



Utilising poly(ethylene terephthalate) wastes in concrete reinforcement: A sustainable approach to waste management, building materials and conservation

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Abstract

Carbon neutrality and carbon footprints' reduction are global development goals that are intrinsically linked to waste management and are both achievable by wastes' valorisations. Herein, 0.5%–10% graded poly(ethylene terephthalate) (PET) wastes were incorporated in M25 concrete as a sustainable and environmentally benign method of managing and/or reducing the nuisance of plastic wastes. The concrete was prepared by the weight-batching method in a 1:1:2 ratio of cement, sand and gravel, respectively; with a proportionate replacement of these constituents by PET to yield PET–concrete composites, after water-curing for 7–35 days. The addition of PET reduced the workability of the concrete by 33%–83% but furnished composites with improved compressive (0.66–2.89 N/mm²; 2.6%–10.6%) and tensile (0.04–0.64 N/mm²; 3.1%–79%) strengths, respectively; peaking with 2% PET content. Durability tests revealed favourable properties of reduced water sorptivity (13.2% → 8.4%) and improved resistance to acid attack (7.4% → 4.9%) but added susceptibility to seawater penetrability (1.5% → 3.1%), as PET contents increased, compared to the control composite (13.4%; 9.5%; 1.3%), respectively. A preliminary life cycle assessment was positive and in concurrence with the notion that incorporating PET into concrete is a feasible and green way of mitigating plastic wastes.

Keywords: Carbon Neutrality; Circular Economy; Concrete; Life Cycle Assessment; PET Bottles; Sustainability; Waste Recycling

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1. Introduction

The trends, future and sustainability of polymeric materials continue to elicit interests and concerns in both academia and industry as well as in the sphere of public opinion. These are, in part, due to the enormous utilities of polymers; often colloquially referred to as “plastics,” and their short/long-term harmful effects on the environment and ecosystems (Adegbola et al., 2021; Askar et al., 2023; Belgacem and Gandini, 2008; Papageorgiou, 2018). It is no gainsaying that polymeric materials are all around us: from clothing; made from synthetic fibres, to home utensils, and from polyethylene and Teflon-coated cookware to Kevlar; made from aromatic polyamides (aramids) (Namazi, 2017; Vilela et al., 2014). They also find uses in healthcare, packaging and carrier bags (Namazi, 2017; Ong et al., 2024). Likewise, fiberglass, nylon bearings and polymer-based paints are used in automobiles whereas epoxy glues and polyurethane foams are used in furnishings (Vilela et al., 2014). Not forgetting silicone heart valves and catheters, employed in medicine (Ong et al., 2024). Additionally, polymer applications are widespread in engineering, pharmaceuticals, biomedicine and agriculture, to name a few (Nakajima et al., 2017; Oladele et al., 2020; Sikder et al., 2021).

Plastics are the general term for a wide range of synthetic and semi-synthetic polymeric materials, which also include poly(ethylene terephthalate) (PET). PET is a high-performance engineering plastic with suitable physical properties applicable to the manufacture of bottles, fibres, films and other engineering applications (Mestry et al., 2024; Nakajima et al., 2017; Singh et al., 2021). It is equally a major material for making blown bottles that are used in the packaging of soft drinks, foods and other consumer goods (Akinyele et al., 2020; Malik et al., 2017). It is instructive that the origin of plastics dates back to 1847 when Shönbein produced celluloid; the first thermoplastic resin, by reacting a natural polymer, cellulose, with nitric acid (Aguado and Serrano, 1999). The commercialisation of plastics, however, began in the 1930s; during World War II, when natural polymers, such as natural rubber, were in short supply. The quantity of plastics used annually has since grown steadily; with over 400 million metric tonnes manufactured globally (Babaremu et al. 2022). The lightweight, strength, user-friendly fabrication capabilities, long shelf lives and low cost are some of the factors responsible for the phenomenal progress of plastics (Singh and Sharma, 2016). They have been used in packaging, textiles, automotive and industrial applications, water desalination, land/soil conservation, flood prevention, and the preservation and distribution of foods and housing, etc. (Aguado and Serrano, 1999; Sikder et al., 2021; Singh and Sharma, 2016).

Accordingly, with such large and varying applications, plastics also contribute to an ever-increasing volume in the solid waste stream (Siddique et al., 2008). The annual consumption of PET bottles, for example, is reported to exceed 300,000 million units, with a projected 15% annual increment (Pacheco-Torgal et al., 2012) while PET wastes account for 8% and 12% by weight and volume, respectively, of global solid wastes (Raheem et al., 2019). Although waste can be generally defined as any unavoidable material resulting from domestic or industrial activities for which there is no economic demand (Aguado and Serrano, 1999; Sridhar and Hamed, 2014), circular economics considers waste as a material waiting to be recycled or re-used (Babaremu et al. 2022; Ong et al., 2024). Aside their uses as concrete admixtures and in construction, therefore, PET waste bottles also find utilities in agriculture and horticulture as well as 3-D printing (Babaremu et al. 2022). Nevertheless, solid waste management has become an issue of increasing global concern as urban populations continue to rise and consumption patterns change rapidly (de Mello et al., 2009; Sridhar and Hamed, 2014).

Typically, PET can be recycled by mechanical, thermal and chemical methods (George and Kuria, 2014; Jai et al., 2023; Kazemi and Fini, 2022). However, the sustainable production of biologically based PET (bio-PET)

is similarly gaining traction as an *ab initio* recycling method (Nakajima et al., 2017). Bio-based polymers are polymeric materials synthesised from renewable resources, such as biomasses and other natural feedstocks (Kimura, 2009). In the case of bio-PET, its bio-based ethylene glycol (bio-EG) monomer has been produced whereas the preparation of terephthalic acid (TPA) from non-petroleum-derived sources is in development (Nakajima et al., 2017). The polymerisation of bio-EG with bio-TPA is, therefore, expected to afford a biodegradable PET (bio-PET) (Kimura, 2009; Nakajima et al., 2017). Biological (enzymatic) recycling is also being investigated (Son et al., 2019); with the recent isolation of the bacterium, *Ideonella sakaiensis*, which uses PET as a major energy and carbon source, and can degrade it into mono(2-hydroxyethyl) terephthalic acid (MHET) monomers, using the enzymes: PETase and MHETase (Yoshida et al., 2016). It is significant to note that naturally-occurring thermophilic bacteria for PET hydrolysis have also been reported (Valdez-Nuñez and Rivera-Jacinto, 2024).

The mechanical recycling of PET involves a number of treatments and operations, such as sorting; from other plastics, washing; to remove dirt and other contaminants, and grinding and crushing; to reduce particle size, as well as extrusion by heat, pelletising and reprocessing into other PET products (Aguado and Serrano, 1999; Barjoveanu et al., 2023). The chemical method, on the other hand, depolymerises PET into its monomers, such as terephthalic acid (TPA), bis(hydroxyl ethylene) terephthalate (BHET), dimethyl terephthalate (DMT) and ethylene glycol (EG) (George and Kuria, 2014; Zhang et al., 2022). It can also be partially depolymerised to its oligomers or other constituents, using any of the following depolymerisation routes: methanolysis, glycolysis, hydrolysis, ammonolysis, aminolysis and hydrogenation (Mârşolea et al., 2023). Lastly, the thermal method of recycling PET involves the use of heat at high temperatures under inert atmospheres, at great costs to energy and the environment (Cunliffe et al., 2003; Fotopoulou and Karapanagioti, 2017).

It is noteworthy that the chemical method of recycling is also expensive due to the high cost of raw materials, scale of operation and the capital investments required. For instance, it has been calculated that for a PET chemolysis facility to be economically viable, a minimum throughput of about 1.5×10^4 tonnes per annum is required (George and Kuria, 2014). This may explain why chemically recycled PET; typically numbered “1” as its recycling symbol, is more expensive than virgin PET. On the contrary, the mechanical recycling of PET is relatively simple and requires lower investments. It is also of minimal impact on the environment, flexible in terms of feedstock volume and utilises more commonly available equipment. Nonetheless, mechanically recycled PET is prone to contamination by other adjuvants or copolymers, such as polyvinyl acetate, ethylene vinyl acetate and polyvinyl chloride; leading to discolouration and rapid degradation as well as diminished mechanical properties and relatively low and heterogeneous intrinsic viscosity values, amongst others (Aguado and Serrano, 1999).

Furthermore, the collection, sorting and separation costs can be exorbitant because of the low-bulk density of PET bottles. Besides, trace metals (e.g., antimony, cobalt and manganese) from catalysts' residues and additives; used for PET production and present in post-consumer PET wastes, can promote transesterification and polycondensation reactions in a mechanically recycled PET. The latter can make the recycled material chemically heterogeneous and, therefore, affect melt rheology behaviour batchwise. Consequently, mechanically recycled PET seldom find use in high-grade applications, such as the production of household bottles, films and industrial fibres (Scheirs, 2001; Yamaye et al., 2002) but can be converted to high molecular weight polymers of uniform and consistent intrinsic viscosities using solid-state polymerisation (SSP), which considerably reduces undesirable side reactions and by-products (Robinson and Vadnais, 2007).

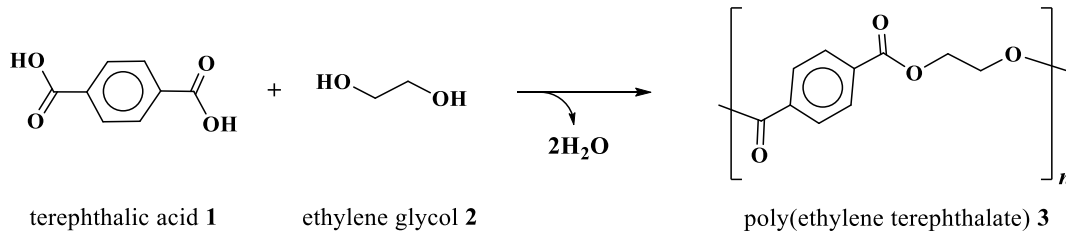


Figure 1. Synthesis of poly(ethylene terephthalate) – PET.

PET is a transparent, thermoplastic polyester with good chemical resistance; except to alkalis, and dimensional stability, under variable loads, as well as good gas barrier and mechanical properties. It is used in the manufacture of bottles, thermally stabilised films (e.g., in capacitors and recording tapes) and production of fibres in the textile industry (Sulyman et al., 2016). PET is formed via the condensation reaction of terephthalic acid (TPA) and ethylene glycol (EG) or by a transesterification reaction between dimethyl terephthalate (DMT) and ethylene glycol (EG). Its crystallinity varies from amorphous to fairly high crystalline and it can be colourless but thicker sections are usually opaque and/or off-white (Ji, 2013). It is produced by the polymerisation of EG and TPA, as shown in Figure 1. EG is a colourless liquid obtained from ethylene whereas TPA is a crystalline solid obtained from xylene. When heated together, under the influence of chemical catalysts, EG and TPA produce PET in the form of a molten, viscous mass that can be spun directly into fibres or solidified for later processing as a plastic (Ji, 2013). Water is also produced as a by-product.

Disparately, concrete is a composite of cement, aggregates (sand, gravel or concrete rubbles), water and additives, commonly used as construction and building materials (Encyclopaedia Britannica, 2024); with an estimated annual global usage of about 11 billion metric tonnes (Umasabor and Daniel 2020). It possesses good compressive but low tensile strengths (Foti, 2013; Oladele et al., 2020). The evolution of carbon dioxide (CO_2) in its production and colossal carbon footprint (Editorial, 2021) as well as rapid surface deterioration, when exposed to harsh alkalinity or acidity, are also causes of concern (Sarde and Patil, 2019). The low tensile strength in concrete has been partly attributed to the presence of cracks caused by concrete shrinkages but, in recent decades, the incorporation of fibres has mitigated the tensile stresses in concrete by the “sewing effect” of the fibres on the cracks (Borg et al., 2016). To improve the features of concrete, therefore, the reinforcing fibre must be easily dispersible within the mixture, have suitable mechanical properties and be durable in the predominantly alkaline cement matrix (Silva et al., 2005). Remarkably, the fibres tend to be more effective in the post-cracking phase wherein they reduce and/or prevent the propagation of the cracks (Foti, 2013).

The use of polymers (Awoyera and Adesina, 2020; Baričević et al., 2018; Oladele et al., 2020), particularly PET bottles (Marzouk et al., 2007; Saikia and de Brito, 2013; Shahidan et al., 2018), as additives, resins and fibres for polymer-concretes (Abdel-Azim, 1996; Gao et al., 2019; Mendivil-Escalante et al., 2017) and concrete reinforcement, respectively, have been reported (Jensen, 2013; Kazemi et al., 2021; Pacheco-Torgal et al., 2012; Sharifianjazi et al., 2022). The PET bottles have also been differently pulverised (Frigione, 2010; Rahmani et al., 2013) and shredded (Osubor et al., 2019; Saikia and de Brito, 2014) as well as cut into various shapes (Dinesh and Rao, 2017; Foti, 2013; Mazenan et al., 2018) prior to their use as concrete reinforcements (Suraweera and De Silva, 2023). Consequently, some of the results have shown that the quantity of PET wastes recyclable was minimal; with fibre-to-concrete ratio ranging between 0.3%–1.5%, in contrast to using PET particles as substitutes for aggregates and mortar (Reis et al., 2011; Tani et al., 2018); which not only lessens

the strain on natural resources and saves energy but equally mitigates wastes and environmental pollution (Rahmani et al., 2013). The inclusion of waste PET in concrete can also decrease the brittleness of concrete without affecting its electrical resistance (Sadrmomtazi et al., 2016). It is interesting to note that differences in the size, shape and texture of PET aggregates similarly affect water-to-cement ratios as well as the slump of fresh concrete mixes, which ultimately change mechanical behaviour (Albano et al., 2009; Saikia and de Brito, 2014). Accordingly, fibre-reinforced materials have been found to be more ductile than unreinforced specimens (Yi et al., 2015).

This study investigates the application of grated, non-virgin PET (water) bottles as fine aggregate reinforcements for concrete, with a view to managing and/or reducing the environmental nuisance of plastic wastes whilst developing lightweight PET-concrete composites as multi-purpose, robust building materials as well as conserving some of the natural resources, such as sand and gravel, employed in the construction industry. It is notable that previous studies have investigated the use of PET wastes as partial replacements for the fine or coarse aggregate constituents of concrete but none of the reports have looked into simultaneously and proportionately reducing the cement, fine and coarse aggregates' contents of concrete by substituting the components with PET wastes.

2. Materials and method

2.1. Collection of materials

The University of Lagos (Unilag) water bottles were collected from open sources, within the Akoka campus, sorted, rinsed with pipe-borne water and air-dried. Whence the bottles were shredded with a mechanical cutter (Shanghai Junsal 39851) and micronized using a grater (GoldModil Ms-1.5hp). The resulting particles were passed through a No. 4 sieve (4.75 mm), according to ASTM D6913-04 standards (2004), and stored in a dry polythene bag at ambient temperature till use. Natural sand (fine aggregate) was collected from Ogun River and sieved (< 4.75 mm) whereas the coarse aggregate, granite (> 4.75 mm), was obtained from the Ibadan quarry. A 42.5N "Dangote cement" was the source of the ordinary Portland cement (OPC) used as it met the ASTM C150-07 standards (2007). Tabulated in Table 1 are the materials used in this study and their sources.

Table 1. Raw materials and their sources

Raw Material	Source
PET bottles	Unilag ^a water
Cement	Dangote cement ^b
Natural sand	Ogun River
Granite	Ibadan quarry
Water	pipe-borne water

^a University of Lagos; ^b Portland cement

2.2. Infrared spectroscopy

The sorting of the Unilag water bottles included separating the bottle cap (cover) and ink-stencilled plastic label from the bottle (container). They were then separately subjected to attenuated-total reflectance Fourier-transform infrared (ATR-FTIR) spectroscopy (Roosen et al., 2020), using an Agilent ATR-FTIR spectrometer,

running on a MicroLab FTIR software. Each analysis consisted of 32 scans with a resolution of 8 cm^{-1} in the wavenumber region of $4000\text{--}600\text{ cm}^{-1}$.

2.3. Mixing of materials

The weight-batching method was used for mixing the concrete. The quantities of materials required were calculated using a 1:1:2 mix ratio of cement, fine and coarse aggregates, respectively, for an M25 grade concrete (IS 456:2000, 2000). Table 2 shows the various mix batches used with 0.5%–10% PET; where the quantity of PET added to the concrete mix is expressed as a percentage of the total mass of the concrete (cement, fine and coarse aggregates). A water–cement ratio of 0.55 (w/w) was employed (Frigione, 2010).

Table 2. Mix design of M25 in cube and cylinder moulds

Mould ^a Code	Cube					Cylinder					
	Cement (Kg)	Sand (Kg)	Gravel (Kg)	PET added (Kg)	PET added (%)	Code	Cement (Kg)	Sand (Kg)	Gravel (Kg)	PET added (Kg)	PET added (%)
CU-1	1.908	2.120	4.027	0	0	CY-1	2.996	3.330	6.326	0	0
CU-2	1.898	2.109	4.007	0.040	0.5	CY-2	2.981	3.313	6.294	0.063	0.5
CU-3	1.889	2.099	3.987	0.081	1.0	CY-3	2.966	3.297	6.263	0.127	1.0
CU-4	1.870	2.078	3.946	0.161	2.0	CY-4	2.936	3.263	6.199	0.253	2.0
CU-5	1.813	2.014	3.826	0.403	5.0	CY-5	2.846	3.164	6.010	0.633	5.0
CU-6	1.717	1.908	3.624	0.805	10.0	CY-6	2.696	2.997	5.693	1.265	10.0

^a Dimensions: 150 mm x 150 mm x 150 mm (cube mould); 150 mm x 300 mm (cylinder mould)

2.4. Slump test

The workability of the composites was determined by the slump test according to the ASTM C143/C143M-10 standard (2010). A mould with a conical frustum of height (300 mm), top (100 mm) and base (200 mm) diameters was filled with freshly prepared composite mixes and compacted with a tamping rod. The frustum was then lifted vertically and the differences in height of the composite were computed.

2.5. Compressive strength test

Freshly prepared composites were poured into cube-shaped moulds (150 x 150 x 150 mm), in duplicates, compacted and their surfaces levelled with a trowel. The moulds were then allowed to dry at ambient temperature over 24 hours and subsequently de-moulded. Whence the dried composites were water-cured in the curing tank for 7–35 days. The samples were tested for compressive strengths after 7 days, 28 days and 35 days, respectively, according to the BS EN 12390-3 standard (2009).

2.6. Tensile strength test

The tensile splitting strengths of the composites were also measured, in duplicates, using cylindrically-cast moulds (150 x 300 mm), according to BS EN 12390-6 standard (2009) after 7 days, 28 days and 35 days of water-curing, respectively. The moulds were cast with freshly prepared composites, which were then compacted, dried at ambient temperature for 24 hours and de-moulded.

2.7. Water absorption test

The saturated water absorption test was carried out on the cube-shaped (150 x 150 x 150 mm) composite casts, after 35 days of curing, in cognisance with ASTM C642-06 (2006) and ASTM D570-98 standards (1998). The cured composites were removed from the curing tank and dried at an oven temperature of 105 °C for 24 hours, allowed to cool to ambient temperature and weighed (dry weight). The dried composites were subsequently immersed in water at 25 °C and taken out at 12-hour intervals, wiped with a dry cloth and weighed till constant weight (48 hours). The percentage of water absorbed was calculated as stipulated (ASTM C642-06, 2006) and recorded.

2.8. Acid resistance test

Cube-shaped (150 x 150 x 150 mm) composite casts were subjected to the acid attack test (ASTM C1898-20, 2020) after 28 days of water-curing. The composites were weighed and then immersed in 5% aqueous hydrochloric acid (HCl) solution (v/v) for 3 days, removed and re-weighed to calculate percentage weight loss. Standard deviations and coefficients of variation were also calculated.

2.9. Chloride attack test

Composites (150 x 150 x 150 mm) cured for 35 days were weighed before their complete immersion in tanks containing 5% aqueous sodium chloride (NaCl) solution (w/w) for 3 days. The cubes were subsequently retrieved from the salt tanks, wiped to dryness and weighed. The percentage loss of weight, standard deviations and coefficients of variation of the composites were collated.

3. Results

3.1. Slump test

The consistency of the freshly prepared PET–concrete composites was measured by the slump test, as shown in Table 3. The slump is observed to decrease with increase in the PET content of the concrete composite. Interestingly the PET–concrete composites showed no slump beyond 1% PET content.

Table 3. Slump performance

Composite	PET content (%)	Slump (mm)
0% PET^a	0	30
0.5% PET	0.5	20
1% PET	1.0	5
2% PET	2.0	0
5% PET	5.0	0
10% PET	10.0	0

^a Control

3.2. Compressive strength

The results of the compressive strength tests, carried out on the PET–concrete composite cubes (150 x 150 x 150 mm) are collected in Table 4. They were subjected to water curing for 7 days, 28 days and 35 days. In the

first 7 days, the compressive strengths peaked at 26 N/mm² with the 1% PET–concrete composite (CU-3); with concretes of higher PET contents showing lower compressive strength values compared to the control (CU-1). The peaks shifted to CU-4 (with 2% PET content) at 30.22 N/mm² and 30 N/mm², after 28 days and 35 days of curing, respectively. The effects of the addition of PET on the compressive strengths of concrete were varied as both increases and decreases in compression tensions were observed with increase in PET contents. Notably, the introduction of 1% PET exhibited increased compressive strengths after 7 days (26 N/mm²), 28 days (28.8 N/mm²) and 35 days (29.1 N/mm²), respectively, when compared to those of the control concrete (25.3 N/mm² and 27.3 N/mm²).

Table 4. Compressive strength of PET–concrete composites

Code	PET content (%)	Composite		Average Compressive strength (N/mm ²)	Change in Compressive strength (N/mm ²)	Change in Compressive strength over days (N/mm ²)
		1	2			
7 days^a						
CU-1	0	24.89	25.78	25.34	--	--
CU-2	0.5	25.33	25.56	25.45	+0.11	--
CU-3	1.0	25.33	26.67	26.00	+0.66	--
CU-4	2.0	24.44	25.33	24.89	-0.45	--
CU-5	5.0	18.44	17.11	17.78	-7.56	--
CU-6	10.0	14.67	12.67	13.67	-11.67	--
28 days^a						
CU-1	0	25.78	28.89	27.33	--	+1.99
CU-2	0.5	28.44	29.44	28.89	+1.56	+3.44
CU-3	1.0	27.56	30.00	28.78	+1.45	+2.78
CU-4	2.0	29.33	31.11	30.22	+2.89	+5.33
CU-5	5.0	23.56	23.11	23.34	-3.99	+5.56
CU-6	10.0	19.56	19.11	19.34	-7.99	+5.67
35 days^a						
CU-1	0	28.44	28.89	27.33	--	+1.99
CU-2	0.5	25.33	27.56	26.44	-0.89	+0.99
CU-3	1.0	30.22	28.00	29.11	+1.78	+3.11
CU-4	2.0	28.89	31.11	30.00	+2.67	+5.11
CU-5	5.0	20.89	22.67	21.78	-5.55	+4.00
CU-6	10.0	20.44	17.78	19.11	-8.22	+5.44

^aCuring period.

3.3. Tensile strength

The cylindrical PET–concrete specimen moulds (150 x 300 mm) were subjected to tensile strength tests after 7 days, 28 days and 35 days of water-curing (Table 5). The 1% PET–concrete composite (CY-3) gave the highest average tensile strength value of 1.45 N/mm² after 7 days while CY-2 (0.5% PET) and CY-4 (2% PET) recorded the highest average tensile strength value of 1.59 N/mm² after 28 days and 35 days of curing, respectively. It

can be surmised, therefore, that the average tensile strength of the PET–concrete composites peaked with CY-3 (1% PET), CY-2 (0.5% PET) and CY-4 (2% PET) after 7 days, 28 days and 35 days of curing, respectively.

3.4. Water absorption test

This test was used to determine the rate of absorption (sorptivity) of water by hydraulic PET–cement concrete composites by measuring the increases in their weights on immersion (in water), over time due to their water absorption. The saturated water absorption potentials of the PET–concrete composites were investigated after 35 days of water-curing. The weights of water absorbed by the composites over 12–48 hours were calculated (ASTM C642-06, 2006) and recorded as percentages in Table 6. All the PET-containing concrete composites absorbed less water (8.37%–13.17%) than the control concrete (13.37%); with the 5% PET and 1% PET composites recording the lowest and highest percentage water absorptions, respectively.

Table 5. Tensile strength of PET–concrete composites

Code	PET content (%)	Composite		Average Tensile strength (N/mm ²)	Change in Tensile strength (N/mm ²)	Change in Tensile strength over days (N/mm ²)
		1	2			
7 days^a						
CY-1	0	0.85	0.78	0.81	--	--
CY-2	0.5	0.99	1.06	1.02	+0.21	--
CY-3	1.0	1.41	1.49	1.45	+0.64	--
CY-4	2.0	0.85	0.92	0.88	+0.07	--
CY-5	5.0	0.78	0.78	0.78	-0.03	--
CY-6	10.0	0.71	0.71	0.71	-0.10	--
28 days^a						
CY-1	0	1.06	1.13	1.10	--	+0.29
CY-2	0.5	1.56	1.63	1.59	+0.49	+0.57
CY-3	1.0	0.99	1.20	1.10	0	-0.35
CY-4	2.0	1.20	1.20	1.20	+0.10	+0.32
CY-5	5.0	1.27	1.34	1.31	+0.21	+0.53
CY-6	10.0	0.99	1.13	1.06	-0.04	+0.35
35 days^a						
CY-1	0	1.27	1.34	1.30	--	+0.49
CY-2	0.5	1.34	1.34	1.34	+0.04	+0.32
CY-3	1.0	1.41	1.27	1.34	+0.04	-0.11
CY-4	2.0	1.56	1.63	1.59	+0.29	+0.71
CY-5	5.0	1.34	1.41	1.38	+0.08	+0.60
CY-6	10.0	1.13	1.13	1.13	-0.17	+0.42

^aCuring period.

3.5. Acid resistance test

The results of the acid resistance test (ASTM C1898-20, 2020) in 5% aqueous HCl, conducted after 28 days of curing the cube-shaped composites, are presented as percentage weight losses in Table 7. A progressive decrease in percentage weight loss was observed with increase in the PET contents of the composites, ranging

from 9.45% (0% PET) to 4.85% (10% PET). Standard deviations and coefficients of variation of 0.28% (2% PET) to 1.7% (5% PET) and 0.05% (0% PET and 2% PET) to 0.3% (5% PET), respectively, were recorded.

Table 6. Saturated water absorption test

Duration (hours)	12	24	36	48	
Composite	Weight Change (Kg)				Absorption (%)
0% PET^a	6.88	7.33	7.80	7.80	13.37
0.5% PET	8.59	9.95	9.50	9.50	10.59
1% PET	8.58	9.06	9.71	9.71	13.17
2% PET	9.01	9.62	9.87	9.87	9.54
5% PET	9.32	9.71	10.10	10.10	8.37
10% PET	9.34	9.87	10.23	10.23	9.53

^a Control

Table 7. Acid resistance and chloride attack tests

Test		Weight loss (%)					
		0% PET^a	0.5% PET	1% PET	2% PET	5% PET	10% PET
Acid resistance	C1	9.00	9.01	5.59	5.42	3.93	3.98
	C2	9.89	5.71	6.98	5.98	7.33	5.71
	μ	9.45	7.36	6.29	5.70	5.63	4.85
	σ	0.45	1.65	0.70	0.28	1.70	0.87
	CV	0.05	0.22	0.11	0.05	0.30	0.18
Chloride attack	C1	1.52	1.98	1.41	2.72	3.83	2.96
	C2	1.11	1.01	1.92	1.51	2.04	3.15
	μ	1.32	1.50	1.67	2.12	2.94	3.06
	σ	0.21	0.49	0.26	0.61	0.90	0.10
	CV	0.16	0.32	0.15	0.29	0.30	0.03

^a Control, C1 & C2: composites, μ : mean, σ : standard deviation, CV: coefficient of variation

3.6. Chloride attack test

Water-soluble chlorides are capable of initiating and/or accelerating corrosion in concrete composites when present in sufficient amounts (ASTM C1218/C1218M-99, 1999). Consequently, the determination of chlorides in hydraulic-cement concretes is pertinent in order to evaluate the potentials of a cement system liable to and/or undergoing such reactions. The interaction of immersed PET-concrete composites with 5% aqueous NaCl solution; measured by the percentage weight loss of the composites, was examined.

The percentage weight loss values, obtained from the immersion of the cube composites in 5% aqueous NaCl for 3 days, are also collated in Table 7. Interestingly, increasing the PET contents of the concrete composites led to increases in the percentage weight loss of the composites. The standard deviation ranged between 0.1% (10% PET) and 0.9% (5% PET) while their coefficient of variation values ranged from 0.03% (10% PET) to 0.32% (0.5% PET).

4. Discussion

The incorporation of PET wastes as reinforcement or partial replacement of natural aggregates in concrete is a sustainable and environmentally benign way of managing and/or reducing the nuisance of plastic wastes (Kazemi et al., 2021). It can also impart synergistic properties, such as anti-cracks (Foti, 2013), strength and electrical resistance (Sadrumontazi et al., 2016) as well as ductility (Yi et al., 2015). The three-pronged goal to reduce PET bottle wastes, develop lightweight PET-concrete composite building materials and, in so doing, conserve some natural construction resources began with the collection and subsequent sorting and shredding of the bottles. The sorting protocol of separating the different packaging components of the water bottle wastes, such as the bottle container, cover (cap) and label, were crucial to avoiding recycling problems; as can be encountered in closed-loop recycling, for instance (Barjoveanu et al., 2023). Also, this can make the inclusion of advanced pre-treatment steps inevitable thereby increasing recycling costs (Roosen et al., 2020). The shredding of the PET bottles into small fragments (< 4.75 mm), on the other hand, was carried out to improve its dispersibility within the concrete mixture (Osubor et al., 2019; Silva et al., 2005).

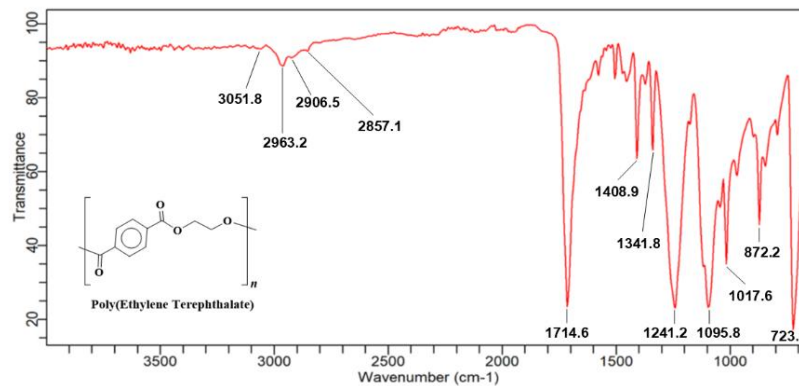


Figure 2. ATR-FTIR spectrum of (Unilag) water bottle, showing strong stretching and bending frequencies for poly(ethylene terephthalate) – PET.

Pertinently, prior to micronisation, the infrared spectrum of the PET bottle was obtained to confirm its microstructure (Chen et al., 2012; Roosen et al., 2020). Accordingly, stretching bond frequencies were observed at 3052 cm^{-1} (aromatic C–H; weak), 2963 cm^{-1} and 2907 cm^{-1} (aliphatic C–H; medium), 1715 cm^{-1} (ester C=O; strong), 1241 cm^{-1} (ester O=C–O; strong) and 1096 cm^{-1} (ester C–O–C; strong), respectively, as well as the characteristic aromatic C–H bond bending absorption at 723 cm^{-1} (strong); as shown in Figure 2. These are in agreement with previous reports (Chen et al., 2012; dos Santos Pereira et al., 2017; Smith, 2022).

Furthermore, the control concrete composite (0% PET) was prepared to M25 grade, which has a high-strength-bearing capacity and is commonly used in the construction of foundations, columns and beams, via the weight-batching method, according to standard (IS 456:2000, 2000). The components (cement, fine and coarse aggregates) of the concrete (0% PET) were mixed in a 1:1:2 (w/w) ratio, with a water–cement ratio of 0.55 (w/w), whereas PET was added as a percentage (0.5%–10%) of the total mass of the concrete;

maintaining the 1:1:2 ratio of the concrete's components, to furnish the PET-concrete composite mixtures (cf. Table 2).

The results of the slump test carried out on the PET-concrete composites, shown in Table 3, indicated a decrease in composite fluidity with increase in the PET content of the concrete composites (cf. Figure 3). It is noticeable that with $\geq 2\%$ PET content, the concrete showed no slump and that the introduction of 0.5% and 1% PET contents led to 33% and 83% reductions, respectively, in the workability of the PET-concrete composites in comparison to the control concrete (without PET). This is in agreement with previous reports where the workability of fresh concrete was inversely proportional to the polymeric material's content (Dinesh and Rao, 2017). Nonetheless, the reduction in the slump of the control concrete (0% PET) on the addition of PET (0.5–10% PET) can be allayed with plasticisers for construction purposes (Titiksh and Wanjari, 2022). The workability of the composites could also be improved by increasing the water-cement ratio (to > 0.55 w/w) but with loss in mechanical properties (Suraweera and De Silva, 2023).

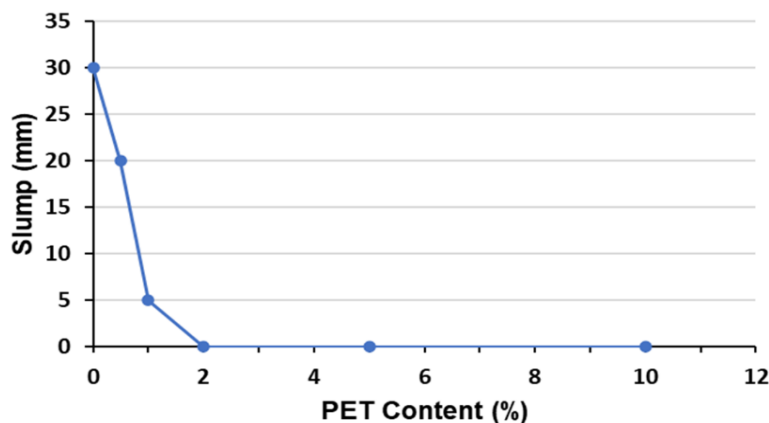


Figure 3. Slump test for PET-concrete composites

Next, the compressive strengths of the composites were tested, after casting and water-curing, to determine the maximum mechanical capacity (load) they can withstand before failure (Jaya, 2020). In the first 7 days, increases in the compressive strengths of the PET-concrete composites peaked with the concrete with 1% PET content (CU-3; $+0.66$ N/mm², cf. Table 4) whereas the peak was observed with the 2% PET-concrete composite (CU-4; $+2.89$ N/mm²) after 28 days. It is plausible that, for the concretes with low PET contents, the increases in compressive strength were due to the increased possibility of inter-locking between PET particles on the fractured surfaces, when the loads reached their maxima, because of their special shapes and flexibility (Rahmani et al., 2013). Conversely, after the 35-day period, CU-2 (0.5% PET; -0.89 N/mm²), CU-5 (5% PET; -5.55 N/mm²) and CU-6 (10% PET; -8.22 N/mm²) experienced reductions in their compressive strengths, respectively, while CU-3 (1% PET; $+1.78$ N/mm²) and CU-4 (2% PET; $+2.67$ N/mm²) increased. It is also notable that all the PET-containing specimens (CU-2–CU-6) showed increases in compressive strength with increasing number of days of curing albeit at varying margins.

Figure 4 shows the average compressive strength values of the PET-concrete composite materials over 7–35 days. It illustrates an initial increase in average compressive strength; peaking between 1–2% PET before

decreasing with increasing PET content. Additionally, average compressive strength increased after the 28-day curing but appeared to decrease 7 days post-28-days of water-curing (i.e., after 35 days; cf. Figure 4).

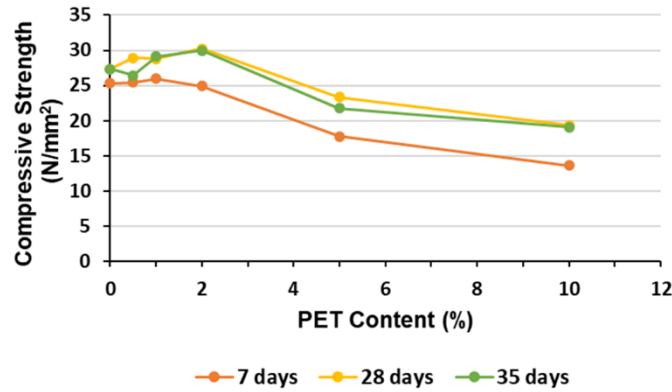


Figure 4. Average compressive strengths of PET-concrete composites

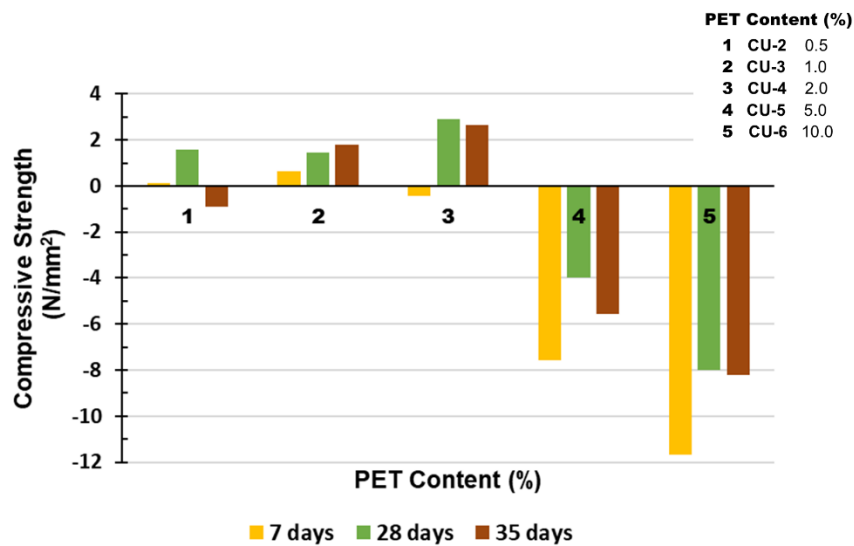


Figure 5. Change in compressive strength of PET-concrete composites

Depicted in Figure 5 are the changes in compressive strengths of the PET-concrete composite materials over 7–35 days, in positive and negative bars whereas Figure 6 shows the changes in compressive strength over days, after the 28-day and 35-day periods. It is evident from Figure 5 that CU-2 (0.5% PET) experienced a negative change in compressive strength after 35 days whereas CU-4 (2.0% PET) recorded a similar negative value but of lower magnitude after 7 days of curing. It is instructive that the specimens of higher PET content (5–10%) gave negative compressive strength values in the period examined whereas CU-3; with a 1% PET content, exhibited a progressively positive change in its compressive strength in the same 7–35-day period, indicating a direct correlation of the composite’s (CU-3) compressive strength with increasing curing time.

A similar scenario was observed with the values of PET-concrete composites' change in compressive strength over days (*cf.* Figure 6) where positive changes were recorded after the 28-day and 35-day periods. Furthermore, a positive differential was obtained for CU-3 (1% PET) within these time frames while the converse was the case for the other composite specimens. Significantly, the control (CU-1); with zero PET content, was unchanged (Table 4 and Figure 6).

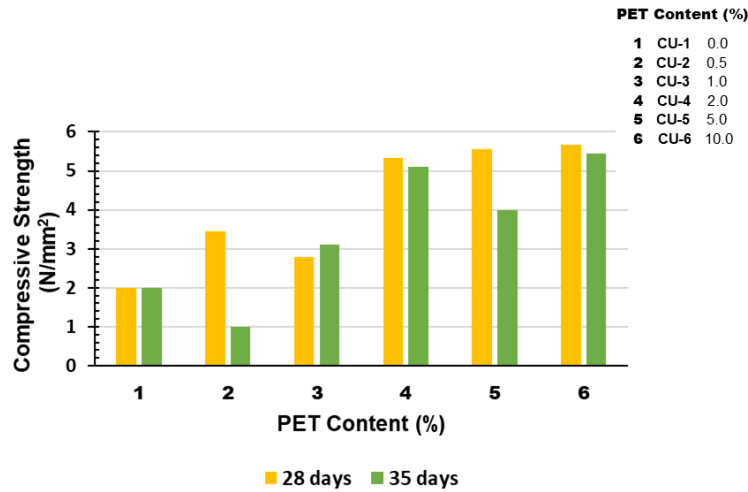


Figure 6. Change in compressive strength over days of PET-concrete composites

It can be gleaned from the foregoing, therefore, that the addition of PET to concrete and their curing times both affected the composites' compressive strengths. With $\leq 2\%$ PET content, increases in the compressive strength values were observed after a 28-day curing period whereas the converse was the case for $\geq 5\%$ PET content. These decreases in compressive strengths of PET-concrete composites with $> 5\%$ PET content have been previously reported by other authors (Osubor et al., 2019). Likewise, the increases in compressive strengths after 7 days and 28 days of curing and subsequent decreases corroborate the notion that concrete gains compressive strength rapidly at the early stages of curing but then increases relatively slowly going forward (Verma et al., 2022).

The tensile strengths of the PET-concrete composites were also determined though tensile stress typically accounts for 7%–15% of compressive stress (Liao et al., 2020). The determination of tensile strength in concrete is equally vital in terms of durability and serviceability (determined via transverse shear, torsion, shrinkage and temperature effects, for example) as well as for making pre-stressed and liquid-retaining structures, roadways and/or runway slabs but of little consequence in the design of reinforced concrete (IS 5816:1999, 1999; Liao et al., 2020).

Figure 7 graphically depicts the average tensile strength trends of the PET-concrete composites after 7–35 days of curing. An initial increase in average tensile strength was observed with the 0.5% PET-concrete composites, in comparison to the control concrete (0% PET); which also showed increased average tensile strength values after the 7-day, 28-day and 35-day curing periods. It is noteworthy that the average tensile strength of the concrete without PET (CY-1) increased with prolonged curing times; i.e., 0.81 N/mm² (7 days) → 1.1 N/mm² (28 days) → 1.3 N/mm² (35 days). Interestingly, CY-3 (1% PET) recorded the same tensile

strength value (1.1 N/mm²) as CY-1 (0% PET) after 28 days thereby showing no change in tensile strength (cf. Table 5).

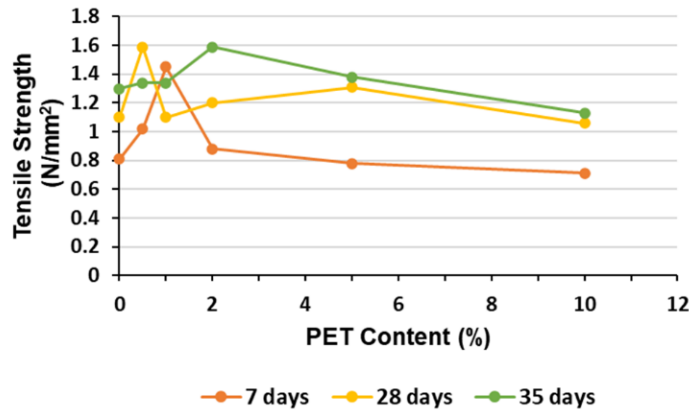


Figure 7. Average tensile strengths of PET-concrete composites

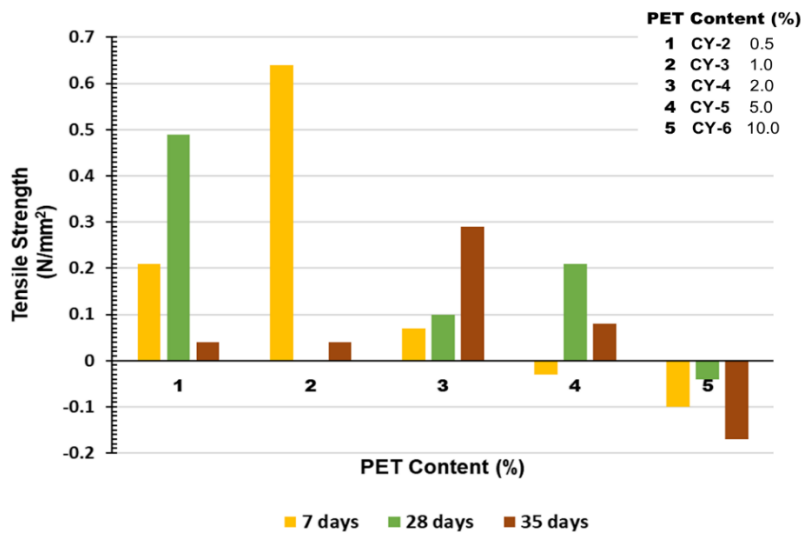


Figure 8. Change in tensile strength of PET-concrete composites

The net changes observed in the tensile strengths of the PET-concrete composites over 7–35 days are shown in Figure 8. Specimen CY-4 (2% PET), labelled **3** in Figure 8, showed a progressive net increase in the composite’s change in tensile strength in the 7–35-day period thereby demonstrating that a positive change in the composite’s tensile strength is associated with extended curing times. Contrarily, CY-2 (0.5% PET, **1**) showed a net positive change up to 28 days but declined after 35 days whereas CY-3 (1% PET, **2**) recorded a decrease in its change in tensile strength between the 7-day and 28-day periods but increased after 35 days. Besides, CY-3 recorded a zero change in tensile strength after 28 days. In addition, CY-6 (10% PET, **5**) gave

negative changes in tensile strengths over 7–35 days while CY-5 (5% PET, 4) only gave a negative change in the 7-day period.

The changes in the tensile strengths over days of the PET–concrete composites, after 28 days and 35 days of curing are represented in Figure 9. All of the specimens tested showed positive changes in the period except CY-3 (1% PET, 3), which recorded values of -0.35 N/mm^2 (28 days) and -0.11 N/mm^2 (35 days), respectively. The changes in tensile strength over days also increased with increased curing time except for CY-2 (0.5% PET, 2) and CY-3 (1% PET, 3).

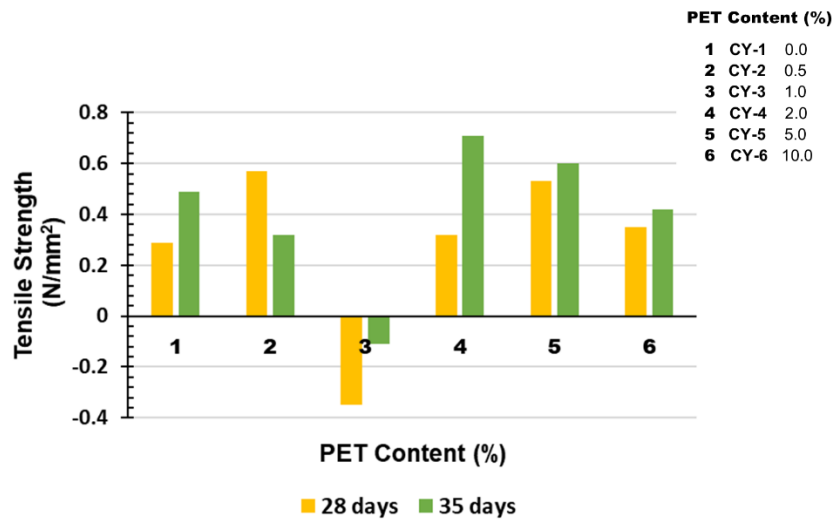


Figure 9. Change in tensile strength over days of PET–concrete composites

A corollary of the mechanical properties of the composites prepared herein showed, therefore, that the addition of 1%–2 % by weight of PET to concrete improved its compressive and tensile strengths by $0.66\text{--}2.89 \text{ N/mm}^2$ (2.6%–10.6%) and $0.04\text{--}0.64 \text{ N/mm}^2$ (3.1%–79%), respectively. These also increased with ageing as changes in compressive strength over days peaked at 5.67 N/mm^2 (10% PET) after 28 days and 5.44 N/mm^2 (10% PET) after 35 days whereas changes in tensile strength over days peaked at 0.57 N/mm^2 (0.5% PET; 28 days) and 0.71 N/mm^2 (2% PET; 35 days), respectively. These increases in strength may be attributable to the homologous binary blends of the composites. The grated PET used was fine ($< 4.75 \text{ mm}$) and was expected to minimise air-filled pores (voids), amongst others, in the PET–concrete composites in order to yield composites of higher unit weights and superior packing densities. This is because concretes of higher unit weights are known to exhibit better strengths; typically accomplished by the adequate packing of their constituent materials (Prithiviraj et al., 2022). In addition, the inclusion of PET in the concrete composite could also be accelerating the hydration of cement, which can cause increases in mechanical strengths (Kim et al. 2021; Umasabor and Daniel 2020). In contrast, the decreases observed in the mechanical strengths of composites beyond the 5% PET content have been attributed to decreases in adhesive strengths between the PET surfaces and cement (Umasabor and Daniel 2020).

The PET–concrete composites were also subjected to durability tests by immersion in water and aqueous solutions of mineral acid and sodium chloride, with varying outcomes. The water absorption test was employed to assess the water sorptivity of the composites according to ASTM standards (ASTM C642-06, 2006;

ASTM D570-98, 1998). It is generally regarded as a good gauge of the concrete's durability because the resistance of concrete to water penetration is directly proportional to the concrete's rate of water absorption (McCarter et al., 1992; Safiuddin et al., 2011). Moreover, water plays an important role in many of the deterioration mechanisms of concrete as well as transports various deleterious agents from the environment. Similarly, sulphate attack and alkali aggregate reactivity depend on the ingress of moisture into concrete through the process of water absorption, which also permits the conveyance of oxygen, carbon dioxide and salts, such as chlorides, that can lead to corrosion (Safiuddin et al., 2011).

The saturated water absorption tests on the 35 days-cured PET-concrete composites showed water saturation within 36 hours of immersion; with all the PET-containing concrete composites absorbing less water than the control concrete (0% PET). This indicates that the introduction of PET into the concrete was advantageous as it resulted in the reduced absorption of water though at different rates; somewhat inducing a pozzolanic effect on the concrete (Sadrumontazi et al., 2016). It is plausible that the addition of PET to the concrete composite yielded a denser interparticle microstructure, which resulted in the reduced absorption of water observed (Titiksh and Wanjari, 2022). Notably, 1% PET and 5% PET experienced the least and most effects, relative to the control concrete (0% PET; 13.4%), with 13.2% and 8.4% absorptions, respectively (*cf.* Table 6 and Figure 10). This bodes well for the utility and sustainability of the PET-concrete composites prepared herein as water in hardened concrete is known to induce cracks and shrinkages (Cortas et al., 2014; Safiuddin et al., 2011). Additionally, concretes with high water sorptivity are reported to be more prone to damages caused by freeze-thaw cycles (Shang et al., 2012).

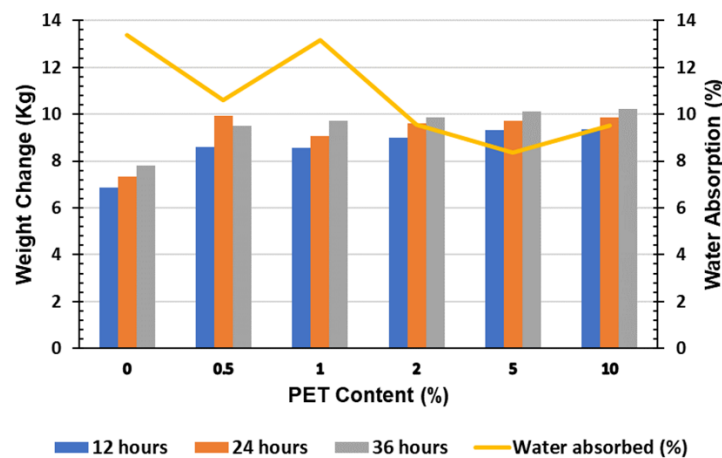


Figure 10. Saturated water absorption tests of PET-concrete composites

It can be also gathered from Figure 10 that the absorption of water increased over time (i.e., 12 h → 24 h → 36 h) in all of the composites prepared, including the control concrete (0% PET), bar 0.5% PET; which atypically exhibited a reduction in the quantity of water absorbed after 24 hours. It is probable that the flexibility, enabled by the possible interlocking interactions between the low-content (0.5%) PET particles and concrete composite, is responsible for the observed reduction in absorbed water (Rahmani et al., 2013). It is equally notable that the percentage water absorbed by the PET-containing concretes decreased with corresponding increases in their PET contents; with the exception of the composites containing 1% and 10%

of PET (*cf.* Figure 10). It would be remiss not to mention that the results of water absorption tests can be considerably influenced by sample preparation due to ambient relative humidity but, significantly, specimens conditioned in the oven at 105 °C, as was done herein, appear to be unaffected (Castro et al., 2011).

The exposure of concrete to acid solutions to assess its vulnerability and durability is important because of its susceptibility to acids, which are usually introduced into the environment from fossil fuels, domestic and industrial discharges, and acid rain as well as anaerobic bacteria (Raghunathan, 2021). The reaction of acid rain or acidic atmospheric particulates with the calcium hydroxide constituent of cement often leads to the degradation of the majorly alkaline concrete. Furthermore, it has been suggested that the degree of resistance of concrete to acid attack is linked to its impenetrability rather than the cement-type used in its preparation (Raghunathan, 2021).

It is pertinent to note, therefore, that the addition of PET to concrete herein improved its resistance to acid attack as the percentage weight loss of the composites decreased with increasing PET content (Figure 11). It is conceivable that the presence of PET particles and its interlocking interactions with concrete increased the impenetrability of the PET–concrete composites; rendering them better able to resist the corrosive effects of HCl, as proposed by Rahmani et al. (2013). The standard deviation and coefficient of variation values obtained also showed within-test variabilities for the composites.

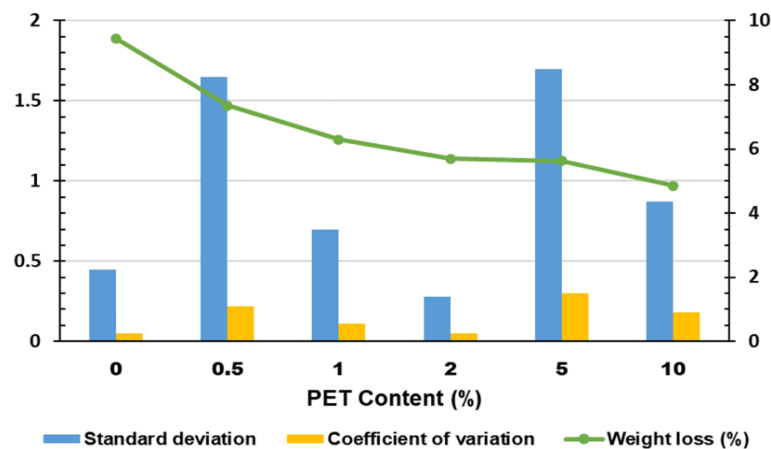


Figure 11. Acid resistance tests of PET–concrete composites

Conversely, Figure 12; which illustrates the effect of 5% aqueous NaCl solution on the PET–concrete composites, depicts increased percentage weight losses in the concrete composites with increasing PET content. The plot shows a steady rise in the percentage weight losses of the composites from 1.5% to 3.1% as their PET contents increased from 0.5% to 10% by weight. The 5% PET–concrete composite exhibited the highest standard deviation of 0.9% whereas the highest coefficient of variation recorded was that of the 0.5% PET–concrete at 0.32%. The results indicate, therefore, that the PET-containing composites were more vulnerable to saltwater penetration than the control concrete. Similarly, it is significant to point out that the performance, strength and durability of concrete structures are also affected by chlorides and sulphates (Mangi et al., 2019a), and that they deteriorate more rapidly under these aggressive environmental conditions.

The implication, therefore, is that the control concrete (0% PET) is less susceptible to chloride ion attack or penetration than the PET-containing composites (0.5%–10% PET).

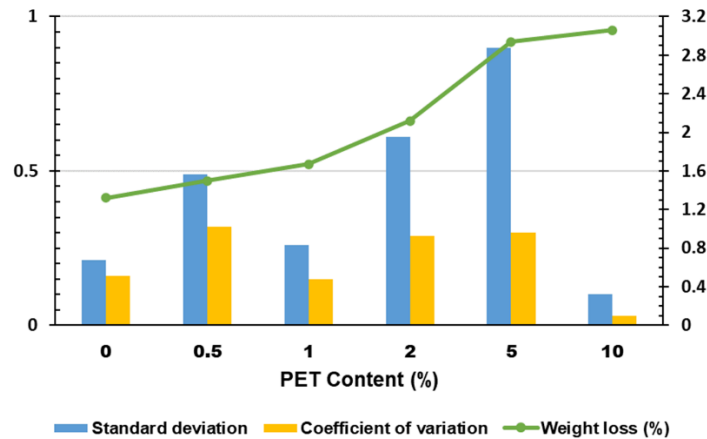


Figure 12. Chloride attack tests of PET-concrete composites

Consequently, it could be inferred that the use of these materials for construction in marine or coastal environments can be problematic (Mangi et al., 2019b). However, this may be mitigated by the use of seawater in the mixing and curing processes of concrete production (Wegian, 2010) whereas chlorides in concrete are determined via the silver nitrate titrate method (Yu et al., 2023). It is generally accepted that the durability of concrete is largely governed by its resistance to the penetration of aggressive aqueous and ionic substances (Noushini et al., 2020). Subsequently, it can be surmised that the incorporation of PET in concrete enhanced the durability and serviceability of the resulting PET-concrete composites as it afforded specimens of reduced water sorptivity and improved resistance to acid attack but more susceptible to saltwater permeability. This renders them less suitable for direct use in marine environments without additional treatments, such as the use of protective coatings or additives, to mitigate this limitation.

Contextually, it is instructive to note that, contrary to the observations enunciated above, the incorporation of powdered PET into Portland cement paste alone has been reported to yield composites of progressively lower compressive strengths (compared to the control) as the PET content was increased (Kim et al. 2021). However, as has been suggested, such PET-incorporated cementitious materials may be unaffected by harsh environmental conditions; implying that the composites may be able to reduce the generation and pervasiveness of microplastics in the environment by immobilising the plastics and retarding their degradative transformations (Kim et al. 2021). Therefore, varying and, sometimes, opposing outcomes could be evident when PET is combined with cement paste, in comparison to when it is incorporated into concrete (Kim et al. 2021; Tripathi, 2024).

Lastly, a cursory life cycle assessment (LCA) (Barjoveanu et al., 2023; Muralikrishna and Manickam, 2017; Rispoli and Ajibade, 2024) to quantify and analyse the environmental impacts, cost implications and sustainability of incorporating PET wastes into concrete revealed a reduction in the quantity of available PET bottle wastes and a 1%–11% reduction in cement, sand and gravel; with projected concomitant reductions in ancillary activities and productions, including the evolution of CO₂, emission of pollutants; such as CO, Pb, PM₁₀,

NO_x and SO₂, and volatile organic compounds as well as electricity and transportation costs (Gursel, 2014). In addition, the project's lower carbon footprint and sustainability are accentuated by the possible applications of the resulting PET-concrete composites, which can find utility in construction as pavers and slabs, for example (IS 456:2000, 2000; IS 15658:2006, 2006; Titiksh and Wanjari, 2022).

5. Conclusions

The major purpose of this study was to reduce plastic wastes, in the form of used PET (water) bottles, in the environment by incorporating them into concrete to furnish lightweight PET-concrete composites, which can find utility as robust building materials, and conserve some natural resources used in the construction industry.

0.5%–10% by weight of grated PET were added to the constituents of concrete in ratios of 1:1:2, mixed to cast and water-cured for 7–35 days. It was observed that the addition of PET progressively reduced the workability of the concrete. On the other hand, the mechanical properties' analyses of the PET-concrete composites showed that the addition of 1%–2% PET improved the compressive strengths by 0.66–2.89 N/mm² and tensile strengths by 0.04–0.64 N/mm²; leading to 2.6%–10.6% and 3.1%–79% increments, respectively; with most of the composites holding > 2% PET contents recording lower compressive and tensile strengths in comparison to the control composite (0% PET). It is noteworthy, however, that the 5% PET-concrete composite also exhibited enhanced tensile strengths after 28 days and 35 days of curing. Furthermore, these mechanical properties also improved with longer curing times as changes in tensile strength over days peaked at 0.57 N/mm² (0.5% PET; 28 days) and 0.71 N/mm² (2% PET; 35 days) whereas the changes in compressive strength over days peaked in the 10% PET composite at 5.67 N/mm² and 5.44 N/mm² after 28 days and 35 days, respectively.

Besides, the PET-concrete composites displayed water saturation within 36 hours of immersion and all the concrete composites with 0.5%–10% PET absorbed less water than the 0% PET composite. It is worthy of note that the composites with 1% and 5% PET contents experienced the least and most effects, with 13.2% and 8.4% absorptions, respectively; compared to the control concrete (0% PET; 13.4%). In the same vein, the composites' resistance to HCl attacks were higher as a progressive decrease in percentage weight loss was observed with increased PET content from 9.45% (0% PET) to 4.85% (10% PET). These durability tests indicate that incorporating PET into concrete can reduce water absorption and increase acid resistance. Gratifyingly, these attributes augur well for the utility and sustainability of the PET-concrete composites prepared herein. On the contrary, the interaction of the PET-containing composites with 5% aqueous NaCl solution resulted in increases in the percentage weight losses of the composites from 1.5% to 3.1%, with increasing PET contents (0.5% → 10%) thereby rendering the control concrete less susceptible to saltwater penetration. Nevertheless, this drawback can be alleviated at the mixing and curing stages of producing the PET-concrete composites. A corollary is therefore that the PET-based composites offer improved durability in water absorption and acid resistance but their increased vulnerability to saltwater underscores the need for targeted solutions for their applications in marine or coastal environments.

The preliminary life cycle assessment conducted to gauge the composites' ecological impact and cost implications was agreeable and positive. It is, therefore, safe to surmise that the preparation of the PET-concrete composites is a feasible and sustainable approach to PET plastic waste management and the

production of robust, non-marine building materials for pavements, slabs and lightweight construction panels. Additionally, it is a veritable route to the conservation of some of nature's resources, including sand and granite.

Declarations

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Conflict of interest

The authors declare no conflict of interest.

Funding

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Data availability statement

The datasets generated during and/or analysed during this study are available upon request

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