Studying some of mechanical properties and microstructure analysis for cement mortar using waste of depolymerized Polyethylene terephthalate by using bubble column technique

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Abstract. In this study, the density and compressive strength together with microstructural analysis of cement mortar by partial replacement of fine aggregate with the powder of Polyethylene terephthalate PET depolymerized by using bubble column technique has been studied. The powder of depolymerized Polyethylene terephthalate DPET was used with four different contents of 1%, 3%, 6%, and 9% by weight of fine aggregate. The destructive compressive strength test was prepared once it reaches ages 7 and 28 days of curing. Before that, the density of mortar determines at some samples for both ages. The lightweight modification mortar was set with ratios of 0.48 for water to binder and 1:2.75 for the cement to sand. After completing specific tests, the data of the result presented that the density of the modification cement mortars drops when using DPET powder as fine aggregate. The compressive strength of the modification cement mortars with DPET powder was slightly decreased with the increase of DPET powder percent. The microstructure of the modification cement mortar containing DPET powder showed that DPET particles plug the holes.

1. Introduction
Due to rapid industrialization and urbanization, the amount of plastic waste generation is increasing day by day. However, due to vast manpower, very small amounts of plastic waste are recycled and used, and the remaining waste is going into landfills which causes serious environmental problems. If the production of waste cannot be prevented, it is necessary to create an alternative use in waste disposal [1].

New categories of engineering discovered by the researchers, such as green engineering and sustainable engineering, these kinds aimed to save energies and natural resources from consumption. The main aim of green engineering is optimum use of natural resources by achieving a balance between the lowest consumption and largest benefits for the environment, society and, the economy. It is dealing with the idea of diminishing the contamination generated via rising the process efficiency, toward being as eco-efficiency. When the wastes return in industrial processes, this means the system will change to a close circle instead of the leaner system [2].

Lately, plastic waste occupies great space in the solid waste of the municipal and adds to that increase the wastes every day led to this problem to become a most essential research matter to working on the option of removing the wastes by using it in the many application of the concrete such as the pavements, lightweight concrete and, self-compacting concrete. Also, others use the wastes such as an aggregate of the concrete, inorganic filling material and a component of a composite construction material [3][4].

Choi et al. (2005) [5], and Batayench et al (2007) [6], were studied the influence of using wastes from polyethylene terephthalate bottles as an aggregate on the concrete properties. The result has shown that the wastes reduced both the weight of the concrete and the compressive strength.

Mariaenrica Frigione et al. (2010) [7], conducted experiments to compare compressive and flexural strength with ductility performance of plain concrete and Waste Polyethylene terephthalate WPET concrete. The waste polyethylene terephthalate aggregates are manufactured from recycled PET bottles and substituted in the concrete of 5% by weight of fine aggregate. Specimens were cast with
different ratios of cement to water ratio. From the results, notice a decrease in workability and ductility of plain concrete when compared to WPET concrete.

Baboo et al (2012), [8], have investigated the effect of using the waste plastic flakes as a partially replacement of sand on the workability and compressive strength of the concrete. The results showed that the workability and compressive strength of the concrete was reduced.

Suganthy et al. (2013) [9] have investigated pulverized finely crushed plastic (HDPE) as fine sand. Replacement of natural sand in concrete was replaced by pulverized plastic sand with 0%, 25%, 50%, 75%, and 100%, respectively. With increase replacement of sand increase in water cement ratio and to achieve desired 90mm concrete slump. The results observed a gradual decrease in the concrete strength of 25% replacement of plastic and the rapid decrease in strength of the concrete with an increase in the replacement of fine plastic aggregate.

The current work, a try applied to verify some points; first, using waste plastic such as PET, it is promising to obtain mortar has less weight than ordinary mortar opposite small decrease in mechanical properties, second, decrease environmental pollution and prevent waste of resources. Third, the use of the bubble column technique in since gas phase (air bubbles) interact to liquid and solid phase have some benefits such as easy construction so low operating cost, no need to stirring parts and mixing, good properties of heat and mass transfer, very good thermal stability, low power requirements.

2. Using Materials

2.1. Binder Cement
As a binder, Ordinary Portland Cement (OPC) that most available in Iraq’s local markets which is commercially known (TASLUJA) used in this study. The physical properties and chemical composition of TASLUJA OPC are shown in Table 1.

| Chemical Composition | % by weight | Spec. Limit | Physical Properties | Test result | Spec. Limit |
|----------------------|-------------|-------------|---------------------|-------------|-------------|
| SiO₂                 | 19.6        | -           | Fineness (cm²/g) by Blaine method | 2733        | >2300       |
| Fe₂O₃                | 3.7         | -           | The cement strength / cube (70.7) mm at, MPa | 18.11       | >15         |
| CaO                  | 61.35       | -           | 3 D                  | 26.14       | >23         |
| MgO                  | 2.24        | <5.00       | 7 D                  |             |             |
| SO₃                  | 1.36        | <2.80       |                      |             |             |
| Al₂O₃                | 4.8         | -           | Soundness using Auto clave% | 0.24        | <0.8        |
| Loss on ignition (L.O.I.) | 1.45 | <4.00   |                      |             |             |
| Time saturation factor (T.S.F.) | 0.89 | 0.66 – 1.02 | Time of Setting (Vicat’s method) | 147         | >45 min     |
| Insoluble residue (I.R.) | 0.97 | <1.50   | Initial setting(min) | 276         | < 10 hrs.   |

2.2. Fine Aggregate
The wide sand use, called alikhaidher came from Karbala city in the west of Iraq, which was used in this research.

This type of sand has properties of sulfate 0.08% by sand weight, the bulk specific gravity of 2.56, and the fineness modulus of 2.62 and particles size 4.7mm.

2.3. Waste PET aggregate
Preparation of PET powder done in some stages:
I. Minimis scale of waste PET bottle: Begin by collect waste PET bottles and then good cleaned, air dried and cut manually into small and more small pieces with diameter about (2-4) mm[11].

II. De-polymerization process of PET pieces: The chemical degradation of PET was done by using Ethylene Glycol (E.G.) as solvent and Nano-MgO with particles size 60nm as a catalyst in bubble column reactor has special design. An electrical air pump was used to offer a gas phase in which the air is making bubble inside the reactor, and that offered good mixing for the contents by increased the interfacial area and the heat mass transfer rate see Figure (1) [12]. The chemical process is done at the boiling point of EG 197°C, and the mixture of PET (solid phase) and EG (liquid phase) becomes fully depolymerized as one liquid phase after about 50 min. and to ensure total condensed as a close system, vertical condensers water cooling was used in the process.

![Bubble Column Reactor](image)

**Figure 1: The bubble column reactor.**

III. Physical separation process: To separating EG from PET solution, used horizontal condenser water cooling. By heating to EG melting point 197°C, the final product drops in flat plan to cooling by air and after few hours the PET grinding by electrical milling machine [11].

IV. Washing and purification process: For a cleansing product from remaining ethylene glycol, added about distilled water to the powder form of product in the ratio (2gm PET/10ml distilled water) with heating and mixing the mixture by magnetic stirrer hot plate, thereby dissolve the EG. And then, the mixture was filtered. The final white product was depolymerized Polyethylene terephthalate DPE formed in the filtrate, then separated and left to dry and milling see Figure (2).
3. Design the Mixture

Two series of mixtures were set in the test center. One of them was without using DPET, and the other was with four different percentages of DPET (1%, 3%, 6% and 9%) by weight of sand. The ratio of the blend of sand to cement is set at (2.75:1). The ratio of the blend of water to cement binder ratio was set at (0.48). The details of the mixture’s design for the two series are shown in Table 2.

| groups | Sand% | DPET % | Cement % | (water/cement) ratio | (sand/cement) ratio |
|--------|-------|--------|----------|----------------------|---------------------|
| Mix.1  | 100   | 0      | 100      | 0.48                 | 2.75:1              |
| Mix. 2 | 99    | 1      | 100      | 0.48                 | 2.75:1              |
| Mix. 3 | 97    | 3      | 100      | 0.48                 | 2.75:1              |
| Mix. 4 | 94    | 6      | 100      | 0.48                 | 2.75:1              |
| Mix. 5 | 91    | 9      | 100      | 0.48                 | 2.75:1              |

4. Procedure of work

4.1. Samplings planning

Samplings planning were accomplished in a way similar to ASTM C 305-12 [13] and add few variants to adapt with the current work:

1. Find the constituent’s weight.
2. First series named Mix. 1 constituent of sand and cement mixed alone up to look homogeneous and then add the water to the mixer device for 2.5-3 min.
3. In other series that mixtures contained DPET in add to sand and cement, DPET added to cement and mixed by hand for 5 minutes.
4. The mixture of cement and DPET, which previously mixed, added to sand accommodated within the mixer together for two minutes, and added 95% of the water with mixing for 60 sec. after that, the residual continent of water adds.
5. Poured the cement paste into lubricated casts in two stages on a vibrating table, which takes 10sec vibrating for each layer [14].
6. Using a spatula, the top face of the samples balances and then is covered to prevent moisture loss.
7. The final step was kept the samples for 24±1 hour in molds at a temperature about 23±1 °C, and then take out from molds into curing pool of water for 7 and 28 days.

4.2. The testing of specimens
At first, were outing the specimens after reaching the testing ages. The calculate of mortar density was recorded according to ASTM C567-14a [15], samples dry by oven at 110 °C until completely exit all moisture. The compressive strength exam of samples done in the laboratory at 23±1 °C temperature and was accomplished agreeing to ASTM C109-02 [16]. The compressive strength for all series was calculated from a middling of three cubic samples and tested after 7 and 28 days of curing in water, see Figure 3. Sampling pieces of broken mortar was done after the compressive strength test for SEM. This test was made by means of VEGA3/Tescan SEM device. Before using the broken sample pieces in SEM, they must pass through the coater device and be coated with gold.

![Figure 3: Testing of specimens](image)

5. Results and Discussion

5.1 compression tests
All the values are the average of the three trials in each case in the testing program of this study. The results of compression tests are shown in Figure (4). Comparison of compression strength obtained in the laboratory tests indicates that there is very little variation in strength with an increase in the percentage of DPET for this study which the maximum replacement was 9% of the sand weight. This result is consistent with reference [6][8][9].

![Figure 4: Results of compression tests](image)
The reduction in compression strength was continue with an increase in DPET replacement. This behavior finds in both 7 and 28 days. The little reduction in compression strength with increase in DPET replacement by weight of sand because the last one tough than DPET. From other said, the small size of DPET particles than the sand particles helped in filling microporous of mortar or minimize the volume of larger pores, and that decrease the effect of the increase in DPET replacement by weight of sand.

5.2 Density
The densities of all mixtures were (Mix 1: 2208, Mix 2: 2155, Mix 3: 2011, Mix 4: 1941, and Mix 5: 1864) kg/m$^3$ at 28 days. Chart 2 show the results. Figure 5 shows a sharp decrease in density of all mixtures along with an increase in the percentage of DPET replacement by weight of sand. That matches the fact that the DPET has less density than sand, so, because of this difference in the density between DPET and sand, the density of mortar decreases along with an increase in the percentage of replacement. Furthermore, this result is consistent with reference [6].

From the results of compression test and density measurement, the strength to weight ratio was calculated, since was (0.0150 at Mix. 1) and increase with the increase in the percentage of DPET replacement by weight of sand up to 3% (0.0153 at Mix. 3) as the maximum value and then dropped to (0.0141 at Mix. 5) as the minimum value.

![Figure 5: Densities of the mixtures](image)

5.3. Microstructure analysis
From comparison, the microstructure of Mix 1, which was without DPET, and Mix 3, which was with 3% DPET (Mix 3, which have a higher strength to weight ratio), by SEM photo found that: the microstructure of cement mortar with DPET replacement was more homogeneous than that without DPET, that because the DPET particles have small particles size than sand, causing good filling of the microporous and also minimize the volume of larger pores and this led to reduces the large crystals of Ca(OH)$_2$ that matches with the result of a compression test. Although DPET intervened between the largest crystals and filled the cavities, the density of mortar drops because the DPET has less density than sand when replace the same amount in mortar see Figure 6.
6. Conclusion
Concerning the experimental results of compressive strength and density, it is funded that replacing depolymerized waste water bottles DPET by weight of sand effect on the mortar and cause:

1. The compressive strength of mortar decreasing when replaces sand by DPET. and this decrease increases with an increase in the percentage of replacement.
2. The density of mortar decreasing when replaces sand by DPET. And this decrease increase with the increase in the percentage of replacement.
3. The DPET fills the microporous and minimizes the volume of pores in the microstructure.
4. The mixture with 3% replacement (Mix. 3) represents acceptable balance pointe between drawbacks of reducing the strength and advantages of drop-in density, in other words, a higher strength to weight ratio.

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