CORRELATIONS ON ULTRA PULSE VELOCITY AND PHYSICAL-MECHANICAL PROPERTIES OF CEMENT MORTAR CONTAINING QUARTZ AND RECYCLED-PP AGGREGATES

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https://doi.org/10.21452/bccm4.2018.02.20

Abstract
The consumption of polymeric materials and their waste increases considerably throughout the world, leading to a constant concern with alternative recycling routes. Polypropylene (PP) is one of the most produced, but it is not recycled in an expressive way. A significant amount of research has been conducted to reuse discarded material into new composites, especially the cementitious ones, combining environmental, economic and technological issues. Non-destructive tests, such as ultra-pulse velocity, can be used to characterize and estimate the physical and mechanical properties of cement-based materials. This work investigates the effect of partial replacement of natural aggregates (NA) by recycled PP aggregates (PP-RPA) on pulse velocity and physical-mechanical properties of mortars and their correlations. Coarse particles of PP (at 4 -10 US-Tyler and 10 – 20 US-Tyler) provided better mechanical behaviour to composites made with fine particles (20–50 US Tyler), the latter being largely affected by the presence of pores. Ultra-pulse velocity can be used to predict the physical and mechanical properties of mortars containing PP aggregates.
Keywords: mortar, recycled plastic aggregate (RPA), polypropylene (PP), mechanical properties, ultra-pulse velocity (UPV)

1. INTRODUCTION

The reuse of solid waste has been a growing concern in today´s modern world. Several residues are disposed in the environment without proper control or treatment. A significant amount of research in materials science has been conducted to find alternative routes for such waste, combining environmental, economic and technological issues [1–4].

In this context, there is a considerable increase in the production and consumption of polymeric materials, as well as the production of solid polymeric waste [3, 5]. This growth has occurred all over the world due to its friendly properties such as low cost, durability, strength and low density [6]. According to a survey carried out by Ferreira *et al.* [2], data from the Central Pollution Control Panel indicate that there is a consumption of about 150 million tons of polymers per year in the world, with a per capita production of 25kg/year. In addition, estimative indicates that every 10 years there is a doubling of the world production of polymer waste [2].

Among possible alternatives for the reuse of polymeric materials, an inexpensive and environmentally friendly way is their use in the production of cement products [2, 3]. The use of polymer fibres for concrete reinforcement was studied for the first time during the 90's. Subsequently, new studies have addressed the production of concrete with polymer admixtures and, more recently, with recycled plastic aggregates (RPAs) [2].

The replacement of natural aggregates (NAs) by RPAs directly affects the physical and mechanical properties of cement-based materials [3, 6–9]. A reduction in density, which is an interesting factor for some cement products, such as tiles, is obtained by the RPA inclusions, but the compressive strength and porosity are impaired. It is therefore necessary to develop experimental studies related to the incorporation of polypropylene – PP-RPA in cement products for their physical-mechanical characterisation [3, 10].

Ultra-pulse velocity is a simple, fast and inexpensive non-destructive test for the characterization of cement products. The UPV test has many advantages compared with the conventional non-destructives testing methods, because it is linked to elastic mechanical properties of the test material [11] and changes of ultrasonic pulses parameters can be used to determine the damage and defects in cement based materials [12, 13]. Some authors have developed theoretical models for the prediction of physical-mechanical properties of cementitious composites based on UPV measurements [11, 14–24].

This work investigates the effect of the partial replacement of the NAs with PP-RPA wastes on the physical-mechanical properties, such as compressive strength, ultra-pulse velocity (UPV), dynamic modulus of elasticity, bulk density, apparent porosity and water absorption, of mortars for concrete tiles. In addition, the relationships between the UPV and the physical-mechanical properties are evaluated.

2. EXPERIMENTAL

2.1. Composite preparation

The cementitious composites were fabricated with ordinary Portland Cement (OPC), quartz and polypropylene (PP) aggregates. The OPC was a Brazilian CP V ARI (ASTM Type III) sourced by Holcim®. The quartz aggregates were sourced by *Moinhos Gerais* Mining Company (Brazil). PP-RPAs were derived from the comminution of Coke® PET bottle caps. The bottle caps were initially washed in water to remove impurities and, subsequently, the polymer was granulated into small pieces using a knife mill.

The composite mix is based in an aggregate particle size distribution (PSD) used in the production of tiles. Table 1 shows the reference PSD as well as the particle size envelopes used in this study.

Table 1: Quartz particle size distribution for cementitious tiles and work setup

General particle size distribution for concrete tiles		Particle size envelope (wt%)
Sieve US-Tyler – (mm)	Retained (%)	

4 US-Tyler – (5mm)	0	4-10 US-Tyler (15wt%)
5 US- Tyler – (4 mm)	5	
7 US- Tyler – (2.8 mm)	11.4	
9 US- Tyler – (2 mm)	15.89	10-20 US-Tyler (40wt%)
16 US- Tyler – (1 mm)	27.08	
32 US- Tyler – (0.500 mm)	28.44	20-50 US-Tyler (30wt%)
60 US- Tyler – (0.250 mm)	10.91	50-100 US-Tyler (15wt%)
115 US- Tyler – (0.125 mm)	1.28	

Six different batches were prepared, including one reference batch and five batches containing PP-RPA as a partial replacement for the NAs, corresponding to the experimental treatments (C1 to C6) shown in Table 2. Four samples for each experimental condition were fabricated, in a total of 24 specimens.

Table 2: Experimental Treatments

Experimental Treatments	NA/RPA replacement particle size (amount)
C1-ref	---
C2-15	4-10 US-Tyler (15wt%)
C3-30	20-50 US-Tyler (30wt%)
C4-40	10-20 US-Tyler (40wt%)
C5-55	4-20 US-Tyler (55wt%)
C6-85	4-50 US-Tyler (85wt%)

A water/cement ratio (w/c) of 0.42 and an aggregate/cement (a/c) ratio of 3.75 were maintained constant for all composites. The w/c chosen in this study was sufficient to permit full hydration of the cement, as well as adequate compaction of the samples. A Marshall Hammer was used to produce the cylindrical specimens in order to reproduce the level of compaction of extruded concrete products in the laboratory (Figure 1). A constant volume and 6 hammed-blows were considered for each sample, resulting in cylinders with approximately 50 mm in height and 54.5 mm in diameter. The cylinders were demoulded immediately after the compaction. The samples were kept in a closed container for 28 days at room temperature for curing. The response variables investigated were the ultra-pulse velocity, bulk density, apparent density, apparent porosity and compressive strength.

Figure 1: Cylindrical samples

2.2. Bulk density

The bulk density (ρ) of the composites was calculated by dividing the dry mass of the samples after 28 days of curing (m_1) by its volume (V). The bulk density and volume were calculated using the equations (1) and (2), respectively, where D is the diameter and L is the length of the sample:

$$\rho = \frac{m_1}{V} \tag{1}$$

$$V = \frac{\pi D^2}{4} \times L \tag{2}$$

2.3. Ultra-pulse velocity and dynamic modulus of elasticity

A pulse of longitudinal vibrations is produced by an electro-acoustic transducer which is held in contact with one surface of the concrete sample under test [25]. After traversing a known path length L in the concrete, the pulse of vibrations is converted into an electrical signal by a second transducer. Electronic timing circuits enable the measurement of the transit time T of the pulse to be measured. The ultrasonic measurement is conducted using a portable ultrasonic non-destructive digital indicating tester (Pundit Plus) with a 150 kHz transducer. The pulse velocity (in m/s) is given by equation (3):

$$v = \frac{L}{T} \tag{3}$$

The dynamic modulus of elasticity (E_d) is a measure that express the relationship between pulse velocity and elastic constants [25]. The dynamic modulus of elasticity is given by equation (4).

$$E_d = Bv^2 \frac{(1+\mu)(1-2\mu)}{(1-\mu)} \tag{4}$$

where μ is the dynamic Poisson's ratio which value for concrete varies generally in the range of 0.15-0.22 [26]. In this work the Poisson´s Ratio was considered at 0.15 which is recommended for mortars [27].

2.4. Apparent porosity

The apparent porosity (p) represents a percentage or fraction of the void spaces in a material, varying from 0 to 100% or 0 to 1. The apparent porosity was calculated according to British Standard 10545-3 [28]. The samples were weighted after 28 days of curing and the dry mass (m_1) was recorded. Subsequently, the samples were saturated with water under vacuum and the saturated mass (m_2) and the suspended saturated mass (m_3) were measured. The apparent porosity was calculated using the equation (5).

$$p = \frac{m_2 - m_1}{m_2 - m_3} \tag{5}$$

2.5. Water Absorption

The water absorption (E) is the percentage of water absorbed by the sample after being immersed in water under vacuum, as described for the determination of the apparent porosity, following the BS 10545-3 [28]. The equation (6) was used to calculate the water absorption.

$$E = \frac{m_2 - m_1}{m_1} \times 100 \tag{6}$$

2.6. Compressive strength

The British Standard 12390-3 [29] was used to measure the compressive strength of the samples. The compressive strength can be determined by dividing the maximum force applied to the sample (F_{max}) by its initial area (A_0), according to the equation (7).

$$f_c = \frac{F_{max}}{A_0} \tag{7}$$

The compression tests were carried out at 1mm/min using a AG-X Plus SHIMADZU testing machine equipped with a load cell of 100 kN.

3. RESULTS

Table 3 summarizes the mean and standard deviation of the investigated properties. It is noteworthy that the investigated factor depends on particle size and amount levels. In general, the physical-mechanical properties are substantially affected by the PP-replacement levels.

In general, the porosity and water absorption increased while the UPV, bulk density compressive strength and dynamic modulus decreased when a large amount of PP particles was incorporated as quartz replacement. It is noteworthy, C3 composite made with 30wt% of PP at 20-50US-Tyler particle range achieved lower elastic properties than C4 and C5 composites made with 40wt% and 55wt% of PP particles, respectively. This behaviour can be attributed to the presence of fine PP particles, with larger surface area, in C3 composites, affecting the packing of particles of the system.

Table 3: Experimental Results

Setup	Pulse Velocity (m/s)	Bulk Density (g/cm^3)	Apparent Porosity (%)	Water Absorption (%)	Compressive Strength (MPa)	Dynamic Modulus (GPa)
C1-ref	4182.78±252.34	2.11±0.07	7.04±2.41	3.25±1.23	29.87±6.60	42.77±6.34
C2-15	3670.20±168.92	1.94±0.07	8.91±2.67	4.47±1.49	20.76±5.71	30.21±3.74
C3-30	2800.71±124.81	1.66±0.05	14.01±1.20	8.09±0.84	7.88±1.52	15.04±1.74
C4-40	2666.40±120.05	1.66±0.06	11.35±2.09	6.63±1.43	11.84±2.26	13.58±1.61
C5-55	2385.62±117.58	1.52±0.02	11.38±1.78	7.22±1.27	8.64±2.00	9.99±0.99
C6-85	1872.84±179.61	1.22±0.12	14.12±1.74	11.54±1.72	5.16±1.14	4.92±0.670

Backscattering electron images were obtained at 30× (Figure 2) and 100× (Figure 3) of magnification to verify the microstructure of the composites and analyse the interface between the PP-RPA and the cement matrix. Higher NA/PP-RPA replacement led to increased amount of pores. Besides, larger pore sizes were observed when fine PP particles were incorporated, as revealed by C3 composites (Figure 3c). Fine low-density particles contribute to increase the volume of PP particles in the system, consequently, affecting the porosity and the elastic properties. The interfacial transition zone (ITZ) between the PP particles and cement was more porous when fine particles were used (Figure 3c), in contrast to coarse PP particles (Fig. 3a,b).

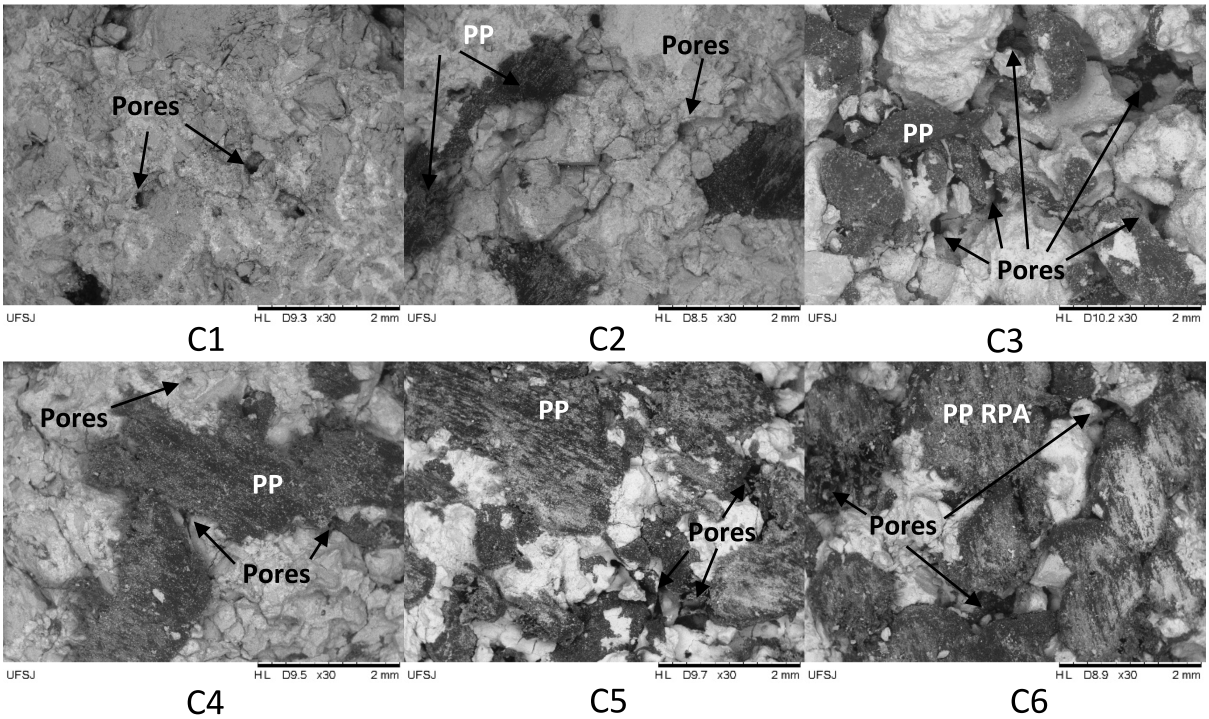
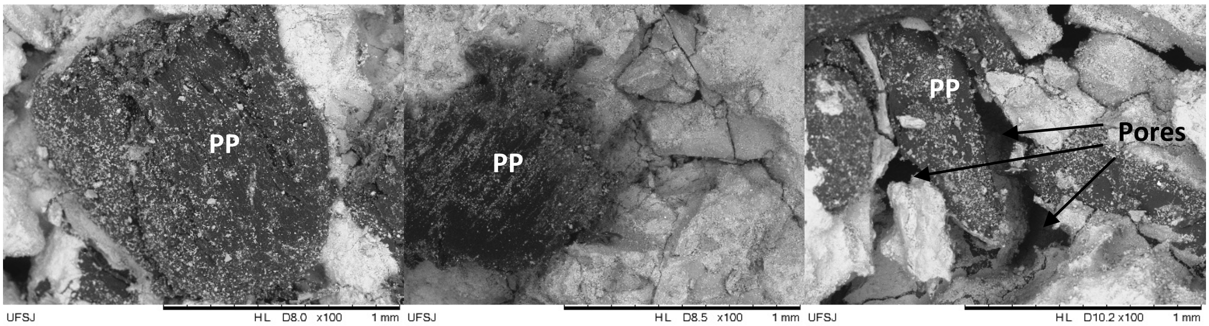


Figure 2: Backscattering electron images of the experimental treatments



4-10 US-Tyler (a) 10-20 US-Tyler (b) 20-50 US-Tyler (c)

Figure 3: PP-RPA and matrix phase interface evaluation

1. 3.1. Statistical Analysis

The software Minitab17® was used to perform all the statistical analysis. A correlation analysis of the responses variables is shown in Table 4. The first and second rows for each response exhibit the Person Correlation (PC) and the P-value, respectively.

Table 4: Correlation Analysis

		Pulse Velocity	Bulk Density	Apparent Porosity	Water Absorption	Compressive Strength
Bulk Density	PC	0.960				

	P-value	0.000				
Apparent Porosity	PC	-0.794	-0.790			
	P-value	0.000	0.000			
Water Absorption	PC	-0.889	-0.926	0.921		
	P-value	0.000	0.000	0.000		
Compressive Strength	PC	0.929	0.899	-0.914	-0.884	
	P-value	0.000	0.000	0.000	0.000	
Dynamic Modulus of Elasticity	PC	0.983	0.938	-0.820	-0.859	0.966
	P-value	0.000	0.000	0.000	0.000	0.00

The PC ranges from -1 to 1, and the further away from 0, the greater the correlation between the variables. A significant correlation occurs when the P-value is less than or equal to 0.05, considering a confidence level of 95% (α=0.05). As shown in Table 5, the correlations between the analysed variables are significant (P-value < 0.05) and strong (PC > 0.600). Based on response correlations, a multivariate analysis of variance (MANOVA) [30] was performed and presented in Table 5. The MANOVA tests the equality of several means at the same time using the covariance structure among the variables, unlike the ANOVA that tests one by one. When the analysed variables are correlated, the MANOVA design is able to detect a smaller mean variation compared to ANOVAs. As successive ANOVAs are performed, the chances of incorrectly rejecting the null hypothesis increase, which does not occur in MANOVA, since the means are tested at the same time, maintaining the error at the alpha level. Significant effect factors are indicated by MANOVA when the P-value is lower than 0.05. Each treatment can be considered a specific composite and its physical-mechanical properties are affected by the size of the NA / RPA replacement particles.

Table 5: ANOVA and MANOVA

		P-value	R^2 (adj.) (%)
ANOVA	Pulse velocity	0.000	95.69
	Bulk Density	0.000	94.18
	Apparent Porosity	0.000	58.56
	Water absorption	0.000	79.03
	Compressive Strength	0.000	83.39
	Dynamic Modulus of Elasticity	0.000	94.46
MANOVA	Wilks' Test	0.000	

A Regression Analysis was performed to model a relationship between the responses (Figure 4). Table 6 shows the equations and the adjustments of the generated models (R^2).

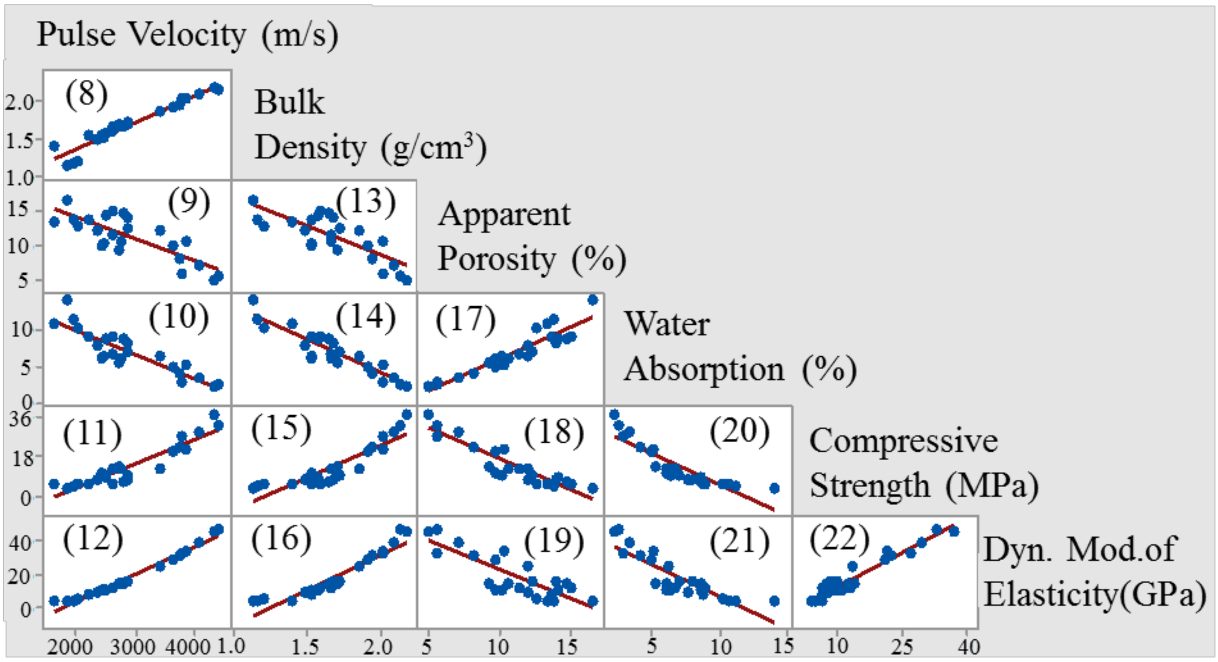


Figure 4: Correlation between the response variable

Table 6: Regression analysis

		Model	R^2 (%)
(8)	$B(v)$	$B = 0.6469 + 0.000355v$	91.74
(9)	$p(v)$	$p = 20.28 - 0.003120v$	61.40
10)	$E(v)$	$E = 16.45 - 0.003272v$	78.08
11)	$f_c(v)$	$f_c = -17.85 + 0{,}010879v$	85.75
12)	$E_d(v)$	$E_d = -29.05 + 0.016544v$	96.55
13)	$p(B)$	$p = 25.29 - 8.39B$	60.73
14)	$E(B)$	$E = 22.41 - 9.210B$	85.08
15)	$f_c(B)$	$f_c = -33.94 + 28.43B$	79.87
16)	$E_d(B)$	$E_d = -52.55 + 42.66B$	87.47
17)	$E(p)$	$E = -2.734 + 0.8624p$	84.05
18)	$f_c(p)$	$f_c = 44.36 - 2.724p$	82.84
19)	$E_d(p)$	$E_d = 58.51 - 3.511p$	65.74
20)	$f_c(E)$	$f_c = 33.33 - 2.810E$	77.08
21)	$E_d(E)$	$E_d = 46.39 - 3.927E$	72.60
22)	$E_d(f_c)$	$E_d = -0.04 + 1.3878f_c$	92.95

A linear relation was identified among the variables. Based on the regression analysis, satisfactory adjustments of the models were evidenced, except for (9), (13) and (19), which exhibited R^2 values close to 60%. This fact can be attributed to the porosity (p) response variable involved, which is determined using three measures of dry mass (m_1), saturated mass (m_2) and suspended saturated mass (m_3). Suspended saturated mass measurements should be further affected because the PP particle density is lower than the water density. The water absorption response (E) is similar to the porosity, however its models have an adequate adjustment. The calculation of water absorption does not consider m_3, which evidences the effect of the suspended mass previously commented.

As most models showed high R^2, it is possible to reliably estimate the physical-mechanical properties based on these models. It is worth mentioning that the models involving UPV (see Table 6 (8-12)) can be extremely useful, since they are obtained by means of a non-destructive test, being possible to estimate the other properties, impacting the cost, ease and speed of the characterization [14].

4. CONCLUSIONS

This paper investigated the effect of the partial replacement of NAs with PP-RPAs wastes on the physical-mechanical properties of sustainable mortar for tiles and the relationship between the ultra pulse velocity and the other responses. The conclusions are described as follows:

(a) MANOVA indicated that the NA/RPA replacement particle size affects the physical-mechanical properties of the mortars. In general, this replacement has a negative effect on pulse velocity, porosity, water absorption, compressive strength and dynamic modulus, however it reduces the bulk density, which is an interesting behaviour for concrete tiles.

(b) Coarse particles of PP (at 4 -10 US-Tyler and 10 – 20 US-Tyler) provided better mechanical behaviour to composites made with fine particles (20–50 US Tyler); the latter is largely affected by the presence of pores, especially in the interfacial transition zone and the packaging of the particles in the system.

(c) Good correlations were obtained between the response variables studied - pulse velocity, bulk density, apparent porosity, water porosity, compressive strength and dynamic modulus of elasticity - being possible to estimate the properties through a non-destructive parameter, like ultra-pulse velocity. The equations are related to the materials studied in this work, however, similar equations can be developed for other precast concrete materials.

ACKNOWLEDGEMENTS

The authors would like to thank CAPES, CNPq and FAPEMIG for the financial support provided.

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