

Study the Effect of Preparation and Diagnosis of Polyethylene Terephthalate (PET) As Additive Modification on Concrete Properties

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Abstract- The polyethylene terephthalate (PET) was prepared from PET flakes of empty local water drinking bottles and diagnosis by infrared spectroscopy (IR). The glass transition temperature, melting temperature T_m and heat capacity were indicated by differential scanning calorimeter (DSC). Part hundred ratios (phr's) of (PET) were added to study its effect on concrete properties. Additives of (phr) impart performance such as increased cement dispersion, enabling drastically reduced water requirements. Enhance the mortar cubes performance; depend on the compatibility of cement with PET. Porosity of mortar cubes with cement, sand and added PET products were calculated. Maximum and minimum Compressive strength were calculated 58.76 MPa, and 24.0 MPa at phr 0.04 and 0.07 of PET with cement. And PET with cement and sand were calculated 16.5 MPa and 4 MPa at dosages 0.03 gm. and 0.08 gm. The relative dosages in calculating porosity percentages are (0.06, 0.1 and 0.3) gm.

Keywords: PET flakes of empty local water drinking bottles, prepared PET, IR, DSC, functional groups, adhesives, concrete, bond, compressive strength, Prosimy.

I. Introduction:

Admixture is a material other than water, hydraulic cementitious material or fiber reinforcement that is used as an ingredient of a cementitious mixture to modify its freshly mixed. It is used to improve concrete's plastic (wet) and hardening properties. Reducing the water-cement (w/c) ratio of concrete has identified as the most important factor to making durable and high quality concrete. On the other hand, some times the cement content may be lowered while maintaining the original w/c ratio to reduce costs or the heat of hydration for mass concrete pour [1].

Polyethylene terephthalate (PET) is, semi-crystalline thermoplastic polyester. This polymer can be transparent when in its amorphous state; or translucent when crystalline state with excellent barrier (CO_2 permeation). Due to the hydrophilic nature of PET, compounds remain its favorite coloration route color is used in very large in entities for applications such as PET beverage bottles [2]. Its molecular weight $(2-3) \times 10^4$ heavy number average molecular weight and the ratio 1.5-1.8. The relative density 1.386, 225 °C temperature, flow temperature 243 °C, glass transition temperature 80 °C, 80 °C heat Martin, thermal deformation temperature 98 °C (1.82MPa) [3]. Amorphous plastics are transparent and brittle, or glasslike, because of restricted molecular motion. This motion of polymer backbone in the glassy state is limited to band distortions at molecular vibrations in contrast, a wiggling type segmental movement occurs in the polymer chain of elastomers [4]. In the glassy state, large scale molecular motion does not take place. Rather, atoms and small groups of atoms move against the local restraints of secondary bond forces, much as atoms vibrate around their equilibrium position in a crystal lattice,

except that the glassy state does not have the regularity of the crystalline state. The glass transition corresponds to the onset of liquid like motion of much longer segments of molecules, characteristic of the rubbery state. This motion requires more free volume than the short-range excursion of atom in the glassy state [5]. The characteristics of polymer by infrared spectroscopy represent one specific application, although at large and important one, of the group frequency concept. To a first approximation the spectrum of single units in homopolymer is identical with that of along sequence of such units and where differences do occur they are capable of providing valuable information on microstructure, such as a stereoregularity and on the way in which the chains pack spatially with copolymers the spectrum may not be the precise sum of the component bands, may provide useful information such as the diagnosis of random or block copolymers. The success of infrared spectroscopy in the characterization of the organic compounds is the result of the almost general validity and applicability of the concept of group frequencies. In comparatively complex molecule individual functional groups such as carbonyl, hydroxyl, amine and olefin are vibrationally independent of the rest of the molecule and give readily recognizable characteristic vibration frequencies [6]. The most common strength test, compressive strength, is carried out on 2-in. or 50 mm. cement mortar test specimen. This test method provides a mean determines of determining the compressive strength of Hydraulic cement and other mortars and result may be used to determine compliance with specifications. Further, this test method is referenced by numerous other specifications and test methods. Caution must be exercised in using results of this test method to predict the strength concrete [7]. The investigation of various properties related to the durability and long term performance of mortars made Fly Ash blended cement, FA and Ordinary Portland cement, OPC. The properties that have investigated in an experimental program include; determination of total porosity and compressive strength. This is depending on the different mixes made of mortar samples and the curing age has taken 28 days [8].

The aim of this investigation was to develop of mix design for concrete that can be used for estimated the required compressive strength with required PET and water-cement ratio. This use of prepared PET is to enhance concrete performance, in which mortars and concrete gives an adequate performance at measured water-cement ratio, leading to both with greater strength.

II. Experimental work:

PET flakes of local empty water drinking bottles were cut by scissors. 25g of PET flakes was weight and emplaced in Tri-neck round flask 500 ml in capacity, this is fixed by stand on a heater and 25% NaOH was dropped, Klaus Englert (EMC LAP Germany) solution PH14, on the amount of the flakes. A stirrer HeDolf made in Germany was inserted into the vertical neck B24 (mm diameter). A thermometer in the side neck B19 (mm. diameter) and the condenser at the other side neck B19 (mm. diameter), which was connected to a path of cold water with pump gw 220 v 50 HZ and 0.6A, to ensure efficient condensation, while the system was run at 100-130°C of the heater (The Numeral controls Electric Heated). And reflex for six hours, until a complete reaction of PET flakes with NaOH was occurred and precipitate at the round flask bottom. As shown in Fig. 1.

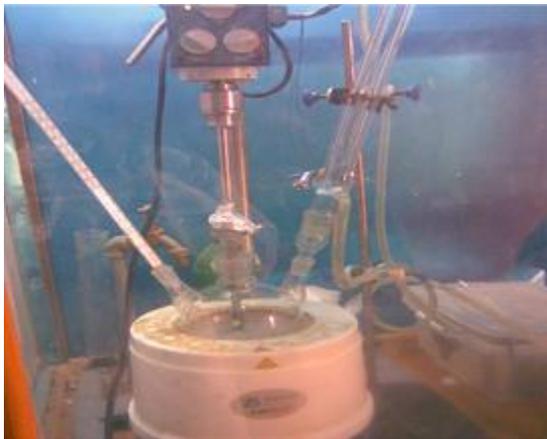


Fig. 1 The setup of PET preparations

The precipitate was filtered at 100°C by using Whitman filter paper chart 15.0 cm. made in England by W&R BALSTON LIMITED, and the precipitate was left to be dried overnight, while the solution was collected into flask. The precipitate was etched and was put into Petri dish is a white powder was emplaced again into the flask round; this was reacted with 25 mil. of ethylene glycol (Gailand Chemical Company U.K.) and reflexes for 6 hours[3] and filtered at 100°C., and Left the precipitate to be dried overnight, the product is a white oil-powder soluble substance of PET and has PH9 by the detection with indicator paper Mecherey-Nagel, Germany, while the solution was collected into flask, and the product of PET was etched into Petri dish, cured at 60°C by using vacuum electric furnace and collected into plastic container. The product has PH7 overlook the amount was dissolved in water.

The standard w/c was measured by vicat instrument as shown in Fig. 2, was adjusted and filled the cylinder of the instrument with cement paste and the pour 10 mm³ was dropped on the cement paste, the penetration was measured and repeated for different (w/c) ratios, as shown in Table 1. Graph of w/c versus penetration was sketched; to determine the standard w/c. A graph of penetration versus setting time for pure cement at the standard w/c was sketched.



Fig. 2 Vicat instrument

Preparation of mortar cubes have been done for compressive strength test, the set of mortar cubes were used the specimens were cast in moulds; this was made of brass mould 2-in. or [50-mm]. The molds comprise of cement, water-cement ratio (w/c =0.28) and phr of PET was prepared as follows:

1- PET product (0.05, 0.1, 0.15, 0.18, 0.22, 0.3 and 0.33) gm was weighted. Each weight was dissolved in water in a separate flask and each dissolved weight was emplaced in a bowel. The cement was added to the mixture and mixing with gloved hand was made within 2 minutes. Molding was started after completion of the origin mixing of the mortar. The mixture was emplaced into two successive layers of the mold and mortar was tamped (ASTM C109).

Molded specimens were placed in a moist room for 24 hours. Keep specimen in their mold for this initial period. After 24 hours specimens were removed from molds. The weight was measured and the dimensions were measured 2-in or [50- mm]. The specimens were immersed in saturated water curing tank, were cured for (28) days. The weight of each cube was determined by carefully weighing and the dimensions were measured, these are the same dimensions of the mould specimens 2-in. or [50-mm]. SERVO TRONIC machine. U.K. and MARUTO testing machine, Japan. Was used for measuring the compressive strength. Using the formula[9]:

$$\sigma = \frac{F}{A} \dots\dots\dots(1)$$

Where F is the forced was applied by the machine on the specimen and A is the area of the specimen.

Sets of mortar cubes using one part of cement to 2.75 part of sieving grad standard sand (2.36 mm; 600 μm and 212 μm) by weight, and (w/c = 0.485) was used for all Portland cements, [Compressive Strength of Hydraulic Cement Mortars, ASTM C109], were prepared as follows:

- 1- All the mixing water was placed in a bowl.
- 2- PET product (0.03, 0.05, 0.06, 0.08, 0.1, 0.15, 0.2, 0.3 and 0.33) gm. each weight for one mortar cube, was added until they dissolve.
- 3-The cement was add to the mixture and mixing was started by hand using slab of wood to the speed 140 rpm for 30 sec. add the sand over 30 sec period.
- 4- Mixing was Stopped and the sieved grad sand (2.36 mm, 600 μm and 212 μm) was added to the mixture and mixing by hand was continued with changing to speed (285) rpm for additional 30 sec. The mixing was stopped and the mortar was left to stand for 1.5 minutes. During the first 15 sec. quickly scrap down into the batch any mortar that may

have collected on the side of the bowl, then for remainder of the interval, cover with led.

Molding within 2 minutes and 30 sec. was started after completion of the origin mixing of the mortar.

a- A layer of the mortar about $\frac{1}{2}$ of the depth was emplaced of the mold in all of the cube compartments.

b- The mortar was tamped in each cube compartment 32 times in about 10 sec. in four rounds, each round at right angles to the other and consisting of eight adjoining strokes over the surface of the specimen as shown in Fig. 3.

c- The compartments were filled with the remaining mortar and tamp as specified for the first layer. During tamping of the second layer, in the mortar was bring, was forced out into the tops of the mold after each round of tamping using gloved finger and the tamper. On completion of the tamping, the tops of all cubes should be extended slightly above the top of the mold.

d- Trowel mortar of each cube laterally and longitudinally was made. Cut off mortar to a plane surface with the top of the mold by drawing the straight edge of the trowel, held perpendicular to the molds with sawing motion over the length of the mold.

e- Molded specimens were emplaced in a moist room for 24 hours. Keep specimen in their mold for this initial period. After 24 hours remove specimen from molds. Determine the weight of each cube by carefully weighing and measure the dimensions, these are the same dimensions of the mould specimen. And immerse in saturated water curing tank, were cured for (28) days. The specimens were removed and weighed. The dimensions of the specimens' were measured 2-in. or [50-mm]. MARUTO testing machine, Japan, was used for measuring the compressive strength as will be discussed.

Other sets of mortar cubes using one part of cement to 2.75 part of sieving grad standard sand by weight (2.36 mm, 600 μm . and 212 μm .) to calculate porosity percentage. The porosity was calculated by using the formula [10]:

$$P = \left[\frac{W_{SA} - W_d}{W_{SA} - W_{SW}} \right] \times 100. \quad (2)$$

where: W_{SA} : Weight of specimen in air after immersed in water.

W_{SW} : weight of specimen saturated submerged in water.

W_d : Weight of specimen after oven driers at 85 °C. for 24 hours to constant weight.

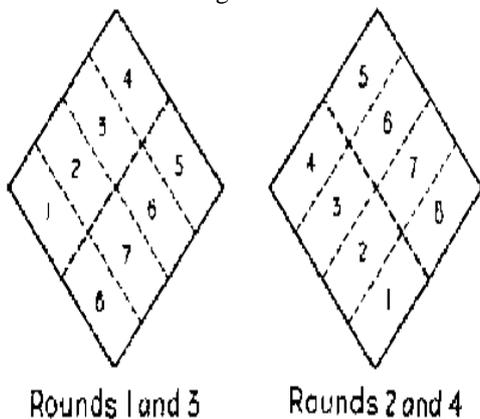


Fig. 3 Order of tamping in molding of test specimens.

III. Results and discussion:

PET flakes of empty local water drinking bottles and the prepared PET of the first reaction with NaOH only, were examined by IR spectrophotometer; Buck scientific, Model 500, made in U.K. The prepared PET in reaction with NaOH only, was examined with KBr. The PET flakes of empty local water drinking bottles give an explanation that most of the ester groups encountered in polymers give rise to characteristics bands at 1150-1200 cm^{-1} . as in Fig. 3. This shows also a typical example, with particularly strong peak at 1050-1060 cm^{-1} these are a specific for terephthalate group. Coupled with band at 730 cm^{-1} this lead to the clear identification of poly(ethylene terephthalate) [6]. The Chemical structure of PET is shown in Fig. 4 [3]. In Fig. 5 the IR spectroscopy of the prepared PET with NaOH only, the functional group (carbonyl group) in this the second of these peaks is the C=O stretching mode of a carbonyl compound and it is precise position at 1680 cm^{-1} suggest a saturated ester.

The success of infrared (IR) spectroscopy in the characterization of organic compounds is the results of the almost general validity and applicability of concept group frequencies, such as carbonyl group. Aromatic absorption occurs at 1600 cm^{-1} . Conformation is also occurs with PET by Infrared spectroscopy both trans and gauche conformation and to the O-H end group of the molecules, a method is proposed that can discriminate otherwise similar the PET fibers. The absorbencies at 1200 and 860 cm^{-1} relative, respectively, to the gauche and trans conformers [6]. The carboxylic acid can be indicated at 1700-1725 cm^{-1} and end group content was evaluated at the absorbencies 3440 and 824 cm^{-1} [11] as shown in Fig. 5. PET was examined with KBr. Conformational isomerism also occurs with poly(ethylene terephthalate) and involves the $-\text{O}-\text{CH}_2-\text{O}-$ unit. There are two isomers, gauche and trans; with former the two oxygen atoms are staggered and with the latter they occur in an extended structure pointing away from each other. Both give several specific bands, of which those at 890 and 1140 cm^{-1} are most characteristics for the gauche conformer and those at 840 and 970 cm^{-1} for its trans counterpart [6].

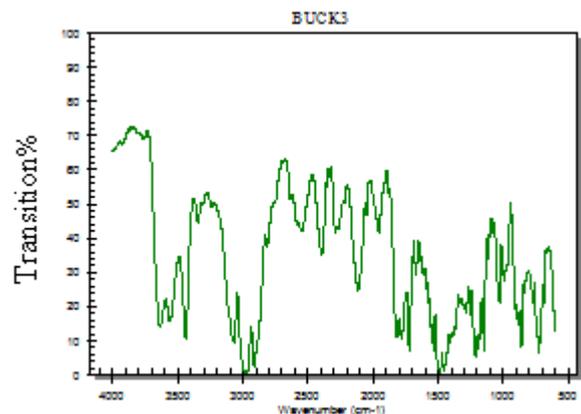


Fig. 4 IR spectroscopy of PET flakes of empty local water drinking bottles.

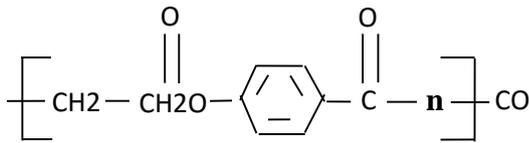


Fig.5 the chemical structure of PET.

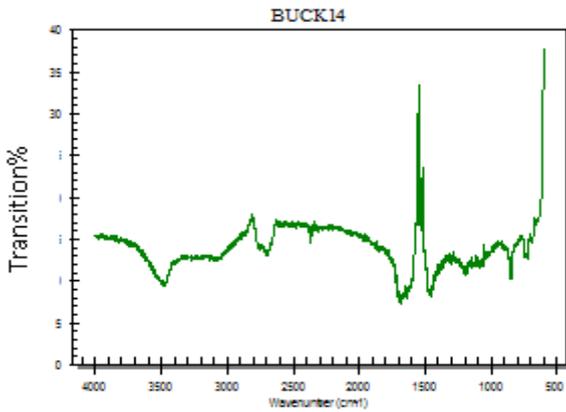


Fig. 6 IR spectroscopy of prepared PET with NaOH.

Differential scanning calorimeter, DSC-60, DELL, SHIMADZU, NO. C30454801026 SA was used to measure the heat absorbed of the sample raised at heat rate 20 °C/min, the results the results are shown in the endothermic process of Fig. 6, for PET flakes of empty local water drinking bottles. The glass transition temperature T_g , on the curve was 116.83°C, the heat flow are 0.66 mW (-0.07mW/mg). The melting temperature T_m 284.45°C, heat capacity is -413.67 mJ (-41.37 J/g). The T_m in the endothermic process of Fig. 7, for prepared PET in the reaction with NaOH only (dry powder) was 116.36 °C, is the same as T_g in Fig. 6, the heat capacity is 1.30 J (130.4KJ) [12]. For glass transition temperatures have indicated in Figs (6) and (7) is the liquid state transition temperature, for short (glass transition) for PET is 70°C, the measured value in this investigation is 116.47 °C, for prepared PET in reaction with NaOH, despite the massive change in the physical properties of material through its glass transition, the transition is not itself a phase transition of any kind; rather it is resbrotary phenomena extended over a range of temperature and defined by one of convention such as convening method a measurement cooling rate (20°K/min.) [13].

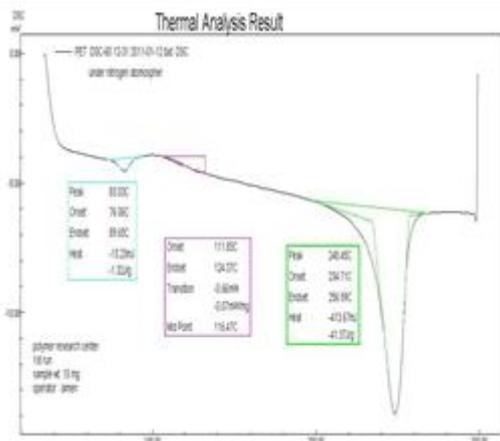


Fig. 7 DSC of PET flakes of empty local water drinking bottles.

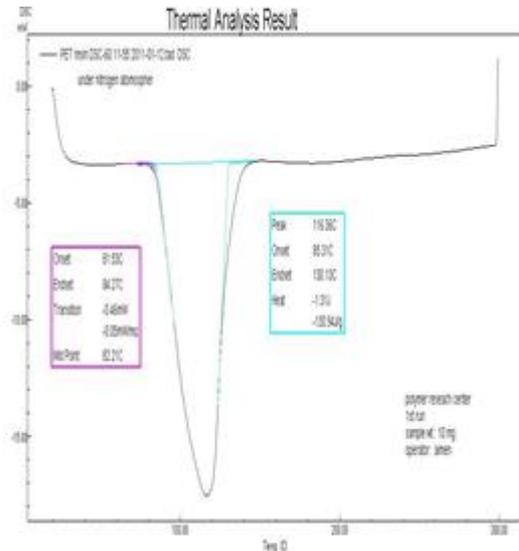


Fig. 8 DSC of PET reaction with NaOH

The penetration was measured and repeated for different w/c ratios, as shown in Table 1. A graph of w/c versus penetration was sketched. To determine the standard w/c. is found equal to 0.28 was measured at (25) mm. as shown in Fig. 9.

Table I The Standard w/c at $t=22\pm 1$ °C.

w/c	penetration m.m.	Weight of water (gm)
0.22	5	80
0.25	11	90
0.26	15	94
0.28	25	101
0.29	35	105
0.30	38	110
0.31	40	112

Table II The setting time against penetration for pure cement at 20 oC.

set.t min	pen. mm.	s.t min.	Pen. mm.	s.t min	Pen. mm.
15	40	50	38	85	37
20	39	55	36	90	35
25	39	60	37	95	36
30	38	65	37	100	34
35	37	70	36	105	36
40	37	75	37	110	35
45	39	80	37	115	36
s.t.mi n	Pen. mm.	s.t. min.	Pen. mm.	s.t min.	Pen. mm.
120	34	155	33	190	16
125	35	160	32	195	14
130	35	165	32	200	26
135	35	170	30	205	12
140	35	175	28	210	8
145	35	180	26	215	4
150	33	185	26	220	4
				225	0

The cement paste was prepare with (w/c) = 0.28 and was enplaced in the cylinder of vicat instrument to measure

setting time. Penetration setting time dependant from Table 2. For pure cement was sketched as in Fig. 10. The cement .

used is from Basrah Cement Plant (The General South State Company of Cement) this is an ordinary Portland cement, Table 3. shows the usual composition limits of Portland cement [14].

Table III Usual composition of limits Portland cement.

Oxide	cement, percent
CaO	60-67
SiO ₂	17-25
Al ₂ O ₃	3-8
Fe ₂ O ₃	0.5-0.6
MgO	0.5-0.4
Alkalis(asNa ₂ O)	0.3-1.2
SO ₃	2.0-3.5

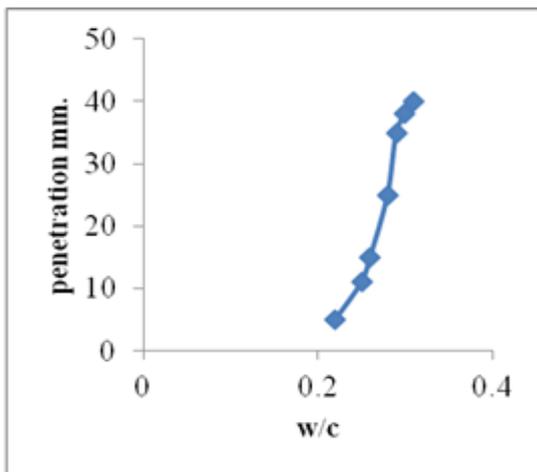


Fig. 9 the w/c. ratios versus penetration

After two hours and the final setting time at 225 min. From Table 4; the compressive strength of mortar cubes was measured at phr's, these factors are accountable to the compatibility between PET and cement are C₃A and C₄AF content in the cement, reactivity of calcium sulfate content C₃A, final from calcium sulfate in ground cement (Hemihydrate, dihydrate, or anhydrite gypsum), these are the fiber position of the end group in the chain, the counter-ion type (sodium or calcium), is the presence of residual sulfate that affects the de-flocculation properties [15]. Table-4-, also indicate a maximum compressive strength = 58.76 MPa. at phr (0.04), minimum compressive strength 24 MPa at phr (0.07). And for pure cement 38.32 MPa. This is example of material with higher compressive strength [16]. On atomic level, the molecules or atoms are forced apart were intention whereas in compression they are forced together. Since atoms in solids always try to find an equilibrium position and distance between other atoms forces arise throughout the entire material oppose both tension and compression the compressive strength is calculated by using the formula(1). Compressive strength is the capacity of material or structure to with stand axially pushing forces, when the limit of compressive strength is reached, Materials are crushed. The compressive strength of mortar is sometimes used as a

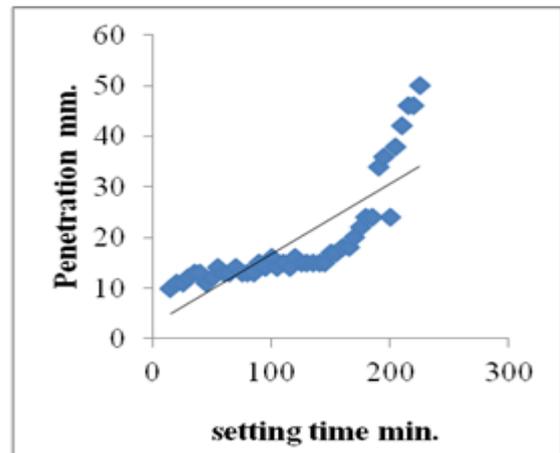


Fig. 10 Penetration setting time dependant of pure cement.

Table IV The compressive strength of mortar cubes. (cement and PET)

Dos. gm.	Age days	W. cube gm.	load Kg.	F KN	Compressive strength. MPa
0.00	28	285	1878.9	18.789	7.516
0.03	28	270	4120	41.2	16.50
0.05	28	270	1710	17.1	6.80
0.06	28	270	4080	40.8	16.30
0.08	28	280	1000	10	4.00
0.1	28	280	3210	32.1	12.84
0.15	28	280	2370	23.7	9.50
0.2	28	290	3040	30.4	12.20
0.3	28	290	2330	23.3	9.30
0.33	28	290	1950	19.5	7.80

principal criterion for selecting mortar type, since compressive strength is relatively easy to measure and commonly relates to some other properties, such as tensile strength and absorption of the mortar, the compressive strength depend largely on cement content and w/c [17]. Fig. 11, shows phr of PET via compressive strength. Although the two sets (three cubes for each set), are located at both side of the straight line. The arrangement was not identical. The results attempted to be a straight line, but the compressive strength results alter points from the straight line and appear as random distribution of particles at both sides. The pure cement with no PET has illustrated. This point was shifted from the straight line.

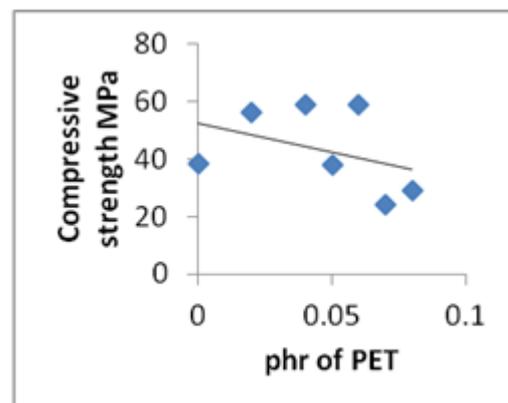


Fig. 11 Phr of PET via Compressive strength.

The predicted and measured compressive strength developed of ASTM C109 mortar cubes, to compute the model compressive strength [18]. Table-5- shows that for mortar cubes, the maximum result is 16.5 MPa at the added weight 0.03 gm. And minimum result is 4 MPa at the added weight 0.08 gm. of PET this is due to the contribution of PET, these factors are accountable to the compatibility between PET and cement are C_3A and C_4AF content in the cement. Low results of compressive strength also indicated in Table-5-.

Table V Compressive strength of mortar Cubes. (Cement, sand and PET).

Phr of PET	age day	W. cube kg.	load kg.	FKN	Compressive strength. MPa
0	28	0.3	9580	95.8	38.32
0.02	28	0.3	14040	140.4	56.16
0.04	28	0.3	14690	146.9	58.76
0.05	28	0.3	9500	95	38.00
0.06	28	0.3	14690	146.9	58.76
0.07	28	0.3	6000	60	24.00
0.08	28	0.3	7300	73	29.20
0.09	28	0.3	12800	128	51.20

This is related to the short effect of the dosage on concrete performance [15]. The most common strength test, compressive strength, is carried out on 2-in or 50 mm. cement mortar test specimen. The test specimen subjected to a compressive load (usually from hydraulic machine) until failure. This loading sequence must take no less than 20 seconds and on more than 80 seconds. The standard cement

Mortar: AASHTO T 106 and ASTM C 109 strength of Hydraulic cement mortar (using 2-in or [50-mm]. cube specimens) [19]. This test method provides a means of determining the compressive strength of Hydraulic cement and other mortars and results may be used to determine compliance with specifications. Further this test method is referenced by numerous other specifications and test methods. Caution must be exercised in using the results of this test to predict the strength of concretes [20].

In Table-6- Calculations of porosity as a percentage at PET dosages in gm. Using formula (2). Shows variations in porosity to limit dosages were indicated. (0.06, 0.1 and 0.3) gm. These are relative dosages for each three cubes. All the samples were cured for 28 days in water tank and oven dried at 85 °C. for 24 hours. The curing conditions of most concretes lie somewhat between these extremes of saturated and sealed curing; improper curing could of course result in water loss due to evaporation and further limit achievable degrees of hydration with concurrent increase in capillary porosity. For saturated curing; it is assumed that an adequate supply of additional water is consistently available to maintain all of the capillary porosity within the cement paste under saturated conditions. This water could be available from an external source or from internal curing for example. The amount of additional water is consistently available to maintain saturated conditions in the paste are conveniently given by the chemical shrinkage [21].

Table VI Calculation of porosity.

Dos. gm.	Age 28 days	WSA gm.	Wsw gm.	Wd gm.	Porosity.
0.00	28	279	140	240	24%
0.03	28	265	120	238	18%
0.06	28	260	115	238	15%
0.07	28	285	150	237	35%
0.09	28	285	148	240	32%
0.1	28	290	143	258	21%
0.15	28	284	135	240	29%
0.2	28	300	205	270	31%
0.30	28	300	200	280	20%
0.33	28	300	190	270	27%

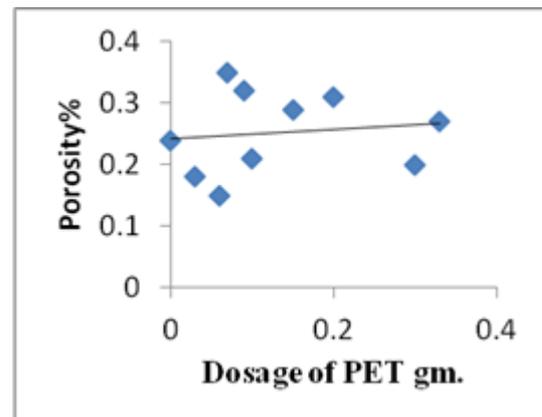


Fig. 12 The Porosity via PET dosages.

Fig. 12 shows PET dosages via the percentages of porosity. Each three cubes are appear to repeat themselves and the straight line pass through make identical distribution for the first set (three cubes) and the second set (three cubes accept for the third set (three cubes) the straight line make them two identical halves. The cube with no PET additives also indicated and the straight line passed through the point.

IV. Conclusion:

In normal cement paste large irregular agglomerates of cement particles predominate. The addition of PET causes them to disperse. Available data suggest that the admixture is absorbed by the cement particles and causes them to repeal each other, resulting in better performance. The main purpose of using the PET in this study is:

- 1- Produce flowing concrete with very high slump.
- 2- To use in heavily reinforced structures and in placements where adequate consideration by vibration cannot be readily achieved.
- 3- The other major application is the production of the high-strength concrete.
- 4- The Tricalcium aluminate C_3A ($3CaO.SiO_2$) present in most cement is comparatively small but its behavior and structural relationship with other phases in cement make it of interest. And its hydrate forms a prismatic dark interstitial material, possibly with other substances in solid solution.
- 5- The Dicalcium Silicate C_2S ($2CaO.SiO_2$) present in (25) percent by weight.

6- The Tricalcium aluminoferrite C_4AF ($4CaO \cdot Al_2O_3 \cdot Fe_2O_3$) is a solid solution ranging from C_2F to C_6A_2F .

7- Gypsum $CaSO_4 \cdot H_2O$ constituents is 3.5 percent by weight. The actual proportions of the various compounds vary considerably from cement to cement, and the fact that different types of cement are obtained by suitable proportioning of the raw materials.

8- In addition to the properties of cement, w/c also affects the rate of gain of strength of concrete. Mixes with low w/c gain strength, expressed as a percentage of long-term strength more rapid than mixes with higher w/c.

9- Preparation assorted PET empty local water drinking bottles, have been cut into flakes and treated into a recovered PET, obviously from Table 4 and 5 PET was used to enhance the performance in mortar cubes and concrete.

10- The porosity percentages were obtained in Table-6- lie in the range (15%-35%). These are prepared with ordinary Portland cement, PET and sand using weight ratios as indicated in ASTM C109 for cement and sand with different percentages by weight of PET.

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