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DESIGN AND FORMULATION OF CONCRETE WITH POLYETHYLENE TEREPHTHALATE (PET) USING STATISTICAL MIXTURE DESIGN

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Abstract: This paper investigated the incorporation of waste Polyethylene Terephthalate (PET) from plastic bottles into concrete as a replacement for natural fine aggregate and found an optimal combination of components that produces a useful concrete product. Six components were considered: cement, water, coarse aggregate, fine aggregate, superplasticizer, and waste PET. A total of 31 mixes including waste PET were prepared based on a statistical mixture design approach. The responses of these mixtures were workability, compressive strength, and splitting tensile strength. The waste PET was first reduced in volume by shredding and then combined with the rest of the components. The responses from the experiments were statistically analysed and a model fitted to each response. Linear models were found to fit the responses best. Using the desirability function approach, four optimal options were selected and then verified in the lab by comparing the experimental with the predicted values. Except for one, all the values fell within the 95% prediction interval. The average response values obtained with the optimal combination were: (1) compressive strength of 23.8 MPa; (2) slump 123 mm, and (3) splitting tensile strength of 3.33 MPa. This mix can be used in basements foundation walls or slabs, inside buildings not exposed to freezing temperatures. It is recommended that future work should consider method of mixing, time of mixing, volume of mix, curing conditions, and other responses to further understand the characteristics of incorporating waste PET into concrete.

Keywords: Polyethylene Terephthalate (PET), concrete aggregate, statistical mixture design.

1 INTRODUCTION

Polyethylene Terephthalate (PET) is one of the main fractions of the plastic waste stream (Silva et al., 2013). PET is mostly employed as a multi-purpose plastic for bottled water, soft drinks, and as single-use packaging material (Andrady 2015). Products made of PET are generally large in volume and can take approximately one thousand years to decompose under natural environmental conditions (Silva, et al. 2013; de Brito and Saikia 2013).

Incorporating waste PET into the concrete industry is an innovative approach that has been seen as promising in recent research (Gu and Ozbakkaloglu 2016; Ge et al. 2014; de Brito and Saikia 2013). It only requires shredding the waste PET into small particles and adding the shredded particles into the natural aggregate mixture (sand, gravel or crushed stone). Using waste PET as a natural aggregate replacement has two important benefits: the waste PET products that occupy an enormous volume in the waste stream can be dramatically reduced by shredding and disposed of; and natural aggregate can be partially replaced, reducing the impact to natural resource availability. Thus, an important reduction in environmental impact of waste disposal while saving natural resources and energy consumption can be achieved (Gu and Ozbakkaloglu 2016; Frigione 2010). Table 2 summarizes the past research on waste PET incorporation into concrete. The main aspects considered in each study were reviewed. The values achieved in the properties and the tests performed in each study were reviewed with special focus on workability, compressive strength, and splitting tensile strength.

One of the goals of this research is to produce a practical and useful concrete mixture containing waste PET. Thus, the performance criterion was oriented to achieve workability and compressive strength requirements for different applications. Only mixes containing waste PET were prepared in order to focus this research on the comparison among different waste PET percentages. According to Beall (2001), the minimal requirements for compressive strength range between 17.5 and 24.5 MPa. Thus, the optimization will be oriented to maximize the compressive strength as much as possible. Additionally, workability was set to range between 25 and 127 mm according to typical values of workability based on (Beall 2001) and (Mehta and Monteiro 2014).

2 EXPERIMENTAL APPROACH

In the present study, the shredder used was built by MUN Technical Services using plans from an international plastic recycling organization called Precious Plastic (https://preciousplastic.com). This organization is devoted to increasing knowledge about plastic recycling worldwide. A complete guide to building a pilot plastic shredder machine is available on the website.

The waste PET was collected from the waste stream at the St. John's recycling centre. The collected waste PET was mostly water and soft drink bottles. Three main types of waste PET were identified based on the volume and thicknesses of the bottles as shown in Table 3. The bottles were unwashed and not separated by color. Only the labels and the lids were manually removed before shredding. Based on past research, the incorporation of waste PET as a fine aggregate produced more advantages than the incorporation of waste PET as coarse aggregate. Thus, the screen of the shredder was selected to generate as fine a particle as possible. The shredded particles were then reprocessed. It is important to note that, shredding waste PET to produce fine particles consumes higher energy and time than shredding waste PET into coarser particles.

2.1 Reduction in volume of waste PET

The volume obtained after shredding the waste PET was measured and compared with the original volume. The bulk density was measured for all types of waste PET according to ASTM standards. Table 2 shows the results for the reduction of volume after shredding the different types of bottles. The reduction of volume of the waste PET bottles was 29 fold or 96.6% for Type 1, 23 fold or 95.6% for Type 2, and 10 fold or 90% for Type 3. This shows that just shredding the bottles alone provide tremendous savings in landfill or storage space. See Figure 1.

2.2 Grading of waste PET

After shredding, sieve analysis was performed to determine the size and grading of the waste PET particles. The waste PET was mixed for all the experiments using the following proportions: 70% Type 1, 20% Type 2, and 10% Type 3. The waste PET gradation was deficient in particle sizes lower than 1 mm and had a high percentage of particles ranging between 4.75 and 9.5 mm. Figure 2 shows the final grading of the aggregates used in all the experiments.

Table 1: Past studies on waste PET incorporation into concrete.

Reference	Recycling method	Gradation / particle shape	Replacement	Admixtures	Workability	Compr. strength 28 days (MPa)	Splitting tensile (MPa)	Observations/ Other properties
Choi et al. (2005)	Mechanical/ Thermal	5 - 15 mm / rounded	25%, 50%, 75% V	Granulated blast furnace	Slump 100 - 205 mm	21.8 - 37.2	1.94 - 3.32 MPa	Modulus of elasticity (15.6 - 25.5 GPa)
Juki et al (2013)	Mechanical	5 mm / flaky	25%, 50%, 75% V	-	-	15.6 - 31.27	Reductions from 15 to 60% compared to a normal blend	Modulus of elasticity (10.4 - 25.9 GPa)
Choi et al. (2009)	Mechanical/ Thermal	5 - 15 mm / rounded	25%, 50%, 75% V	Water reducer	Slump 100 - 222 mm	21 - 35	1.9 - 3.2	Modulus of elasticity (18 - 30 GPa)
Albano et al. (2009)	Mechanical	Fine 0.26 cm / flaky Coarse 1.14 cm / flaky Mix 50% each one / flaky	10%, 20% V	-	Slump 20 - 90 mm	12 - 27	1.4 - 2.8	Modulus of elasticity (12 - 29 GPa)
Frigione (2010)	Mechanical	<2 mm / flaky	5% W	-	Vebe 37 - 62	40 - 69.7	4.1 - 6.3	Shrinkage 1 year (650 - 987 10^{-6})
Ackaozoglu et al. (2010)	Mechanical-washing	0-4mm / flaky	50% V	Granulated blast furnace	-	22.4 - 27	-	Water absorption of concrete with WPET (11.9 - 22%)
Silva et al. (2013)	Mechanical/ Thermal	Fine 4mm / flaky Coarse 2 - 11.2mm / flaky Pellet 1-4 mm	7.5%, 15% V	-	Slump 133 - 141 mm	19.7 - 36.7	-	Carbonation depth (14 -28.8 mm)
Ferreira et al. (2012)	Mechanical/ Thermal	Fine 4mm, flaky Coarse 2 - 11.2mm, flaky Pellet 1- 4 mm	7.5%, 15% V	-	Slump 120 - 140 mm	22 - 38	1.5 - 3.4	Modulus of elasticity (17- 38 GPa)
Ismail and Al-Hashmi (2008)	Mechanical	0.15 - 4.75 mm / flaky	10%, 15%, 20% V	-	Slump 20 - 80 mm	22 - 43	-	Flexural strength (3 - 6 MPa)
Batayneh et al. (2007)	Thermal	0.15 -4.75 mm / flaky	5%, 10%, 20% V	-	Slump 57 - 78 mm	10 - 34	0.6 - 4	Flexural strength (0.6 - 5 MPa)
This study (2018)	Mechanical	0.15 -4.75 mm / flaky	8.4-17%V	Super plasticizer	Slump 51-164 mm	7.4 - 27.8	1.4 - 3.2	Used statistical mixture design.

Table 2: Reduction in volume of waste PET

Type of bottle	Thickness (mm)	Weight of 1 bottle (g)	Initial volume (ml)	Bulk density (g/ml)	Final volume of 1 bottle (ml)	Reduction in volume
1	0.10	6	500	0.34	17	29x (96.6%)
2	0.25	30	2000	0.34	88	23x (95.6%)
3	0.40	20	591	0.33	60	10x (90.0%)

(a) (b) (c)

Figure 1: Initial and final volume of the bottles used in the experiments. (a) Type 1, bottle of 500 ml and 0.1 mm of thickness; (b) Type 2, bottle of 2000 ml and 0.25 mm of thickness and (c) Type 3, bottle 591 ml and 0.40 mm of thickness.

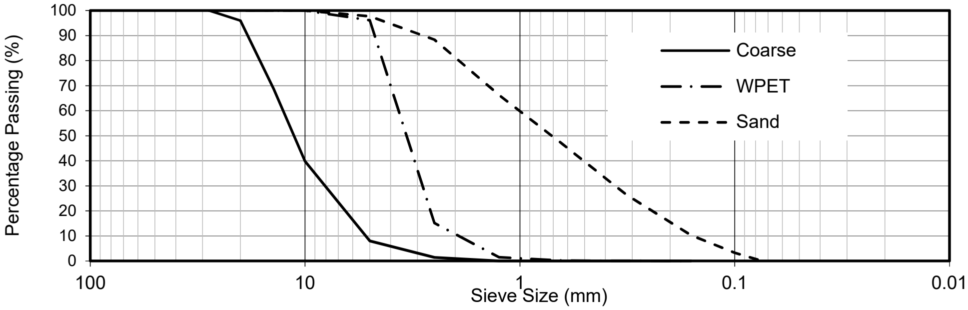

Figure 2: Comparative sieving analysis of sand, coarse aggregates, and waste PET

2.3 Ranges of the components of the mixture

Based on past research the proportions for all the components of the concrete mixture are defined. Table 3 shows the summary of components and their proportions used in the present study. The table shows the proportions in mass for practical calculations and the proportions in volume according to the design requirements.

Table 3: Summary of the proportions of the components in mass and volume.

Component	Mass fraction (kg/m^3) Low	Mass fraction (kg/m^3) High	Volumetric fraction (m^3) Low	Volumetric fraction (m^3) High
Cement (A)	306	365	0.097	0.116
Water (B)	144	177	0.144	0.177
Coarse (C)	1153	1257	0.44	0.48
Fine (D)	506	653	0.193	0.249
Waste PET (E)	24	53	0.018	0.04
Superplasticizer (F)	3	14	0.003	0.014

3 EXPERIMENTAL DESIGN

In mixture design, the variables to be modified are considered ingredients or components and the measured response depends on the proportion of each component (Myers and Montgomery, 2009; Cornell, 2002). When a proportion of a component increases, the proportion of some of the other components should decrease (Myers and Montgomery, 2009). Cornell (2002) also states that a mixture design is considered constrained when the components of a mixture have additional restrictions, such as maximum or minimum limits. The constrained mixture design is appropriate for concrete mix preparations and the responses can be modelled and predicted using a mixture polynomial model which takes the inherent constraint into account (Simon et al. 1997; Kharazi et al. 2013).

3.1 Mixture (Scheffé) polynomials for mixture designs

Mixture polynomial models are also known as Scheffe polynomials after the author who developed them. These models do not have an intercept and quadratic terms from an ordinary polynomial are rolled into the interaction term to create a mixture quadratic term which is a measure of non-linear blending (Cornell 2002). In this study it is assumed that the responses might follow quadratic models, and the mixture polynomial for q components is given by:

$$\hat{y} = \sum_{i=1}^{q} \beta_i x_i + \sum_{i<j}^{q-1} \sum_{j}^{q} \beta_{ij}\, x_i x_j \qquad [1]$$

Where: q is the number of components, and $\hat{y}$ is the predicted response. An optimal IV design was used to obtain the experimental run combinations for the six-component quadratic model. The IV-optimal design seeks to minimize the integral of the prediction variance across the design space which will model the true response surface with greater precision (Myers and Montgomery 2009). Twenty-one (21) experiments would be necessary to determine all the coefficients for a mixture with six components. Additionally, three extra points are necessary to provide information about the error and lack of fit, and three replicated points was included to better understand the behaviour of the data due to repeatability. Finally, four additional center points were added to monitor the process. Hence a total of 31 run combinations were used. Design-Expert V10 from Statease Inc. (2017) is used for both experimental design and subsequent analysis of the results.

4 ANALYSIS OF RESULTS

Table 5 shows the combinations of components on a mass basis and the obtained results for slump, compressive and splitting tensile strengths. The statistical analysis was performed on the data for compressive strength as shown in the Table 4.

Table 4: Analysis of variance for compressive strength

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value prob> F
Linear vs. Mean	473.11	5	94.62	17.52	< 0.0001
Quadratic Vs. linear	112.91	15	7.53	3.40	0.0280
Sp. Cubic vs. Quadratic	5.89	4	1.47	0.55	0.7100
Residual	16.22	6	2.70		

The level of significance selected for this design was 0.05. Table 6 shows that the linear model presented a p-value < 0.0001. Thus, the null hypothesis (H_0) was rejected and the data of the experiments on compressive strength fitted a linear model. The components A (cement), B (water), C (Coarse aggregate), D (Fine aggregate), E (waste PET), and F (superplasticizer) significantly influenced the compressive strength. On the other hand, the quadratic model has p-value of 0.028 that is also lower 0.05. However it will be shown that the linear model is the correct choice. Once the linear model was fitted to the data, lack of fit test showed that there is no statistically significant lack of fit which meant that the residual error did not exceed the pure error. Therefore, the model was adequate to fit the data from the experiments. The adequacy of the model was next evaluated by the adjusted R-squared and predicted R-squared shown in Table 7. The adjusted R-squared represents the variation of the rest of experiments compared with the mean, while the predicted R-squared explains the accuracy on the predictions of the model (Myers and Montgomery, 2009).

Table 5. Mass fraction of the components and results of the experiments.

Run	Cement (Kg/m^3)	Water (Kg/m^3)	Coarse (Kg/m^3)	Fine (Kg/m^3)	WPET (Kg/m^3)	Superp (Kg/m^3)	w/c	a/c	% V WPET	Slump (mm)	Comp. Str (MPa)	Split. Tens (MPa)
1	365	157	1251	544	37	14	0.43	5.0	11.9	128	17.5	3.0
2	320	176	1151	615	51	10	0.55	5.7	13.9	146	15.1	2.2
3	306	153	1231	649	37	4	0.50	6.3	10.2	102	12	1.5
4	364	157	1211	630	29	3	0.43	5.1	8.4	121	27.8	3.2
5	338	165	1217	581	44	8	0.49	5.4	12.9	87	12.9	2.3
6	365	177	1195	573	26	12	0.48	4.9	8.4	154	23.6	3.1
7	365	177	1256	528	29	3	0.48	5.0	9.8	136	21.89	3.2
8	306	166	1203	635	42	3	0.54	6.1	11.7	128	8.8	1.4
9	338	165	1217	581	44	8	0.49	5.4	12.9	116	11.6	1.7
10	334	144	1196	646	48	10	0.43	5.7	12.7	67	19.3	2.7
11	335	175	1256	513	39	14	0.52	5.4	12.9	142	13.5	2.4
12	332	160	1198	624	34	14	0.48	5.6	9.5	140	12.39	2.7
13	306	168	1214	601	38	14	0.55	6.1	10.9	129	7.46	2.1
14	365	177	1155	592	48	3	0.48	4.9	13.7	140	19.05	2.7
15	338	165	1217	581	44	8	0.49	5.4	12.9	115	16.05	2.4
16	306	165	1169	633	48	14	0.54	6.0	12.9	121	10.1	1.5
17	324	177	1193	625	30	3	0.55	5.7	8.4	155	18.8	2.7
18	328	150	1241	622	28	14	0.46	5.8	8.1	164	15.45	2.8
19	335	175	1256	513	39	14	0.52	5.4	12.9	149	11.5	2.2
20	326	158	1198	594	53	14	0.48	5.7	15.0	99	14.4	2.1
21	359	164	1151	619	43	14	0.46	5.1	11.9	121	19.06	2.9
22	365	164	1244	519	53	7	0.45	5.0	16.8	51	16.6	2.9
23	334	144	1256	600	50	3	0.43	5.7	14.2	84	13.5	1.9
24	365	164	1244	519	53	7	0.45	5.0	16.8	94	17.3	2.7
25	365	177	1183	528	53	14	0.48	4.8	16.5	116	16.3	2.6
26	314	173	1256	575	26	8	0.55	5.9	8.3	154	13.5	1.6
27	338	165	1217	581	44	8	0.49	5.	12.9	117	15.6	2.5
28	365	177	1183	528	53	14	0.48	4.8	16.5	97	15.6	2.6
29	306	157	1256	558	53	13	0.51	6.1	15.8	127	10.65	1.1
30	322	177	1234	540	53	3	0.55	5.7	16.3	103	8.03	1.5
31	328	155	1182	645	53	3	0.47	5.7	14.0	84	16.69	2.1

Table 6: Adjusted R-squared, Predicted R-squared, and PRESS of compressive strength.

Source	Std. Dev	R-squared	Adjusted R-squared	Predicted R-squared	PRESS
Linear	2.32	0.778	0.7336	0.6530	211.01
Quadratic	1.49	0.9636	0.8909	-0.6567	995.33

Table 6 shows that the predicted R-squared value was 0.65 for the linear model whereas it was negative for the quadratic model. A negative predicted R-squared means that the model is no better than using the mean value. Hence the choice of the linear model is the correct choice. All the assumptions of ANOVA were also fulfilled. The linear model was established and the coefficients of the components for the linear predictive equation were defined. These coefficients represent the influence of each component on the compressive strength. The predictive equation of the linear model in real values is shown in Equation [2]:

$$[2]\ Compressive\ strength\ (MPa) = +535.96(A) - 72.61\ (B) - 65.16(C) + 39.76\ (D) - 225.38(E) - 176.51\ (F)$$

Analyzing the predictive equation with real values of compressive strength, the components that influence the most the compressive strength can be identified. The cement (A), waste PET (E), and superplasticizer (F) highly influence the compressive strength. A moderate variation in one of these components will significantly impact the compressive strength outcome. The positive sign in cement content (A) indicates that increasing the cement content will increase compressive strength, whereas the negative sign in the remaining components indicates that increasing these components decrease compressive strength. The same analysis was applied for each one of other properties. All the properties fitted linear models. Table 7 shows the equation for each property.

Table 7: Summary of the predictive models.

Property	Predictive model (real values)	R-squared adjusted	R-squared predicted
Compressive strength	$Comp.strength\ (MPa) = +535.96(A) - 72.61\ (B) - 65.16(C) + 39.76\ (D) - 225.38(E) - 176.51\ (F)$	0.73	0.65
Slump	$Slump\ (mm) = -755.04(A) + 1310.60\ (B) - 4.61(C) + 208.15(D) - 2360.66(E) + 1636.75\ (F)$	0.64	0.52
Splitting tensile strength	$Splitting\ tensile\ (MPa) = +72.59\ (A) - 7.52\ (B) - 7.78\ (C) + 1.37\ (D) - 31.06(E) + 12.2\ (F)$	0.79	0.73

5 NUMERICAL OPTIMIZATION

In the numerical optimization, using the desirability function approach, the components or properties can be optimized. The desirability function allows the experimenter to maximize, minimize or keep within target of the goals. The importance of each goal can also be established. By setting a higher importance, the goal is prioritized over the other goals (Anderson and Whitcomb, 2005). Once the goals and the importance are selected, the desirability function analyses all the possible sets of components that achieve the goals, and ranks them from 0 to 1. This is used to rank the options based on their desirability. Finally, the experimenter tests the predicted combination and verifies the results against the prediction intervals (Anderson and Whitcomb, 2005).

On the basis of the previously mentioned goals, the desirability function proposed a set of optimized options. Four options were selected from the list and further tested. Some components such as cement, waste PET, and superplasticizer content produced a strong influence over the compressive strength. Thus, their proportions were very similar in all the optimization options. Components such as water, coarse aggregate, and fine aggregate had slight variations in the optimized options. Table 8 shows the combination of components proposed by the desirability function to be the most convenient to reach the desired properties.

Table 8: Optimization options ranked based on the desirability function.

Number	Cement	Water	Coarse	Fine	PET	Plasticizer	Slump	Comp 28 days	Splitting 28 days	Desirability
1	0.116	0.157	0.461	0.241	0.022	0.003	120.615	24.860	3.352	0.915
2	0.116	0.160	0.459	0.240	0.022	0.003	124.107	24.759	3.346	0.912
3	0.116	0.163	0.456	0.239	0.022	0.003	126.999	24.527	3.324	0.905
4	0.116	0.157	0.458	0.241	0.024	0.003	115.165	24.489	3.298	0.903

5.1 Verification

According to the combinations proposed by the numerical optimization, the mixes were tested in the lab and the results were compared with the prediction intervals for all the selected options. The tests were performed in the same fashion as the 31 previous mixes.

5.1.1 Verification option 1

Table 9. Components for optimization option 1.

Component	Value m^3	Value mass kg/m^3	Lower limit	Upper limit
(A) Cement (m^3)	0.116	365.4	0.097	0.120
(B) Water (m^3)	0.157	157.0	0.14	0.18
(C) Coarse (m^3)	0.461	1209.0	0.44	0.48
(D) Fine (m^3)	0.241	631.0	0.19	0.25
(E) PET (m^3)	0.022	29.3	0.018	0.040
(F) Superplasticizer (m^3)	0.003	3.0	0.003	0.014

The prediction in option 1 had good agreement with the laboratory tests. The experimental values of the properties fell into the 95% prediction interval. The values of slump (123 mm) and compressive strength (23.8 MPa) are suitable values for the concrete utilization. All four optimization options were tested in the lab, compared with the predicted values and the optimization goals. The option that fulfils both, the workability and compressive strength requirements was option 1. It is important to note that option 4 obtained a higher compressive strength but did not satisfied the workability requirements. Thus, option 1 was selected as the most suitable combination as shown in Table 10.

Table 10. Optimization option 1. Predicted results vs. Experimental results.

Response	Predicted value	Experimental value	Standard deviation	95% Prediction interval Lower limit	 Upper limit
Slump (mm)	120.6	123.0	16.337	82.33	158.90
Compressive strength (MPa)	24.9	23.8	2.324	19.41	30.31
Splitting tensile (MPa)	3.4	3.3	0.270	2.72	3.99

6 CONCLUSIONS

The statistical mixture design approach is shown to be a useful and practical tool for the examination of the influences among components in mixtures. The components with the largest influence on the responses were cement, waste PET, and superplasticizer. Based on the predictive equations, high cement contents had a positive influence on compressive strength and splitting tensile strength, as expected and high waste PET contents decreased the measured properties. High superplasticizer contents had a large positive influence on slump of the mixtures. Finally, through multi-objective optimization, the optimal combinations of components were found. The optimized combination with highest compressive strength was: (1) cement= 365 kg/m^3 (2) water=157 kg/ m^3 (3) coarse aggregate= 1209 kg/m^3 (4) fine aggregate= 631 kg/m^3 and (5) waste PET= 29 kg/m^3 The response values achieved by this combination were: (1) compressive strength 23.8 MPa; (2) slump 123 mm; and (3) splitting tensile of 3.33 MPa. This mix can be used in basements foundation walls or slabs, inside buildings not exposed to freezing temperatures. The amount of fine natural aggregate replaced by waste PET was 8.4%, 58 kg/m^3. Additionally, when substituting this natural aggregate 3755 bottles of waste PET can be recycled. Although the incorporation of waste PET into concrete reached the standards for the properties proposed in this study, additional tests, such as chloride penetration, column leach test, and thermal properties, should be tested to determine the feasibility of the use of waste PET as a fine aggregate substitute.

The present study found that all the properties followed a linear model. If this is known ahead of time, the number of experiments required would be about 15 saving resources and time to investigate other factors and responses.

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