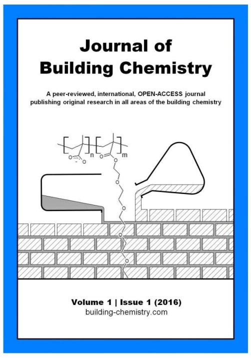

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Application of fibres from recycled PET bottles for concrete reinforcement

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Abstract

Waste PET can be reused as partial or complete substitute of an aggregate in a concrete composition or as a concrete reinforcement. However, the main drawback of such applications is the hydrolysis of ester linkages of poly(ethylene terephthalate) in highly alkaline environment of the cement matrix. To prevent alkaline hydrolysis, the PET fibres were coated with commercially available ethylene/vinyl acetate copolymer (EVA). Effectiveness of the use of copolymer EVA as a protection layer against strong alkali solutions has been demonstrated and discussed. Chemical changes in PET fibres after alkaline treatment have been referred to mechanical properties of the fibres. Mechanical properties, like compressive and flexural strength of the composites as well as the long-term durability performance of recycled PET fibres in alkaline environment were also investigated. The preliminary results indicated that the introduction of the PET fibres does not deteriorate the mechanical strength of the concrete composite.

Keywords: PET bottles; recycling; fibres; reinforced concrete; hydrolysis

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1. Introduction

Concrete is the most important and commonly used building material. The reasons for that are, among others, the availability of the raw materials, high compressive strength and relatively low cost of production. Concrete has also some disadvantages, namely brittleness, low tensile strength (as compared to the compressive strength) and low crack-propagation resistance. Modification of concrete with polymers creates substantial possibilities of designing the concrete properties, including improvement of the above mentioned weaknesses (Banthia et al. 2014). However, the polymer additives are relatively expensive. The possibility of using polymer wastes for modification of cement concretes and mortars is, therefore, particularly important. The amount of such wastes is continuously increasing, bringing serious ecological and economic problems, since the extent of biodegradation of commodity plastics is very low.

Poly(ethylene terephthalate) (PET), is an example of a plastic stored in rubbish dumps. It is used, among others, for the production of packages such as bottles. PET has high mechanical strength, impact resistance,

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rigidity and good resistance to environmental factors (Lepoittevin and Roger 2011). Because of extremely short time of use of PET bottles, they become wastes very quickly. The plastic wastes can be burnt with retrieving of energy, but such polymers seem to be too valuable raw materials for utilizing them in this way. The building materials industry creates a possibility of more rational reuse of PET wastes (Wiliński et al. 2013).

The studies on the possibilities of using PET bottles in the building materials industry have been carried out for more than 20 years. The PET waste can be used as the raw material for production of unsaturated polyester resins, which in turn can be applied for manufacturing the polymer concrete (Rebeiz et al. 1991; Rebeiz 1995; Rebeiz and Craft 1995; Jo et al. 2008; Tonet and Proszek Gorninski 2013). Flakes obtained from PET bottles can be applied as a component of the cement concretes and mortars (Choi et al. 2005; Marzouk et al. 2007; Albano et al. 2009; Akçaözoğlu et al. 2010; Frigione 2010) or asphalt concretes (Hassani et al. 2005). PET wastes can be also utilized in the synthesis of polyurethane materials (Mazurek et al. 2014) which are used, for example, in system of steel-elastomer sandwich plates for strengthening bridge decks (Feldmann et al. 2007).

Rebeiz (1995) produced and tested the polymer concrete based on recycled PET. For minimizing the cost, they used crude material (separation of PET from pigments, paper and aluminium is the most expensive phase of PET packages recycling). The obtained polymer concrete exhibited after one day more than 80% of the final compressive strength, while the ordinary cement concrete reaches after one day about 20% of the final strength. The authors indicated that compressive strength of the polymer concrete decreases with growing temperature; e.g. the increase of temperature from 25 to 60°C causes lowering of the compressive strength by about 40%.

Choi et al. (2005) obtained the lightweight aggregate by mixing the waste PET flakes with granulated blast-furnace slag carried out at melting point of PET (250°C). The density of such an aggregate is equal to about 50% of that of a typical aggregate. The substitution of 75% of the sand by the lightweight aggregate causes lowering of the concrete mix density by above 15% (for the concrete with water/cement ration, w/c = 0.45). However, the compressive strength of the concrete decreased from 37.2 MPa to 24.9 MPa (after 28 days) as well as the tensile strength decreased from 3.32 MPa to 2.04 MPa.

Another method of reusing of the PET bottles waste can be the production of fibres for concrete reinforcement. The dispersed reinforcement such as fibres significantly reduces the volume changes in the concrete, thus preventing the shrinkage cracks propagation (Banthia and Nandakumar 2001). However, polymer fibres including PET fibres exhibit poor wettability and adhesion to cement paste (Machovič et al. 2013). Del Rey et al. (2011) used the recycled PET fibres in the building acoustics field as sound absorbing materials.

The action of fire on the concrete leads to the reduction of the strength and material degradation, and in the case of high-strength concrete even explosive spalling of the concrete pieces. The presence of polymer fibres improves the strength of the concrete elements during fire: after polymer burning, the voids – air channels are created, what enables water evaporation and consequently prevents the increase of the internal pressure, which can destroy the concrete. The low resistance to elevated temperature can be, therefore, turned to the advantage of the concrete reinforcement with polymer fibres (Hager and Tracz 2009).

The PET fibres can be obtained by cutting yarn or sheet. The sheet is cut to stripes, which are then treated in such a way that the surface of the fibres – obtained by the cutting of the stripes – was not smooth. The yarn is also treated this way before cutting into the fibres. After this process the fibres exhibit improved adhesion to the cement matrix. The fibrillated fibres are more effective in controlling shrinkage cracking than their comparable monofilament counterparts (Banthia and Gupta 2006).

Ochi et al. (2007) have found that PET fibres, cut from the yarn with 700 μm diameter and 30 mm length, positively affected the strength of the resultant concrete. Addition of 1.5% (by volume) of the fibres causes an increase of the flexural strength of the concrete by about 30% and increase of the compressive strength by about 10%. Additionally, the fibre reinforced concrete can withstand the loads from cracking.

The fibres with the diameter significantly smaller than 700 μm do not influence the mechanical properties of the concrete in such a favourable way. Silva et al. (2005) used the PET fibres obtained from the yarn. They revealed that the addition of 0.4% (by volume) of the fibres with 26 μm diameter and 20 mm length did not cause changes in the compressive strength, tensile strength, flexural strength and Young modulus even after long time of concrete curing.

The influence of the PET fibres, cut from the sheet, on the reduction of the shrinkage cracks has been studied by Kim et al. (2008). They used fibres with 50 mm length, 0.5 mm thickness and 1 mm width. Substantial decrease of the cracks area (by 40-70%) was observed for the amount of fibres as small as 0.1% by volume. Higher amount of fibres (0.5 vol.% and more) led to reduction of the shrinkage cracking by about 90%.

Won et al. (2010) demonstrated that the concrete composite containing PET fibres exhibits better frost resistance than ordinary concrete (after 300 freezing and thawing cycles).

Foti (2011) analyzed the stress-strain curves for the concrete reinforced with the fibres obtained by simple cutting of the PET bottles. The investigation showed that addition of even a small amount of the fibres can have a large influence on the post-cracking behaviour of concrete elements.

Won et al. (2010) treated the cement composite samples, containing 1% (by volume) of the recycled PET fibres, with an alkaline solution (pH = 12.6). The authors demonstrated that the compressive strength of the specimens decreased with time, which was attributed to the hydrolysis of poly(ethylene terephthalate).

The initial pH of the young concrete is equal to 12.5-13 (Räsänen and Penttala 2004). This strongly alkaline medium is originated from calcium hydroxide in the porous liquid inside the concrete. Polyesters, i.e. polymers containing ester groups, like PET, are susceptible to the hydrolysis under alkaline conditions of the concrete (Fig. 1). Due to the degradation of PET fibres in the cement matrix, the mechanical properties of the fibres decreased with time (Silva et al. 2005; Pelisser et al. 2012). Thus, polyesters are not recommended for concrete modification.

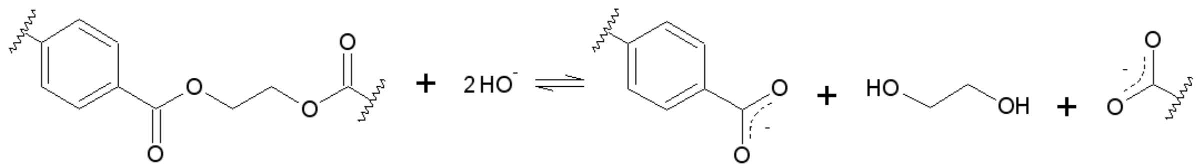

Fig. 1. Scheme of PET hydrolysis under alkaline conditions.

We propose coating of the PET fibres with a protective layer as a method of preventing their degradation in concrete. Additionally, the coating should improve adhesion to the fibres surface. The copolymer EVA has been proposed as the material for making the protective coating on the PET fibres. This copolymer has polar groups in its structure, which improve the adhesion to the fibre surface. PET has similar polar ester groups like EVA. Pending ester groups of EVA hydrolyze in the highly alkaline environment (Fig. 2). However, in the case of the protective coating it is a favourable phenomenon, because buffering zone is created to separate the fibres from the alkaline environment (Fig. 3). What is more important, the main chain of the copolymer – responsible for the fibre strength – is not degraded. The final product of EVA hydrolysis is the copolymer of ethylene and vinyl alcohol, which additionally improves adhesion of cement matrix to fibres (due to the presence of the hydroxyl groups). The other product of hydrolysis – acetate anions forming crystals of calcium acetate are the other product of hydrolysis.

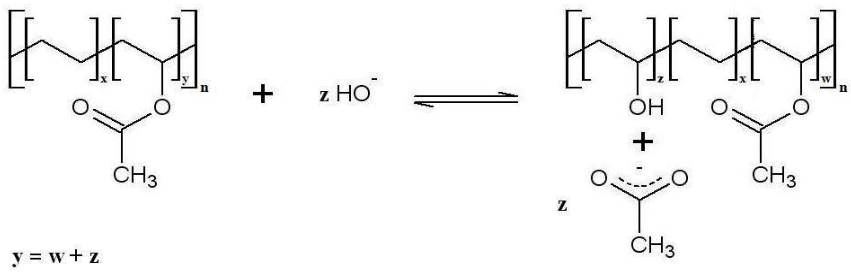

Fig. 2. Scheme of hydrolysis of EVA copolymer in alkaline environment; the terpolymer of vinyl acetate, ethylene and vinyl alcohol is formed together with acetate anion.

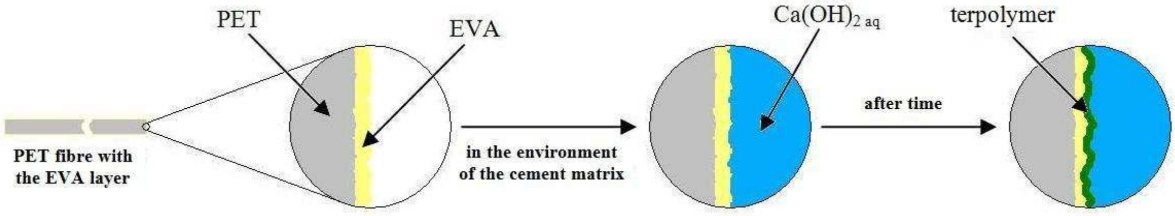

Fig. 3. Concept of EVA action as the layer which protects the PET fibres against the alkaline environment of the cement matrix. The product of the total hydrolysis of EVA is terpolymer of ethylene/vinyl acetate/vinyl alcohol.

The evaluation of protective efficiency of EVA coating on the PET fibres was carried out within the research presented in this paper. The influence of the PET fibres addition on the consistence of the concrete mix as well as the compressive strength and flexural strength of the concrete (after 28 days of curing) was studied. The adhesion of polymer-concrete was tested by measuring the force required for pulling out the polyester fibre from the cured concrete. A method of modification of the protective layer towards improvement of friction coefficient between fibres and cement matrix was proposed; the silica powder was proposed as a modifier.

2. Experimental

2.1. Materials

Mix proportions of the concrete and mortar are given in Tables 1 and 2. The ordinary Portland cement and high-range water-reducing admixture were used. The poly(ethylene terephthalate) fibres, as shown in Fig. 4, were formed by mechanical cutting of PET bottles. The bottlenecks and the bottom of the bottles were discarded. The uniformity of fibres is ensured, especially for the length and width, by fine adjustment performed in a semi-automatic cutting machine. The fibre volume fraction was categorized into three levels: 0% (reference), 0.1% and 0.3%. The main characteristics of the recycled bottle PET fibres are presented in Table 3.

Table 1. Concrete mix ingredients and their proportions.

Material	Unit weight (kg/m^3)	Weight proportion
Cement	360	1
Fine aggregate	595	1.65
Coarse aggregate	1264	3.51
Water	162	0.45
Superplasticizer	1.55	0.0043

Table 2. Mortar mix ingredients and their proportions.

Material	Weight proportion
Cement	1
Fine aggregate	3
Water	0.5

Table 3. Properties of the recycled-bottle-PET fibres.

Material	Length (mm)	Width (mm)	Thickness (mm)	Density ($g \cdot cm^3$)
PET fibre	50-70	2-3	0.20-0.25	1.38

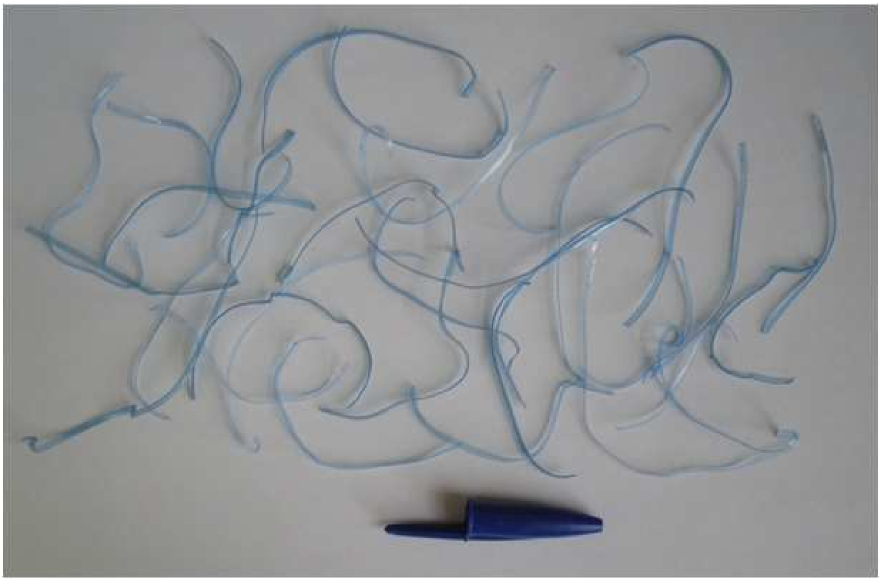

Fig. 4. Fibres obtained from waste PET bottles.

2.2. Test methods

2.2.1. Conditions of PET hydrolysis

In order to investigate the durability of the PET fibres in the alkaline environment, the samples were cut from bottles and immersed into 0.316 M NaOH solution (pH = 13.5). The immersion time was 30 days, and the temperature during the immersion was kept at 37 ± 2°C. The chemical resistance of PET against hydrolysis was improved by using dip coating method in a solution of EVA copolymer.

2.2.2. Measurement of PET specimens tensile strength

The influence of the alkaline treatment on the tensile strength of PET (PET/EVA) was investigated. The specimens were prepared and tested according to EN ISO 527 standard. The specimens in the form of dumbbells were cut from the waste PET-bottles.

The dimensions of the specimens were as follows:

- total length: 63 mm,
- neck length: 28 mm,
- neck width: 6.1 mm,
- thickness: 0.25 mm.

Mechanical properties of PET specimens were determined using testing machine Instron 5566. Head speed – 30 mm/min.

2.2.3. Measurement of the concrete mix consistence

The consistence of the concrete mix was measured using slump method. In this method the cone (with standardized dimensions) of the concrete mix is formed and compacted by taping (EN 12350-2). The concrete consistence is the value of a cone slump after raising of the cone mould.

2.2.4. Measurement of concrete compressive strength

Compressive strength is defined as the maximum stress which can be withstood by the material under compressive load. The compressive strength of the concrete was measured using cubic specimens 150 x 150 x 150 mm, according to EN 12390-3 standard.

The value of the compressive strength was calculated from the formula $f_c = F_n/A$ [Pa], where F_n is the compressive (destructive) load [N] and A is the area of the cross-section of the compressed specimen, perpendicular to the loading direction [m^2].

2.2.5. Measurement of concrete flexural strength

Flexural strength is defined as maximum stress which can be withstood by the material under bending load. The flexural strength of the concrete was tested by one-point centric loading of the prismatic specimens 100 x 100 x 500 mm, according to EN 12390-5 standard.

The value of the flexural strength was calculated from the formula $f_b = M/W$ [Pa], where M is the bending moment [N·m] and W is the strength index [m^3]. The bending moment for the beam, on which the destructive load is acting in the centre of the distance between two supports is equal to $M = Fl/4$ [N·m], where F is the destructive load [N] and l is the distance between two supports – the span of the specimen between the supports [m]. The strength index W was calculated from the formula $W = bh^2/6$, where b is width of the beam [m] and h is height of the beam [m].

2.2.6. Measurement of adhesion between PET stripes and mortar

The following test was performed for evaluation of the influence of the protective layer modifiers on the mechanical anchoring, adhesion and friction of the PET fibres in the cement matrix. The cement mortar mix was cast into the mould 40 x 40 x 160 mm, up to the 15 mm height, and then the PET stripe (80 mm length, 15 mm width) was introduced into the mortar. The mortar was cured in water for 14 days and then the samples of 40 x 20 x 15 mm (Figs. 5 and 6) were cut using an angle grinder. The relation tensile stress – deflection was determined using testing machine. Head speed was 2 mm/min.

Fig. 5. Specimen for testing adhesion of the PET stripe to the cement matrix.

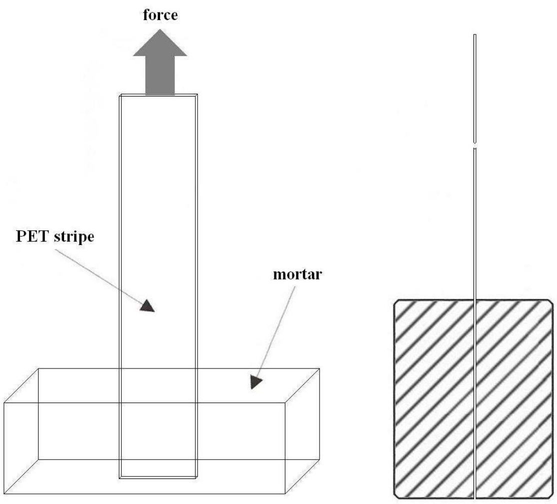

Fig. 6. The concept of testing adhesion of PET to the cement matrix.

3. Results and discussion

3.1. Concrete slump

The dependence of concrete slump on fibre concentration in the concrete mix is given in Table 4. It is clear that the slump of the concrete mix significantly decreases with growing content of PET fibres (Fig. 7). These results indicate that the workability of the mix is worsened by addition of PET fibres. The fibres content above 0.3% by volume causes serious problems concerning homogeneity and workability of the concrete mix.

Table 4. PET fibres volume content and slump of the concrete mixes.

Fibres content, (volume %)	Concrete slump, (mm)
0	100
0.1	50
0.3	10

Fig. 7. Slump test for the concrete mix without PET fibres (left) and with 0.1% (by volume) PET fibre (right).

3.2. Mechanical properties of the modified concrete

The influence of fibres addition on the mechanical properties of concrete was investigated. The compressive and flexural strength of the concrete containing the PET fibres and without the fibres were measured after 28 days of curing (Table 5).

Table 5. The strength of concrete with and without PET fibres.

PET fibres content, volume (%)	Compressive strength (MPa)	Flexural strength (MPa)
0	34.6 ± 0.4	3.7 ± 0.5
0.1	35.2 ± 0.3	3.6 ± 0.4
0.3	33.8 ± 0.5	3.6 ± 0.4

Addition of 0.1% (by volume) of the PET fibres slightly improves the compressive strength of the concrete as compared to the concrete without the fibres. For the concrete containing 0.3% (by volume) of the PET fibres, a slight decrease of the compressive strength was observed.

The flexural strength of the concrete containing 0.1% (by volume) of the PET fibres is slightly lower as compared to the concrete without the fibres and similar to that of the concrete with 0.3% (by volume) of the PET fibres (the difference is within the measuring error).

3.3. Evaluation of efficiency of EVA as a protective layer for PET fibres

The PET specimens, cut from the waste bottles, were coated with the EVA layer with the thickness of about 2 µm, and then immersed in the solution simulating the alkaline environment of the cement matrix. After 30 days of immersion the tensile strength of the dumbbell specimens was determined (Table 6).

Table 6. Influence of protective coating on tensile strength of PET specimens after 30 days of alkaline treatment.

PET specimens	Tensile strength (MPa)	Decrease of tensile strength referring to not treated specimens (%)
not treated	105.8 ± 12.8	–
after alkaline treatment, without protective coating	89.4 ± 7.5	15.0
after alkaline treatment, with EVA protective coating	103.7 ± 12.5	1.4

The tests showed a more than tenfold smaller change of the tensile strength for the protected PET fibres, as compared to unprotected ones.

3.4. Measurement mechanical anchoring of the PET stripes

The addition of the silica powder to EVA copolymer (protective layer for PET fibres) with mass ratio 1:1 was for increasing the mechanical anchoring of the PET fibres in concrete.

The stress necessary to start the pulling out of the fibre from the mortar was measured, i.e. the force (referring to the original area of contact) above which the fibre begins easily to go out from the mortar specimen (Table 7).

Table 7. Stresses necessary for pulling out the PET fibres from the cement matrix after 14 days of the mortar curing.

PET stripe	Stress at pulling out (MPa)
no coating	33.7 ± 3,6
EVA coating	78.7 ± 3,7
EVA coating modified with silica powder	89.9 ± 5,9

Due to the hydrolysis reaction, the unprotected PET stripes lose their mechanical properties with time. For this reason the tests were made prior to 14 days; after longer period the stripes would most likely break before the total pulling out.

As it is clearly seen, the modification of EVA copolymer with silica powder improves significantly the adhesion of the stripes to the cement matrix – the stress necessary for pulling them out increases by almost 15%.

4. Conclusions

The PET fibres prepared by cutting of waste PET bottles can be applied for concrete modification. To protect the fibres against hydrolysis during long-time exposition to the alkaline environment of the cement matrix, EVA copolymer was used as protecting coating. The efficiency of such coating has been demonstrated and discussed.

Taking into account that the efficiency of dispersed concrete reinforcement depends on the adhesion of the fibres to the cement matrix (Santos et al. 2015), silica powder was added to the EVA coating. The presence of silica particles improved mechanical anchorage by increase of friction between coating layer and concrete matrix.

It was demonstrated that the introduction of the PET fibres does not deteriorate mechanical strength of the concrete composite. However, the presence of polymer fibres caused worsening in workability of the concrete mix. Therefore, the addition of PET fibres above 0.3 vol.% is not recommended.

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