Three-phases bubble column to polyethylene terephthalate depolymerization for cement mortar composites improvement

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ABSTRACT

Purpose: This paper aims to prepare depolymerized polyethylene terephthalate (DPET) powder from recycled plastic water bottles. Adding this DPET powder to the cement mortar was also studied.

Design/methodology/approach: The adopted PET depolymerization process includes the usage of both ethylene glycol (EG) as solvent and nano-MgO as a catalyst. A bubble column reactor was designed for this process. Five different mortar groups were made; each has different DPET content of 0%, 1%, 3%, 6% and 9% as a sand replacement. The flexural strength test and the water absorption measurement are done after two curing periods: 7 and 28 days.

Findings: The research finding demonstrated that the flexural strength of mortar was reduced by increasing the DPET powder percentage and the maximum dropping was 15% when 9% of DPET was added. The ability of the mortar to absorb the water was reduced by 14.5% when DPET powder was 9%. The mortar microstructure is featured with fewer cavities and porosity.

Research limitations/implications: This work's employed bubble column technique is limited only to the laboratory environment and needs to be scaled up within industrial mass production. For future research, it is suggested to decrease depolymerization time by using smaller pieces of plastic water bottle waste and trying other types of nanocatalyst.

Practical implications: The modified mortar can be utilized in areas where moisture, rainfalls, and sanitation systems exist.

Originality/value: The article claims that depolymerized waste PET improves chemical process efficiency by lowering reaction time and improving mass and heat transfer rates. Besides, this approach saves money. It is found out that the depolymerized plastic waste is much more functional due to its high cohesion capability than being used as small PET pieces.

Keywords: Depolymerization, DPET, Plastic waste, Bubble column, Mortar, Water absorption, Flexural strength

PROPERTIES

Research paper **5**
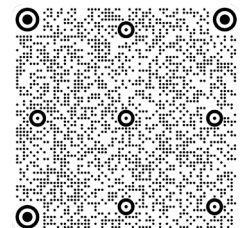
1. Introduction

The amount of all kinds of plastic waste used per year in the world has increased incredibly in the past fifty years. The increasing awareness of environmental issues has played a tremendous role in helping to control the huge plastic products waste accumulation. It has been believed that solid waste management is a massive environmental concern worldwide. As a result of the limited space on landfills and the increasing waste removal cost, waste plastics re-utilization has become one of the most practical and efficient alternatives for the disposal of plastic items [1].

Presently, there is a wide range of lightweight concrete applications that have been made with either artificial or natural lightweight aggregates. Nonetheless, the costs of producing the artificial lightweight aggregates are high due to the requirements of high temperature of incineration or thermal treatments [2].

The substitution method that has been provided by Ochi et al. [3] have proposed using waste PET bottles as PET fibres to produce lower-in-cost fibre reinforced concrete (FRC). The drawback of this approach is that it has a small volumetric fibre content in fibre the reinforced concrete ranging from 0.30 to 1.50%. Thereby, only a small amount of this waste can be used.

Utilizing the shredded waste PET bottles directly as aggregate in the production of the mortar or concrete is viewed as the most inexpensive approach. PET bottles present advantages in waste disposal and pollution reduction since the natural resources of the mineral aggregates can be preserved. However, adequate research has not been conducted yet on using the waste PET granules as aggregates in the mortar or concrete. In those studies, PET and other plastic wastes (PP and PE) were used to partially substitute mineral aggregates[4]. Reusing PET and other plastic types in the construction business is one of the most effective approaches in preventing environmental pollution and designing inexpensive buildings [5].

Ramadevi and Manju [6] researched the potential to use waste PET (WPET) bottles as a partial substitute for the cement aggregate. In that research, WPET fibre has swapped 0.50%, 1.0%, 2.0%, 4.0% and 6.0% volume of the sand. They found that the flexural strength has been increased when 2.0% sand is replaced with WPET, and it has been reduced gradually by 4%. the flexural strength stays the same at 6% WPET sand substitution. Compared to the other percentage values, fine aggregate replacement with 2% WPET fibres has reasonable properties.

Benosma et al. [7] studied the use of WPET in concrete contents. WPET aggregate was used in three different partial replacement percentages of 6%, 12%, and 17% cement weight. The findings revealed that the flexural strength of concrete, including WPET aggregate, was lower than that of concrete containing no WPET aggregate. Furthermore, this drop in the flexural strength got greater when increasing WPET, which flexural strength was 44% when the replacement was 17%.

Hasan et al. [8] studied the use of WPET fibre as coarse aggregate for concrete. Three partial replacements of 1%, 2%, and 3% replaced the coarse aggregate volume. The results showed that the flexural strength of the concrete increased by 65.2% when adding 2% of PET to the concrete.

One of the methods to reuse WPET is depolymerization. The depolymerized PET can be used in concrete or as mortar. The bubble columns approach is used to improve the depolymerization process' efficiency. The bubble column technique uses gas bubbles in the liquid alone or in combination with solid materials to achieve a high heat transfer rate. The multiphase conductor's technique is utilized in various industries, including petrochemicals and biochemicals. In terms of high-quality yield, the multiphase reactor is considered efficient. Furthermore, it has a wide range of uses. Bubble column reactors are employed because of its high operation characteristics, such as a high heat transfer coefficient [9,10].

The bubble column-equipped reactor is a cylindrical vessel with a gas inlet at the bottom to let gas bubbles form into the reactor. The cylindrical vessel has either a liquid or liquid-solid phase. There are some advantages of the bubble column. It is easy to remove or modify the catalyst during the reaction, and the effectiveness of the catalyst or other packing can persist for a long time [11,12].

In this study, the depolymerization process of PET is achieved using a bubble column reactor designed to utilize both EG as a solvent and Nano MgO as a catalyst. DPET was produced from the reaction and employed as a mortar modifier. Exploring and creating low-cost, environmentally friendly, and lightweight construction materials necessitate the development of more flexible, innovative, and diverse composite materials.

2. Experimental work

2.1. Constituents

Ordinary Portland Cement (OPC) type II was utilized in the current research. The OPC is available in the local markets and is termed commercially as (TASLUJA) manufactured in Al-Sulaimaniya-Iraq. The chemical and physical specifications of the OPC are presented in Table 1 and Table 2.

Table 1.
Chemical composition of OPC type II

Chemical composition	% by weight	Spec. limit
SiO_2	19.6	-
Fe_2O_3	3.7	-
CaO	61.35	-
MgO	2.24	<5.00
SO_3	1.36	<2.80
Al_2O_3	4.8	-
Loss on ignition (LOI)	1.45	<4
(Vicat´s approach) Setting time		
Initial setting	147	>45 min
Final setting	276	<10 hrs.
Time saturation factor (TSF)	0.89	0.66-1.02
Insoluble residue (IR)	0.97	<1.50

Table 2.
Physical properties of OPC type II

Physical properties	Test result	Spec. limit
Fineness (cm^2/g) by Blaine method	2733	>2300
Compressive strength for OPC mortar cube (70.7mm) at, (in MPa)		
3days	17.11	>15
7days	24.14	>23
Soundness using Auto clave%	0.24	<0.8
(Vicat´s approach) Setting time		
Initial setting	147	>45 min
Final setting	276	<10 hrs.

AL–Ukhaidher sand has been used in the experiments. It comes from Karbala-Iraq and conforms to Iraqi standard specification No. 45/1984. The sand passed through a sieve size of 4.75 mm and was characterized by the bulk specific gravity of 2.56, fineness modulus of 2.62, and sulfate content of 0.09% of the sand weight. Figure 1 presents the sand grading.
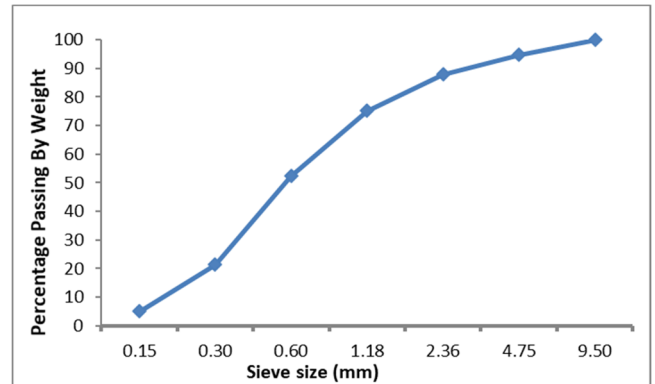

Fig. 1. Grading of AL–Ukhaidher sand

The production of DPET powder follows the following steps: the PET waste bottles were turned into smaller pieces. The bottles were firstly washed with distilled water and dried by air. Then, they were cut into smaller pieces of 2-4 mm with scissors, as seen in Figure 2 [13].

A 100 g PET has been depolymerized in a three-phase bubble column reactor (see Fig. 3). A 0.1 g of 60 nm nano-MgO was employed as a catalyst and 100 g of EG as a solvent. Nitrogen gas was supplied from the bottom side of the reactor by an electrical air pump. The experiment device was equipped with a vertical condensers cooler to provide a total condensation of the vapours [14,15]. Figure 3 illustrates the bubbles column device.

The bubbles were formed when the gas passed through the ceramic filter (fixed inside the reactor), which acts as a sparger. Then, the bubbles penetrate the mixture to boost the rate of the mixture heat transfer. The reactor's mixture is made up of liquid EG and solid PET particles. The mixture is entirely depolymerized and transformed into a homogeneous liquid phase after 40 to 50 minutes at 197°C (EG's boiling point) [16].
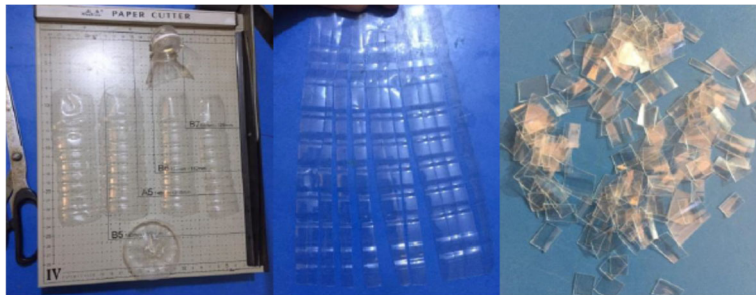

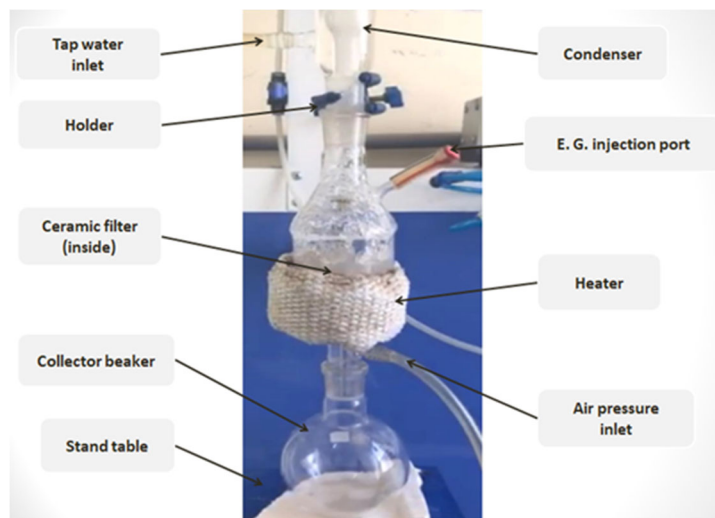Fig. 2. Turning PET bottle waste into smaller pieces

Fig. 3. Bubbles column device

The nitrogen gas stopped after obtaining the homogenous liquid phase and allowing the homogenous liquid to drop down in the beaker. The homogenous liquid is dried to become solid at room temperature. The final product consists of DPET and EG, and consequently, it is separated (see Fig. 4) [16].

Fig. 4. The homogenous white DPET and EG product of PET depolymerized

DPET was obtained as a liquid after heating the white material at a temperature of 197°C (Fig. 5). The DPET liquid is poured into a plane to freeze at room temperature. Finally, a milling machine milled the frozen DPET into powder form.

The DPET powder is washed with 2 g DPET/10 ml distilled water and heated and mixed with a magnetic stirrer hot plate device to guarantee it is free of any EG remains. The remaining EG is dissolved and then filtered. Ultimately,

the DPET powder is dried and then milled. A 100 g of white DPET particles was obtained.
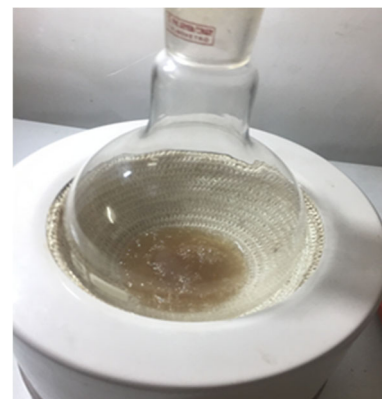
a)

b)

Fig. 5. The Physical split-up process. B. DPET residual in the beaker

2.2. Design of the mixtures

Two mixes were prepared in the laboratory, one without DPET and one with DPET. The mixture containing DPET has been further grouped into four mixtures depending on DPET content. For each mixture, the proportion of the fine aggregates to OPC (binder) was set to 2.75:1, and the proportion of water to OPC was fixed at 0.480. The mixture quantities are listed in Table 3.

3. The procedure of work

3.1. Test specimen preparation

The manner in which mixtures were prepared guarantees that they comply with ASTM C305-12 [17], with minor modifications. The components were firstly weighted using a sensitive digital balance (0.01). Sand and cement were manually blended to get the desired ratio. The mixture is then placed in an electric mixer along with water and stirred for approximately three minutes. The DPET-contained mixture is prepared by adding DPET into cement and mixed manually for about 5 minutes. This mixture is then added to the sand, and the entire mixture is stirred for two minutes in an electric mixer. The mixture is allowed to settle for 90 seconds before being stirred again for 90 seconds. After removing the mortar from the mixer, it is put into clean, lubricated moulds. Two layers of samples are intensified for ten seconds, each using a vibrator[18]. The surface of the samples is handled and finished with a spatula. Following moulding, the moulds are coated with plastic sheets to retain moisture, and the specimens are stored at room temperature in the laboratory for one day. Finally, the specimens were unmoulded and placed in water to cure. Curing of specimens continues for seven and twenty-eight days.

3.2. The testing of specimens

The water absorption measurement is done based on ASTM C642-06 [19]. At first, the samples were taken out from the curing path after the setting time. The samples are placed in an electric oven between 100 and 110°C for one day to eliminate any moisture. Then, each sample is weighed. Following that, the samples are dipped again in water for 24 hours, then dried using a piece of cloth to ensure that all of the samples' surfaces are fully dry. Each sample is weighed to determine the difference in weight before and after soaking in water. Any weight change observed reflects water absorption.

Three 160x40x40 mm samples were taken from each group are undergone flexural strength test based on ASTM C293-03[20]. A Universal Mechanical test machine (TINIUS OISEN H100KU) was utilized, as seen in Figure 6. The test was carried out to three samples for each group, and then the average value was calculated.

For microstructure analysis, a scanning electron microscopic (ZEISS) device was used for the samples (tested after 28 days). The samples have been coated with gold.
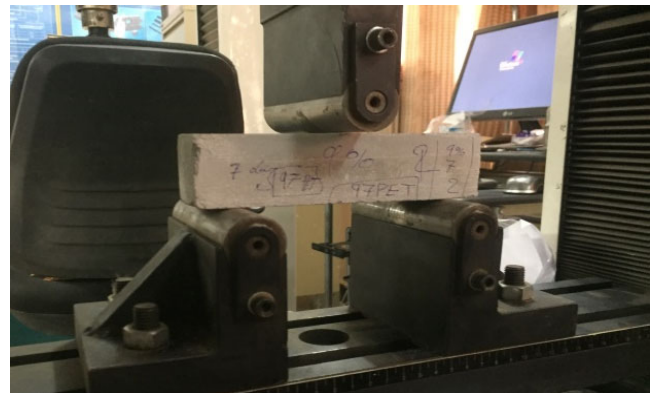

Fig. 6. The flexural strength test

Table 3.
Mixture details

Groups	Sand, g	DPET, g	OPC, g	Water, mL	(water/OPC) ratio	(sand/OPC) ratio	DPET, %	Sand, %
1. Mix.	550	0	200	96	0.48	2.75:1	0	100
2. Mix.	544.5	5.5	200	96	-	-	1	99
3. Mix.	533.5	16.5	200	96	-	-	3	97
4. Mix.	517	33	200	96	-	-	6	93
5. Mix.	500.5	49.5	200	96	-	-	9	91

Note: Each group's material amount is used to cast three samples of the flexural test.

4. Results and discussion

4.1. Flexural tests

Figure 7 depicts the results of flexural tests. It is revealed that there are two modifications in the flexural strength's behaviour. The first one reveals that when 1 per cent DPET is added after 7 days of curing, flexural strength improves marginally but falls when DPET is raised. After 28 days, the second behaviour implies that flexural strength declines as DPET increases.

This tendency has a greater effect when DPET grows. This is because sand particles have a coarse exterior surface, whereas DPET particles have a fine outward surface. In other words, unlike sand particles, the smooth nature of DPET particles results in a limited interlocking capacity. As a result, flexural strength decreases. This conclusion is consistent with Benosman et al.[7], but not with Hasan et al. [8] or Ramadevi & R. Manju [6].

4.2. The water absorption measurement

The results of the water absorption measurement for mortar are reported in Table 4. The results indicate that when DPET increases, the water absorption of mortar reduces for both the 7 and 28 day curing processes.

Table 4.
Effect of DEPT content on water absorption measurement

Group symbols	water absorption, %	
	For 7 days	For 28 days
Mix 1	2.914	2.382
Mix 2	2.877	2.380
Mix 3	2.870	2.368
Mix 4	2.651	2.118
Mix 5	2.492	2.034

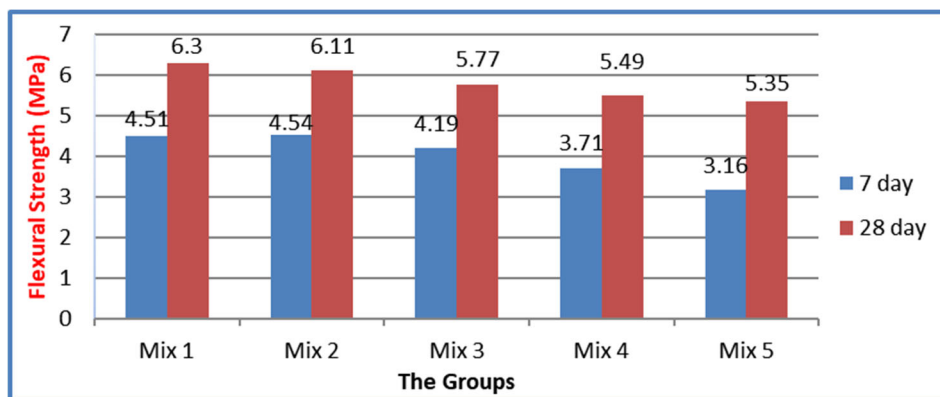Fig. 7. Effect of DEPT content on the flexural test results of the mortar

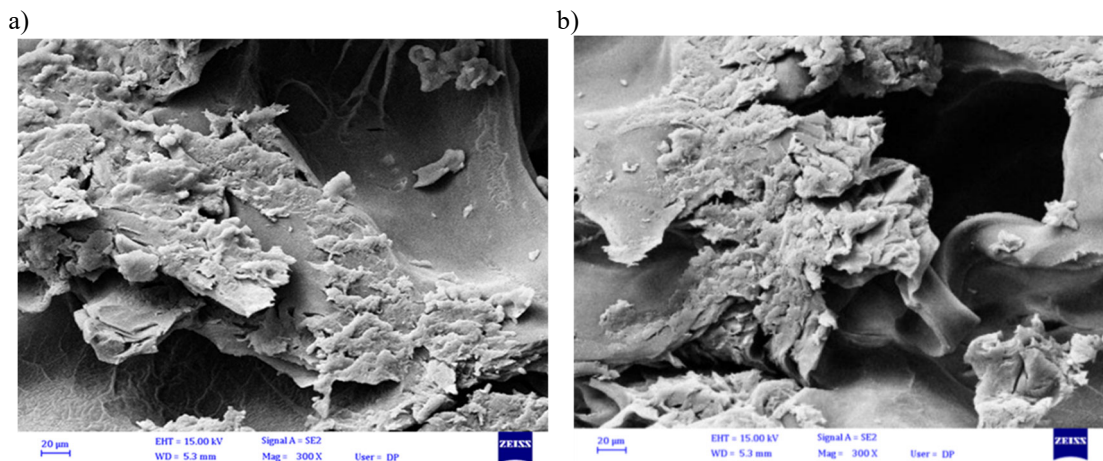a)   b)

Fig. 8. SEM photo for a) Mix 3, b) Mix 1

Water absorption is reduced due to the DPET particles acting as a filler, reducing the size of existing voids in the mortar. This impact appears to be minimal when DPET is between 1% and 3% but is significant at other percentages, according to research conducted by Frigione [21].

4.3. Microstructure analysis

The SEM images of MIX1 and MIX3 demonstrate that MIX1 has a more porous microstructure than MIX3. This is due to the fact that MIX3 contains DPET. DPET particles assist in cavity reduction. The microstructures of the two mortar samples appear to be comparable in terms of homogeneity but differ in the presence of cavities, as seen in Figure 8. The SEM image of Mix 3 demonstrates that the cavities have been reduced in size owing to the presence of DPET, but the image of Mix 1 demonstrates the presence of cavities due to the lack of DPET. The SEM images verify the results of the water absorption test. Due to the smooth surface and tiny size of DPET particles, the particles are dispersed effectively inside the mortar, whereas sand particles lack these characteristics. As a result, the ideal distribution of DPET particles acts as a filler, preventing cavities from forming.

5. Conclusions

According to the findings of the SEM, water absorption measurement, and flexural strength test, it was discovered that when sand is replaced with DPET, the mortar characteristics are altered. The cavities in microstructure were decreased in number and size because DPET acts as a good filler and cohesion element. This is due to the properties featured in the DPET particles: smooth surface and small size. The flexural strength of the OPC mortar was decreased when the DPET part was increased. While other research, such as Hasan et al.'s [8], state that PET waste was utilized as fibre. These experiments indicate that when the PET fibre content rises, the flexural strength increases as well. When the sand was replaced with DPET, the mortar's water absorption was decreased. As DPET increases, the water absorbency of mortar decreases. This is due to the DPET particles' propensity to fill cavities.

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