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STATISTICAL ANALYSIS OF THE 3-POINT BENDING PROPERTIES OF POLYMER CONCRETE MADE OF MARBLE POWDER WASTE, SAND GRAINS AND POLYESTER RESIN

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Abstract *This study focuses on the effect on the mechanical performance of concrete polymer beams subjected to 3-point bending that incorporate marble powder waste and quarry sand. This new conglomerate is evaluated for possible substitution of commercial-grade sand in the concrete. Two classes of polymer concrete beams, each with seven formulations, have been produced with dimensions according to the ASTM C580-02 standard. The specimens have been made with identical polyester matrix resin (weight fraction of 14%) and reinforced with marble powder waste in two types of granular skeletons, one based on commercial sand grains and the other with waste from local quarry sand. The target of the analysis was to identify the conglomerates with the maximum strength and highest degree on re-use of waste material. The results have been statistically post-processed using a ANOVA technique, and show that the type of sand, amount of marble powder and sand aggregate have significant effects of the resistance of the polymer concrete beams. The increase of the marble waste provides benefits on the increase of the bending stress due to the consequent reduced porosity of the composite concrete material.*

1. Introduction

Polymer concrete, also known as synthetic resin concrete (PC) [1] is a composite material consisting of dry aggregates in which the monomer (binders) undergo polymerization(curing) after the addition of additives, catalysts, of accelerators [2]. Coarse and fine aggregates, such as crushed stone, sand, gravel and fly ash from waste power plants, are widely used as mineral fillers in the production of PCs. In order to recover the waste and provide improved mechanical properties, minerals such as marble, basalt, and quartz can replace the sand. Burnt rice ball also gives the ash cement properties, and they are used as reinforcing fillers [3-5].

Polymer concretes are usually composed by a thermo setting resin used as a binder for natural or artificial aggregates, which replaces the Portland cement paste / water conventional hydraulic concretes [6]. The first applications of PCs relate to the 1950s and were limited to building coating and to the production of synthetic marble. However, the excellent properties exhibited by these materials have favoured a rapid increase and broadening of their applications. PCs' fast curing, excellent bonding to concrete and reinforcing steel, high strength and durability make them a very attractive repair material despite their poor fire resistance at temperatures exceeding 150° C [7]. As a mortar, PCs can be deposited with less than 10 mm of thickness. Polymer concrete is also widely used for bridge coating surfaces and industrial floors because of their weak layers, rapid curing, very low permeability, high resistance to chemical attacks and freeze-thaw. Nowadays PC begins to be used effectively because of its high strength and lightness as precast in the building sector for bridge decks, hazardous waste containers, machine bases, manufacturing of tiles synthetic marble floors and stairs panels; polymer concrete can also be used in the house and building fronts [8-12].The main advantage of PCs over traditional concrete is their great propensity to integrate recycled waste products, because of the isolating nature of the resin matrix [13]. Recycling and waste encapsulation are now an emerging market in the manufacture of PC. Industrial waste, such as marble and granite powder, fly ash, slag, wood shavings, powder and granulated cork and tire rubber, foundry sands, as well as granules plastic material from crushed waste electrical cables have been used with success to replace the feed components and mineral aggregate in the PC [13-21].Large quantities of waste are produced annually in different countries. Marble and granite waste pose serious environmental threats, like pollution of the soil and water. Moreover when soils are dry, these wastes are transformed into a fine layer of dust that becomes a danger to the public health. Currently most of the waste is land filled and alternative solutions are being examined in many countries to recycle waste in a

durable material, such as polymer concrete. Saboya *and al.* [22] and Gencel *and al.* [23] reported that the amount of cutting waste from sawing and polishing Brazilian decorative stones easily reached 20-25% of the total volume of the raw material. Several studies have also addressed the recycling of marble powder waste in concrete Portland cement, as well as for tarmac floors [21-25]. It is however worth of notice that the use of marble waste in PC components has been scarcely investigated in open literature, apart from the work of Tawfik and Eskander [13]. In the latter work one can find an optimization of the granular structure of a polymer concrete made from polyethylene terephthalate (PET) recycled resin, marble waste and basalt. The PC samples produced have shown high compression for mass fractions of 30% of marble powder ($\Phi > 0.1\text{cm}$), 30% of basalt (0.5-1.0 cm) and 40% of marble (0.1-0.5 cm). The polymer concrete obtained from this study has a rapid hardening, acceptable physical performance and good chemical properties. Martinez-Barrera and Brostow [27] have studied the influence of the gamma radiation and marble particle size on the compression properties and dynamic elasticity module of polymer concrete. Both compression and the dynamic modulus depended upon the specific combination of marble particles sizes and doses of the gamma radiation applied. The increase of the number of dispersed particles per unit volume provided more resistance to crack propagation. Medium size (1.4 mm) marble particles provided an improved compression module. An optimization of the PC granular skeleton was carried out by Elalaoui *and al* [7] by using the compressible stack model approach of the René LCPC software. The resulting granular mixture showed a good compactness and minimum voids. A mass ratio G/S of 0.54 (a blend in terms of weight of 35% of gravel and 65% of sand) gave a compactness of 0.808 sufficient to improve the manufacturing phase of the polymer composite.

The aim of this study is to explore a potential solution for the management of marble powder waste (MW) from local Algerian quarry sand (SW) that provides a total or partial substitution of the marketed sand (MS), which is imported from abroad. Thus, besides the obvious environmental benefits, this approach would also provide a viable and sustainable strategy for the local economy. A statistical study using ANOVA variance analysis was also performed to validate the experimental results by comparing the differences of several groups for a specific significance level selected. The F ANOVA test allows the verification of effects that are significantly different from zero [28].

2. Experimental Procedures

2.1. The constituents of the materials

This work is focused on a synthetic concrete made from granular material with a polymer matrix type. The matrix is unsaturated polyester resin 716.09 manufactured by *Technobell* London characterized experimentally in tension and bending using the ASTM D5083 and D790-03 standards. Two categories of composites are developed and all contain 14% in weight of the same resin. One composite is constituted by marketed sand (MS) having a density of 1.524 g/cm^3 and marble powder waste (MW) having a density equal to 1.425 g/cm^3 . The other category is constituted by quarry sand waste (SW) having a density of 1.675 g/cm^3 with also the same marble powder. The choice of 14% of resin is based on the Elalaoui *et al* [7] work who reported that the porosity and the mechanical strengths in bending show that the optimum polymer content that guarantees obtaining a polymer concrete with the highest mechanical performances and the lowest cost is about 13% of resin. Moreover, Gorninski *et al* [2] have used 12% of orthophthalic polyester and 13% of isophthalic polyester for their polymer concrete preparation. The SW is washed and steamed in an oven at 105°C for 24 hours to reduce the humidity and ensure a good bonding between the aggregates and the polyester matrix, while the MS is only steamed in an oven under the same conditions of the SW. Particle size analysis(NFP18-560)standards indicated for the two sands the presence of particles with varying diameters (1 to 3 mm), while for the marble waste the powders showed smaller sizes ranging from 0.02 to 1.4 mm. Figure 1 shows the particle size distribution curves of these two aggregates. To design a durable polymer concrete with adequate physical and mechanical properties requires the optimization of its granular skeleton to minimize the porosity. To this end, further seven formulations of composites have been investigated, all with varying compositions of the sands (GC and G: 0%, 16%, 26%, 43%, 60%, 70%, 86%), and waste marble powder (M: 86%, 70%, 60%, 43%, 26%, 16%, 0%) (See Table 1).

Fig.1.Particle Size Distribution

The polymer concrete is made by mixing the sand with marble waste powder to obtain a mixture of aggregates that is then gradually introduced in the polyester resin, premixed with a solution of methyl ethyl ketone Peroxide in dimethyl phthalate (AKPEROX A.60) as catalyst and cobalt ethylhexanoate in aliphatic ester as an accelerator (1 % each), while stirring with a spatula.

TABLE 1. Polymer concrete samples made using marketed sand (MS) and quarry sand waste (SW).

Formulations		Resin (%)	Sand (%)	Marble waste (MW)(%)
Sand (MS)	Sand (SW)			
MS0 /MW86	SW0 / MW86	14	0	86
MS16/ MW70	SW16/ MW70	14	16	70
MS26/MW60	SW26 /MW60	14	26	60
MS43 /MW43	SW43 /MW43	14	43	43
MS60 /MW26	SW60/ MW26	14	60	26
MS70/MW16	SW70/ MW16	14	70	16
MS86/ MW0	SW86 /MW0	14	86	0

The resulting polymer concrete is ready to be poured in two prism molds, which can each contain seven specimens of dimensions 25x25x300 mm according to ASTM C580.02 standard. The mold filling is done in two steps; the half filled molds are firstly placed on a vibrating table to obtain a good compaction of the polymer concrete, and then the filling is completed. Wax serving as a liner has been used on the walls of the molds to facilitate the demolding, which has been performed after 24h (see Figure 2). The samples are dried at room temperature for at least 7 days before being tested.

Fig.2. Polymer concrete samples with different formulations

2.3 Experimental procedures

Three-points bending static tests (Figure 3) have been performed in the laboratory of the University of Guelma using a universal servo-electric testing machine type Zwick/Roell Z005 with a 5 kN load cell and displacement rate of 3 mm/min. The machine is controlled by a computer allowing the launch of testing and data acquisition via the testXpert V10.11 software. The bending tensile strength is given, according to ASTM C580.02, by the following formula:

$$\sigma_f = (3Pl)/2bh^2 \quad (1)$$

where: P (N) is the ultimate strength, l (mm) the distance between supports and b (mm) and h (mm) are the width and height of the specimen. Four specimens were tested for each formulation under ambient temperature (25°C) with 60 % of humidity.

Fig. 3. Static Bending test

3. Results

3.1. Experimental Results

A good knowledge of the porosity is essential for any technical work on granular materials. The water accessible porosity was calculated by vacuum saturation with a hydrostatic balance, according to the NF P18-459 standard. The porosity is obtained from the mass of the samples measured in saturated air (M_{air}) in water (M_{water}) and oven dried at 50 ° C (M_{dry}) using the equation:

$$Porosity (\%) = 100 (M_{air} - M_{dry}) / (M_{air} - M_{water}) \quad (2)$$

where: M_{air} and M_{water} (kg) are the saturated specimen mass weight in the air and the water respectively, while M_{dry} (kg) is the dry mass of the specimen.

In construction materials, the porosity is generally measured by using the Archimedes method, from which the apparent densities of the specimens are also obtained. The bulk densities and porosities (measured according to the NF EN 18-459 standard) obtained for the SW and MS specimens are presented in Table 2. The lowest density is present in specimens containing no marble. With the incorporation of the marble powder the density increases gradually until reaches the maximum values of 2267 Kg / m³ and 2432 Kg / m³ for the specimens prepared with SW and MS, respectively.

TABLE 2. Bulk density of different formulations.

Marble weight fraction (%)		0	16	26	43	60	70	86
Bulk density (Kg/m ³)	SW*	2062	2174	2242	2257	2266	2267	2192
	MS*	2082	2390	2432	2417	2241	2236	2192

* Weight fraction of S Wand MS = 86% – marble weight fraction

Fig. 4. Porosity versus marble weight fraction.

The maximum porosity is 12% and 9% for specimens prepared with SW and MS containing no marble powder (Figure 4). The porosity then reduces and becomes almost constant for formulations containing 26% to 70% of marble in the range of 0.9 to 1.10% for the SW specimens, and 1.09 to 1.45% for the MS samples. A slight increase in porosity (2.61%) is however observed for specimen made completely with marble.

The mechanical properties of the resin alone (MS0 MW0) under tension and bending loading are shown in Table 3. The characteristics obtained from the tests are similar to the ones of cured unreinforced polyester resin 716.09 manufactured by Technobell London [29] and used in the manufacture of polymer concrete pipes (MAGHREB pipe industry M'sila, Algeria).

TABLE 3. Characteristics of the resin

Resin	Type of solicitation					
	Tensile			flexural		
	Stress MPa	Strain %	Young modulus GPa	Stress MPa	Strain %	Young modulus GPa
Present study	29.36 ± 2.87	2.25 ± 0.24	12.128 ± 2.04	81.74 ± 11.04	2.56 ± 0.52	3.33 ± 0.25
<i>Technobell</i>	50 - 70	4 - 5	3.2 - 3.6	70 - 90	---	---
London [29]	ISO R 527	ISO R 527	ISO R 527	ISO 178		

The bending test results related to the fourteen PC formulations are given in Table 4. The values in the table represent the means stresses and displacements obtained and their corresponding standard deviations. These results have been compared with the ones of Shokrieh, *et al.* [30]. In the latter reference the samples were produced using a mixture with 25% of resin and 75% of fine silica sand and maximum grain diameter of 5 mm.

TABLE 4. Mean values of the bending stress for the different formulations and the respective displacements.

	Formulation	Stress, MPa	Displacement, mm	Flexural module, GPa
Present study	MS0/MW86	28.49 ± 1.94	0.717 ± 0.083	21.10 ± 1.97
	SW0/MW86	28.49 ± 1.94	0.717 ± 0.083	21.10 ± 1.97
	MS16/MW70	32.68 ± 1.32	0.756 ± 0.096	19.55 ± 2.80
	SW16/MW70	32.89 ± 2.29	0.710 ± 0.081	20.86 ± 2.80
	MS26/MW60	34.54 ± 2.06	0.726 ± 0.010	19.96 ± 1.80
	SW26/MW60	31.89 ± 1.69	0.677 ± 0.023	20.92 ± 0.78
	MS43/MW43	35.19 ± 1.75	0.674 ± 0.014	23.00 ± 1.40
	SW43/MW43	29.06 ± 0.34	0.766 ± 0.100	16.52 ± 1.93
	MS60/MW26	37.98 ± 1.06	0.663 ± 0.043	25.90 ± 1.80
	SW60/MW26	25.71 ± 1.24	0.667 ± 0.032	12.58 ± 1.08
	MS70/MW16	29.38 ± 2.20	1.755 ± 0.391	5.23 ± 2.60
	SW70/MW16	18.07 ± 1.68	0.946 ± 0.224	3.91 ± 3.70
	MS86/MW0	12.57 ± 3.34	1.332 ± 0.859	7.60 ± 5.0
	SW86/MW0	12.00 ± 0.16	1.278 ± 0.149	3.08 ± 2.90
Shokrieh <i>et al.</i> [30]	MS75	19.69	2.96	

The dimensions of the specimens tested in bending with 250 mm of span are 400mmx75mmx75mm. The polymer concrete in [30] has an average flexural strength of 19.69 MPa with a displacement of 2.96 mm. The ones obtained in this study show a broad range of performance, with the lowest resistance at 12 MPa for specimens made with sand only to a maximum of 37.98 MPa and 32.89 MPa obtained with 26% and 70% of marble powder in specimens made with marketed sand and quarry sand waste, respectively. The reason behind this increase of the flexural strength is linked to the significant reduction of voids, leading to a decrease in porosity. The lowest displacement of 0.663 mm is obtained for the composite MS60/MW26 while the highest one that is found equal to 1.755 mm is obtained for MS70/MW16. The displacement of 2.96 mm obtained by Shokrieh et al. [30] is much greater than that of the present work. This is due, on one hand, to the proportion of the resin used which is higher (25% instead of 14 % in our case). On the other hand, the nature of the resin used which has 4% of elongation at break that is much bigger than that used in the present work which is only 2.56%. With regard to flexural modulus, the MS70 / MW16 formulation that gave the best resistance (37.98 Mpa) also gave the best stiffness (25.90 Gpa) but with the smallest displacement (0.663 mm).

Figure 5 shows the variation of the bending stress as a function of the porosity. It is quite evident the decrease of the stress with the increasing porosity.

Fig. 5. Flexural stress as function of the polymer concrete porosity.

Figures 6a and 6b represent the stress-displacement curves for the four samples tested by type with the best PC formulations (MS60/MW26 and SW16/MW70). The evolution of the stress versus the displacement occurs in three steps. During the first the behaviour of the beam is almost linear, followed by a displacement stiffening effect and a very short third step during which the stress falls sharply with an abrupt failure of the specimen. Table 4 shows a remarkable dispersion of the stress at failure (CV=28%) between the different formulations of the polymer composite. The main effects for each factor and their interactions can be investigated by assessing the level of the average response using an ANOVA variance analysis.

Fig. 6. Stress-displacement curves for the specimens subjected to 3-points bending: a) MS60/ MW26 and b) SW16 /MW70 specimens.

3.2. ANOVA analysis

The analysis of variance of the experimental results for the samples prepared with MS and SW is presented in Table 5. The ANOVA results are listed in terms of degrees of freedom (DF), squares sum (SS), squares mean values (SM), Fisher ratio test (F-value) and p-value. The calculated values allow an assessment of the relative importance of each test factor. The analysis of the variance provides a comparison between the differences existing in multiple groups for a certain significance level selected. The Fisher test allows verifying which effects are significantly different from zero. The F-values are calculated as the ratio between the average squares of the group (or interaction factor) and the mean square of the random errors [28]. The F-critical value (F_{crit}) can be found on the F Snedecor table, depending on the significance level (α) and the degrees of freedom of the numerator and the denominator. If F is less than F_{crit} , the

null hypothesis is accepted, which means the groups are not significantly different. F_{crit} was determined at a significance level of 5%, which is commonly used in practice [28].

As the P value of the F test is less than 0.05 for the MS and SW specimens' ultimate stresses ($F = 56.59$ and $F = 93.61$ respectively above $F_{crit} = 3.23$), it can be concluded that a statistically significant difference is present in the average mechanical characteristics from a formulation to another at a 95% confidence interval. The null hypothesis H_0 is therefore is rejected.

Table 5. Variance analysis of different formulations with MS and SW.

Source	DF	P-value	Formulations with MS			Formulations with SW		
			SS	MS	F-value	SS	MS	F-value
Formulation	6	0.00	1699.2	283.2	56.59	1418.3	236.4	93.61
Error	21		105.1	5.00		53.03	2.52	
Total	27		1804.3			1471.4		
DF : degree of freedom			MS : squares average					
SS: sum of squares			F : ANOVA F-test					

4. Discussions

4.1 Marble powder waste (MW) influence on the flexural strength

The results from the experimental tests obtained (Figures 7a and 7b) indicate that the partial substitution of marketed sand aggregates (MS) or quarry sand waste (SW) by marble powder waste (MW) leads to a gradual increase of the flexural strength up to a 70% marble powder inclusion. Beyond this value, the strength decreases due to the increase of the porosity. For the first type of polymer concrete with MS, the best flexural resistance is provided by the MS60/MW26 formulation in which a 26% substitution of MW leads to an increase of 202% in flexural stress compared to the basic formulation containing no marble. For the other types of SW polymer concrete the best formulation is represented by the SW26/MW70 that contains 70% MW and provides an increase of 174% of the tensile flexural strength compared to the basic

formulation (Table 4). The variability of the results is due to the granularity induced by the increase of the reinforcement constituted by the marble content in the granular skeleton. The increase of the marble content up to 70% contributes to an improvement in the flexural resistance, and this is related with the reduction of the porosity with a lower volume of voids, from 12% to 0.9% for the SW samples and 9% at 1.09% for samples with MS.

Fig. 7. Curves representing the values of the stress versus the various formulations.

a) With marketed sand (MS) and marble powder waste (MW); b) With quarry sand waste (SW) and marble powder waste (MW).

4.2 Substitution of marketed sand (MS) by quarry sand waste (SW)

Figure 8 shows the variation of the flexural stress versus the marble content for polymer concretes fabricated using MS and SW. The histogram shows a significant increase of the flexural stress with increasing marble content. The best formulation of PC using MS (MS60/MW26) appears to provide an increase of 15% in flexural strength compared to the best one fabricated using SW (SW16/MW70). The best formulation of the PC fabricated with sand waste of 70% marble rate, while the best one for the concrete with MS has 26% of MW only.

Fig.8. Flexural stress for SW and MS specimens versus MW content.

A cost comparison has also been made between the two best formulations to determine the most economically viable type of polymer concrete (Figure 9). The replacement of marketed sand by sand waste and mixed with MW can indeed provide both an environmentally sustainable and economical viable material with acceptable flexural properties. This polymer concrete material is also less expensive than the baseline one because of the high content of marble powder.

Fig. 9. Comparison of the costs for the different constituents of the PC and their best formulations.

5. Conclusion

In this study fourteen formulations with different levels of marble powder waste content (MW) and marketed sand (MS) replaced by waste quarry sand (SW) have been investigated, and their effect on the bending strength of beams has been evaluated. Based on the results obtained, the following conclusions can be drawn:

- The 3-point bending tests carried out show that the stress/displacement behaviour is nearly linear or quasi-linear up to failure. The results obtained are characterized by dispersions statistically evaluated using ANOVA;
- The incorporation of the marble powder provides an increase in density due to a decrease in porosity;
- The analysis of one-way ANOVA shows that the P value of test F is less than 0.05. One can therefore observe a statistically significant difference in the mechanical characteristics, with the marble powder and aggregates content factors affecting the bending strength ;
- The partial replacement of the marketed sand aggregate by the marble powder waste contributes to an increase up to 202% (for MS60/MW26) of the bending strength of the concrete against the only sand marketed (MS86/MW0) case. Similarly, the replacement with the quarry sand waste (SW) provides an improvement of 174% on bending strength of the PC with its best formulation (SW16 MW70);
- The maximum stress observed for the polymer concrete developed with the SW and MW inclusions is only 15% lower than the one featured by the samples with MS and MW. The proposed polymer composites provide an environmentally friendly material made with marble powder and sand waste, lower costs and acceptable flexural resistance.

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