂S and β -C₂S phases were subjected to curing to give the main carbonation product and minor hydration product.

2.2. Curing Mechanism. The concentrated carbon dioxide gas reacts with the prepared dicalcium silicate cement according to the carbonation conditions given in Section 3. CO₂ gas is converted to a hard carbonate phase, where the gaseous phase is diffused inside the silicate interfacial. This new mechanism restricts the additional release of CO₂ gas in air

and reduces the related greenhouse gas emissions. The formed carbonate is responsible for the enhanced mechanical performance certainly.^{50,51} The two obtained forms of dicalcium silicate, γ -C₂S and β -C₂S, were cured according to the following mechanisms. In the presence of humidity and warm temperature, CO₂ gas is converted to carbonic acid (H₂CO₃) which is ionized to bicarbonate (HCO₃⁻) and finally to carbonate (CO₃²⁻) anions, as shown in eq 1. Simultaneously, with this reaction, the prepared dicalcium silicate cement is dissociated to calcium cations (Ca²⁺) and a silica gel trace, as seen in eq 2. Eq 3 indicates the final hard calcium carbonate (CaCO₃) product which is precipitated through the reaction between CO₃²⁻ and Ca²⁺ obtained from eqs 1 and 2.^{52–54} Extra curing may occur to produce CSH, in addition to carbonate, as indicated in eq 4.^{55,56}



2.3. CO₂ Uptake. The percentages of CO₂ stored in the cured composites were measured and are listed in Table 1. The

Table 1. CO₂ Uptake in the Optimized Polymer–Cement Composite

sample	blank	TPM-cement	TTPM-cement
CO ₂ uptake%	16.63	16.61	16.62

gas uptake (CO₂ uptake%) is calculated using eq 5 as the percentage of weight loss in the temperature range 500–900 °C. This percentage represents the amount of emitted CO₂ gas during ignition. (*W* CO₂) and (*W* dry) are the weight loss during ignition and the weight of original dry cured sample, respectively.^{57,58}

$$\text{CO}_2 \text{ uptake\%} = (W \text{ CO}_2 / W \text{ dry}) \times 100 \quad (5)$$

Table 1 indicates the CO₂ uptake% of the proposed optimized composite (7% polymer–cement) for treated and untreated composites. All composites and blank cement have almost the same CO₂ uptake%, as an average percentage of 16.6%. These values provide the evidence for the successful storage of CO₂ gas in the form of carbonate during the curing reaction, which is responsible for the improved mechanical and physical properties. It is clear that the filling of dicalcium silicate cement with the polymer waste mix did not affect the percentage of gas uptake. Similarly, the treatment reaction of the polymer filler has no effect on this percentage. It is a good behavior of the proposed polymer–cement composite to keep the percentage of gas stored and to improve the overall properties at the same time.

2.4. Mechanical Properties. The effect of the chemical treatment of polymer on the mechanical properties of cement is discussed here, along with the mechanical properties of different concentrations of cured composites. The polymer waste mix has a positive effect on the dicalcium silicate base. Figure 2 shows the values of the flexural strength and

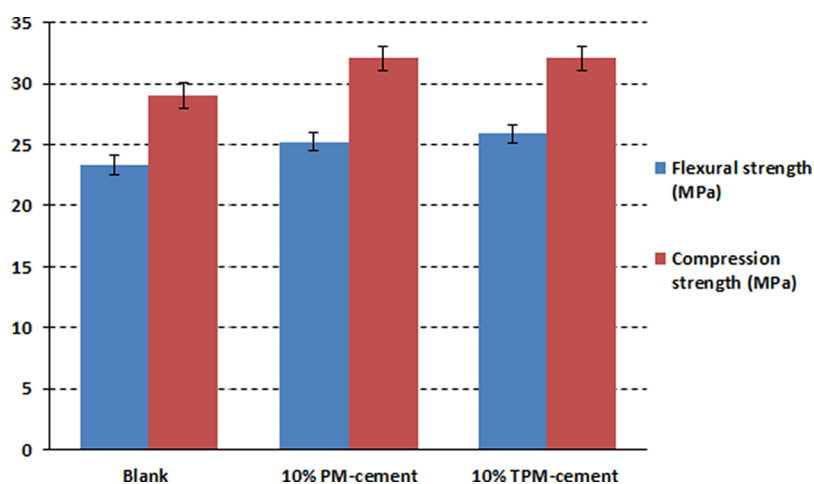


Figure 2. Effect of treatment on the mechanical properties of a 10% polymer–cement composite.

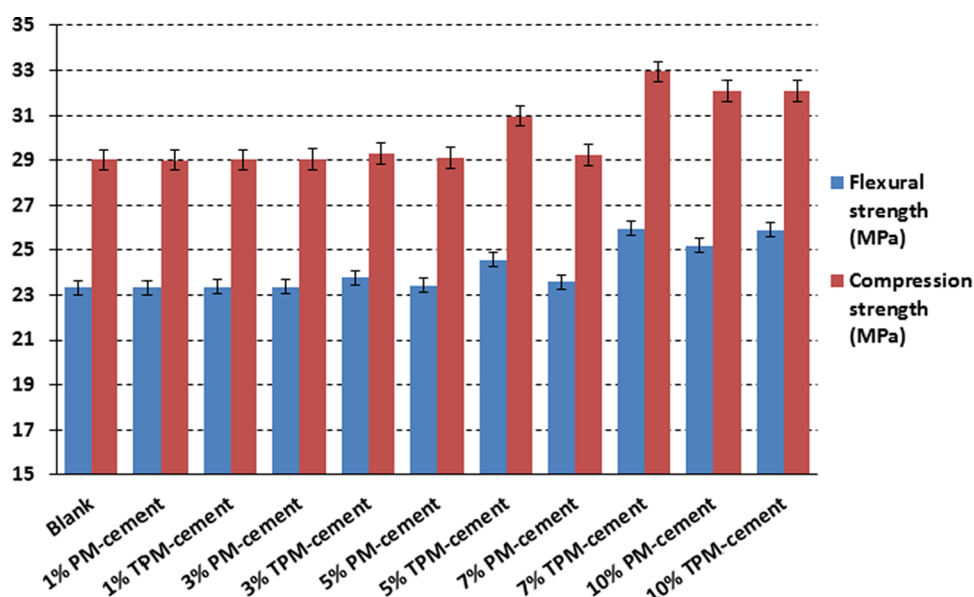


Figure 3. Mechanical properties of PM–cement and TPM–cement composites.

compression strength of PM–cement and TPM–cement composites with 10% concentration. First, this concentration is selected to investigate the effectiveness of the chemical treatment, performed to PM, on the composite properties compared with blank. It is found that 10% PM–cement and 10% TPM–cement composites have close mechanical properties (compared with other concentrations indicated later) due to the excess concentration of the plastic filler. The cement material cannot cover the filler in this case, leading to some decline owing to the overcontent of the filler and its agglomerations. This behavior is common regarding the formation of agglomerated structures of filling materials in different composites.^{59,60} Hence, the 10% composite shows limited enhancement in mechanical strength, even after the treatment reaction.



However, for other composites (with lower concentrations of the polymer), we can observe the difference between treated and untreated samples. Figure 3 indicates the mechanical properties of all concentrations based on untreated and treated polymer fillers. The most distinguished effect of treatment is noticed by the 7% concentration, where the flexural strength

and compression strength were increased by about 10.2 and 12.7%, respectively. However, other concentrations did not achieve this noticed enhancement. The flexural strength and compression strength have increased with more concentrations of the polymeric filler, starting from the 5% concentration. The noticeable enhanced mechanical performance in the proposed composites, especially the 7% concentration, is because of the treatment of the polymer mix, which improved the combination between the filler and the prepared dicalcium silicate. The given results match with that noticed previously regarding the enhancement of the composite properties after different modes of treatment.^{61,62} In fact, the treated polymer waste mix succeeded in toughening the hydrophilic cement material, resulting in distributing the applied force over the matrix. More mechanical stability is reached certainly. Overall, the 7% TPM–cement composite is the more mechanically stable concentration due to the maximum compression and flexural numbers, compared with blank cement and other composites.

For assuring these mechanical findings, the contact angle was measured for the treated and untreated fillers, as provided

in Table 2. The PM specimen shows a relatively high contact angle (at 109.5°) because of its hydrophobic nature. Basically,

Table 2. Contact Angles of PM and TPM

Sample	PM	TPM
Photo		
Contact angle (°)	109.5	81.6

the hydrophobic sample is characterized by the contact angle greater than 90°. However, after the treatment reaction, the contact angle of TPM decreased to 81.6°. The observed decrease is attributed to the hydrophilic property gained after the treatment of the plastic filler. We illustrate that the hydrophilic TPM filler could interact with the hydrophilic cement material. The resulting stronger filler–cement interface can improve the mechanical properties of the composite after the treatment reaction, as indicated previously.

2.5. Composite Surface. The pull-off test investigates the traction force required to axially pull a metallic disc that is adhered to the surface of the composite sample. This section evaluates the resistance and adhesion of the composites' surface by a group of tests including pull-off and impact. The pull-off test was conducted for studying the stability of the surface interface in different cementitious composites. In Table 3, it is noticed that the traction force has increased

Table 3. Pull-Off of the TPM–Cement Composites

sample type	pull off (MPa)	impact @ 10 Joule
control	2.23	pass
1% TPM–cement	2.23	pass
3% TPM–cement	2.25	pass
5% TPM–cement	2.28	pass
7% TPM–cement	2.28	pass
10% TPM–cement	2.26	pass

slightly with an increase in the concentration of TPM filler. The maximum pull-off strength (2.28 MPa) is observed for 7 and 5% TPM–cement composite, compared with the pull-off strength of control at 2.23 MPa. Generally, the pull-off strength of all polymer cement composites shows similar values, ranged from 2.2 to 2.3 MPa, which increased with the polymer waste additive. The impact test was performed to confirm the improved surface properties of the prepared composites. All the proposed TPM–cement composites passed the impact testing without failure at the same applied force. The combination between the polymer waste mix and silicate cement shows a strong surface adhesion on the proposed cement. This can be attributed to the relatively higher strength of the polymer mix and dicalcium silicate base, which led to a compatible interface, as observed in Section 2.4. The recorded values of the pull-off test and the resistance to rapid impact deformation confirm the high stiffness of the proposed dicalcium silicate cement filled with the treated polymer waste mix.

2.6. Water Absorption. The stability of the prepared composites against the absorption of water is shown in Figure

4. Eq 6 identifies the percentage of weight increase ($\Delta W\%$) caused by water absorption, where W_{dry} and W_{wet} represent the

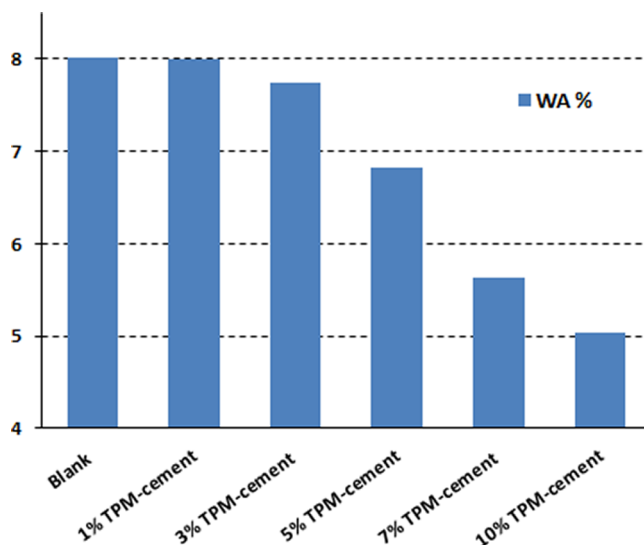


Figure 4. Water absorption of the TPM–cement composites.

weights of the composite before and after immersion in distilled water for 24 h.

$$\Delta W\% = \frac{W_{\text{wet}} - W_{\text{dry}}}{W_{\text{dry}}} \times 100\% \quad (6)$$

All TPM–cement composites have recorded lower percentages of water absorption due to the effect of the added plastic filler. The specimens of 1% TPM–cement composite and blank cement almost have the same percentage because of the low concentration of the filler. The obvious effect is started by 3% TPM–cement and 5% TPM–cement composites, which recorded 7.75 and 6.83%, respectively, compared with the blank (8.02%). The least percentage of water absorption is achieved by the 10% TPM–cement composite since it reduced by more than 37% in comparison with the blank cement. The 10% TPM–cement composite shows the more stable concentration against the diffusion of water. The effective filling of the plastic waste mix, after treatment, to the cement base has improved the composite stability. Overall, the different concentrations of the TPM–cement composite are more stable against water absorption compared with the blank cement material.

2.7. Carbon Footprint. This section discusses the carbon equivalent emissions of the proposed TPM–cement composites. Minimizing the carbon footprint is recommended for controlling global gas emissions and climate change. The value of CO₂ gas equivalent (CO₂-e) involves carbon footprint as a measurement for contributing to the greenhouse gas emission. CO₂-e is identified using eq 7, where “Q” and “F” represent, respectively, the related material amount and the emission factor for producing 1 m³ of the formed material.

$$\text{CO}_2\text{-e} = \sum (Q_x F_x + Q_y F_y + Q_z F_z) \quad (7)$$

Related literatures report this aim to get useful environmental friendly composites, based on low carbon materials, recycled and natural reactants, CO₂ storage, and low energy processing. The CO₂ equivalent emission is analyzed with regard to the parameters of materials collected, energy used,

transportation, casting, processing technique applied, gas emitted, and waste produced.⁷¹ The CO₂ equivalent emission of TPM–cement composites, corresponding to a dicalcium silicate cement base and TPM filler material, is given in Table 4. The CO₂-e of TPM–cement composites is set between

Table 4. CO₂-e Emissions (kg of CO₂-e/m³) of TPM–Cement Composites

sample type	dicalcium silicate base	TPM filler	total value
blank	301		301
1% TPM–cement	301	1.39	302.39
3% TPM–cement	301	1.39	302.39
5% TPM–cement	301	1.31	302.31
7% TPM–cement	301	1.22	302.22
10% TPM–cement	301	1.15	302.15

302.39 ppm of CO₂-e/m³ and 302.15 CO₂-e/m³. No obvious difference is present between these numbers and CO₂-e of the blank cement base (301 CO₂-e/m³), which too report the lower gas equivalent emission of all composite materials. CO₂-e of all proposed composites, and also of blank cement, is much lower than that of the commercial cementitious material which averaged as 354 CO₂-e/m³.^{72–74} The corresponding lower CO₂ gas emission of the proposed TPM–cement composites could share the global aim of reducing the carbon footprint and greenhouse gas emissions as improved environmentally friendly polymer cement composites.

3. MATERIALS AND METHODS

3.1. Materials. In this research, CaCO₃ and SiO₂ (Loba Chemie) are used for preparing the proposed cement material. The filler is a polymer waste mix (PM), from a local source, as a mixture of PET and PVC waste. Ethyl alcohol (Merck) was used for washing. Sodium hydroxide, potassium dichromate, and sulfuric acid (Merck) were used for preparing the treatment solutions for treating PM. The reactants were used without purification, except for a part of PM subjected to the treatment reaction.

3.2. Polymer Cement Composites. The cement base proposed in this research is dicalcium silicate (C₂S). CaCO₃ and SiO₂ reactants were dried first at 75 °C for 3 h, and then a mixture of molar ratio 2:1 was prepared by mixing and milling to obtain the powder with fineness of #200 mesh. The reactants' powder mix was heated finally to around 1390 °C to obtain the C₂S cement base. Afterward, the obtained C₂S starting material was filled with the PVC/PET polymer waste mix (PM). The mix of PM, in the form of scraps, was collected, grinded, washed with ethanol/water solution, and then dried for the removal of moisture. PM was subjected to a treatment reaction for a better filling with cement, as reported elsewhere,⁷⁵ using sodium hydroxide and chromic acid solutions to obtain the treated PM (TPM) filler. Finally, the blank C₂S and its composites based on 1, 3, 5, 7, and 10% TPM were cured using the carbonation process under the curing conditions of 65 °C, 0.3 MPa, and 60% RH for 24 h.

3.3. Characterizations. The percentage of CO₂ uptake in the proposed composite was identified as the percentage of weight loss in the 500–900 °C temperature range. Panalytical-X'pert-PRO wide-angle X-ray diffraction (XRD) was used within 5–80° 2θ, with Cu K-alpha source, 40 kV, and 1.54 Å wavelength. A Nicolet IS-10 FTIR spectrophotometer (Thermo Fisher Scientific) was used for the detection of

silicate. The thermal property of the cured cement was tested by a Mettler Toledo SF-TGA system: the specimens were heated in N₂ gas to 900 °C with 5 °C/min heating rate. The stability of composites against water absorption was studied after 24 h of immersion in distilled water. The carbon dioxide emission, as a measurement of the carbon footprint, was evaluated. The mechanical testing (flexural strength and compression strength) was conducted on the blank cement and different concentrations of composites following ASTM C78. Using the XLC H-universal testing machine, a loading rate of 20 KPa/s was applied to specimens with dimensions 162 × 42 × 42 mm³. The surface mechanical properties were evaluated as follows. The impact test was measured by a BYK H-duty tester according to ASTM C1870; the pull-off test was measured by a BGD tester according to ASTM D7234; 40 mm × 40 mm specimens were used for implementation with a rate of 5 mm/min.

4. CONCLUSIONS

- Exploitation of polyvinyl chloride/polyethylene terephthalate waste mix for filling low-energy calcium silicate cement was done to prepare environment-friendly polymer–cement composites, for reducing climate change.
- The microstructural analyses affirm the formation of dicalcium silicate, and its curing yields carbonate and some calcium silicate hydrate. The curing technique succeeded in CO₂ uptake by 16.6%.
- Composites were cured by the carbonation technique, leading to the conversion of CO₂ gas to the main carbonate product which is responsible for the improved behavior.
- The recycled waste mix filler has promoted the physical and mechanical properties of dicalcium silicate cement and has maintained the gas uptake.
- Mechanically, the flexural and compression strengths were increased after treatment due to the strengthening of the polymer waste mix–cement interface. Both values increased gradually with more contents of treated polymer waste. 7% concentration achieved improved properties.
- The maintained values of the pull-off test and the resistance to rapid impact deformation approve the surface stability of the proposed composites.
- Compared with blank, all composites absorbed lower water percentages due to the effect of the plastic filler. The 10% composite is the more stable concentration against the diffusion of water.
- The proposed composites are environmentally recommended for construction applications due to the sharper decreasing carbon footprints for all composites than that of commercial cement. Formation of low-energy cement and reuse of polymer waste are other sustainable impacts.

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Author Contributions

The manuscript was written through the contributions of all authors. All authors have approved the final version of the manuscript.

Notes

The authors declare no competing financial interest.

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