Implementation of Interaction Diagram of the Properties in Fresh for Mortars with Ceramic Aggregates

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Abstract
As the natural resources needed for the construction sector are limited, new practices are being adopted for the management of waste generated nowadays, including the use of construction and demolition waste as aggregates for concrete and mortar. Considering the different typologies in construction wastes, ceramics are the second most representative material; therefore it is important to validate their feasibility as a total or partial replacement of natural aggregates. This work presents a study of the properties in fresh state (consistency, density and air content) of mortars containing aggregates obtained from recycled ceramics, and their influence on the subsequent properties in the hardened state. A statistical analysis of experimental data was carried out by establishing regression coefficients, and then a triple-entry graph was obtained, allowing the different properties of mortars to be easily linked and simplifying the prediction of the relationships they will present since the mixture design phase.

Keywords
ceramic recycled aggregates, recycled mortars, prediction of properties of mortars, sustainable materials, friendly construction

1 Introduction
Since the natural resources necessary for the construction sector, such as water, raw materials and energy, are limited [1] and it is estimated that their use will continue to increase, governments of various countries and industrial sectors are adopting new practices for better management of the different waste generated, even proposing the recovery of these resources [2] with a view to achieving more sustainable growth in both the economy and society [3]. A feasible alternative for achieving this goal is the recycling of waste and by-products [4] which can be reused as aggregate for mortar and concrete. This gives plausible results related to sustainability benefits, such as the conservation of natural resources and the reduction of transport costs, as well as lessening the environmental impact caused by irregular waste disposal methods [5]–[8]. Besides their possible use in different applications in the construction industry, they may also even improve some properties thereof [9].

In southern Europe the recycling of construction and demolition waste is a low-priority activity. In the case of Italy, for example, which generates 20 M tons per year mostly from masonry and reinforced concrete, just 10% is recycled; the remainder is diverted to landfill [10]. Among the different types of construction waste, those classified as ceramic are the second most representative material [11], accounting for 54% of waste from construction and demolition (C & DW) [12] and currently there is no applicable legislation for them. Likewise, recycled ceramic (so-called second generation) may originate in rejected materials from manufacturers of blocks, bricks, tiles, sanitary ware and electrical insulation. Regarding Spain, as a result of the increase in production (tile manufacture is over 600 million m² per year), these solid wastes have led to an increase of more than 50,000 tons per year [13]. Therefore, it is important to validate their use by incorporating as in either total or partial replacement of aggregate, or as cement in mortars or concretes [14] [15].

In order to establish the feasibility of using this waste, it is necessary to evaluate its properties, ensuring its possible application in competition with traditional materials. The physical properties in their fresh state have been selected as a
preliminary study of the application of these recycled ceramic mortars (CeRM), since they relate and guarantee the proper performance of subsequent hardened-state properties (mechanical and durability) [16].

Previous research into the consistency property of the CeRM indicates that this decreases when the percentage of replacement of the ceramic aggregates (CA) by usual aggregates (UA) increases [17], as more water is needed to make a mixture with suitable consistency [15]. This is explained by the high water absorption that the CA can have. With regard to the density, the CeRM presents a decrease of around 5% when replacing up to 40% of the CA by UA [11] [18]; reaching values of 10% when replacing 50%, and up to 17% for replacements of 100% (compared to the reference mortar). These losses of density (in all cases), were justified by the apparent density of the CA, which is lower than that of the UA [15]. Concerning the air content, a single reference to previous investigations was located; it indicates that for percentages of CA of 5, 10, 20 and 40%, similar values (between 14.3 and 15%) were obtained, which were accepted as being within the specified range of the regulations (British Standard BS 4721) [11].

Since in some cases these properties of the CeRM are not established for all possible CA percentages, and there are not enough references for this subject in previous studies, this research has focused on establishing a system of a triple-interaction diagram and regression equations to determine, in a simple manner, the analogies of relations between these properties, simplifying the prediction of the relations that the different CeRM will present from the mixture design phase. The obtained diagrams will reduce the time required for testing before the optimal desired behavior is obtained, as well as that for selecting the optimal dose for the specific characteristics of strength and durability to which the CeRM will be subjected. Thus, the fresh properties for a particular use are guaranteed as well as laying down the minimum principles for obtaining subsequent properties of hardened concrete.

The triple interaction diagram and equations of regression presented are the result of numerical and statistical analysis of the experimental data. Based on the previous, it is possible to establish the real experimental properties of the consistency, density and air content for the ratio of water/cement (w/c) and cement/sand (c/s) used with different replacement factors (RF) in the CeRM.

2 Materials

CA was used with particle size of 0 - 5 mm, composed of residues of ceramic tiles which had proven defective in their shape or size. This material was put through a No 4 sieve (4.75 mm), in order to separate the fine fraction to be used. UA was employed as the reference aggregate, composed of silica sand with a particle size of 0–4 mm.

Granulometric profiles of the two materials were adjusted to meet the limits indicated in the standard ASTM C144 [19]; both were separated by means of a No. 30 sieve (0.59 mm) thus obtaining two ranges of particle sizes; subsequently, different compositions were made among them until the combination that would result in the maximum compactness (for each material separately) was found. The result of the profile adjustment was as follows: CA with 60% of particles bigger than the No. 30 sieve, and 40% of particles smaller than it, and UA with 50% of particles both bigger and smaller than the No. 30 sieve [20].

The physical properties of the used aggregate were tested according to ASTM standards C128 [21], C136 [22] and C117 [23]; the results are shown in Table 1. The notable differences between both materials can be observed in the lower density of CA with regard to UA (differences of 760.7 and 468.2 kg/m³), while for absorption the CA achieved a higher coefficient than the UA (an increase of 16.8%).

| Property | UA          | CA          |
|----------|-------------|-------------|
| Density oven-dry: $D_{ow}$ (Kg/m³) | 2581.6 | 1820.9 |
| Density saturated-surface-dry: $D_{sw}$ (Kg/m³) | 2623.6 | 2155.4 |
| Water absorption (%) | 1.6 | 18.4 |
| Fineness modulus | 2.4 | 2.8 |
| Materials Finer that 75-μm (sieve No. 200) (%) | 2.9 | 8.2 |

Portland cement, classified as CEM I 42.5 N/SR (UNE EN 197-1: 2011 [24]), was used as a binder because it is a regular product, with the usual properties and components, along with tap water.

3 Methodology

Three series of CeRM mixes were studied, each with different c/a (1:3.25, 1:4 and 1:4.75), and for different replacements of CA by UA (10, 20, 30, 50 and 100%), evaluating their behavior in fresh state (consistency, density and air content). Samples of reference mortars (UM = usual mortar, 100% of UA and for each c/a of study) have been identified as UM-3.25, UM-4.00 and UM-4.75; and for identifying the CeRM, the following agreement was established: CeRM-1:dd-XX. Where: 1:dd = c/a (1:3.25, 1:4 and 1:4.75); and XX = % CA replacing UA in CeRM (10, 20, 30, 50 and 100). All mixes were made with a relationship w/c = 0.5 initial. Table 2 presents the dosing employed in each of the mixes of CeRM.

From the high absorption coefficient of the CA, and to prevent the mobility of water needed for the hydration process, the procedure continued by making a prior one-minute saturation with the resultant water of the initial w/c, before incorporating the cement into the mixer (Mod. E93, Matest brand), and then starting the mixing process at medium speed for 60 seconds. After this period, the mixer continued at high speed for 30 seconds more, with a subsequent 90-second repose and finally 60 seconds at high speed.
The consistency test was carried out at the end of the mixing process, by means of the flow table according to the ASTM C230 [25] standard. It was decided that a flow of $110 \pm 5\%$ would be achieved for this study (in accordance with the ASTM C109 [26]), after applying 25 drops to the sample in the flow table in a period of 15 seconds according to ASTM C1437 standard [27]. This flow was determined by the average measurements of two perpendicular diameters when the mix experienced its induced extension. According to the diameter of the initial mold, the proper consistency was established at $210 \pm 5\ mm$; therefore, if the mix does not achieve this requirement, it will be necessary to increase the water content by small amounts, as many times as necessary, in order to achieve the desired flow.

Once the consistency had been determined, the density of the CeRM in its fresh state was calculated by weighing the content of mortar in the cylindrical container (standard, one liter capacity), and substituting the known data (weight and volume) into Eq. (1).

$$\rho = \frac{M}{V}$$  \hspace{1cm} (1)

Where:

$\rho$ = Density (g/cm$^3$)  
$M$ = Mass (g)  
$V$ = Volume (cm$^3$)

To obtain the air content, a normalized measurer was used (LuftgehaltsprüferTESTING, 1 Liter, brand TESTING), which uses the pressure method (ASTM C231 [28]) with the help of a pre-regulated one liter container. The result of the air content is obtained from the reading on the calibration manometer.

With the above parameters and the studied variables, the statistical analysis computer program SPSS V22 for Windows was applied to the data in order to establish the coefficients of regression, which define the trend equations between the different variables. In this research the linear-type regressions were selected as the most suitable since these presented a better fit in the dispersion diagram of the parameters studied; and with them it is possible to obtain the parameter $R^2$ higher (establishes the degree of reliability of the trend and the accuracy of the forecast, $R^2 \leq 1.0$). As a previous requirement for obtaining regressions, the following requirements were validated for determining a linear regression:

1. For each value of the independent variable, the distribution of the dependent variable is of the normal type.
2. The variance of the distribution of the dependent variable should be constant for all values of the independent variable.
3. The relationship between the dependent variable and each independent variable is linear.
4. All observations are independent.

In each pair of variables of the studied CeRM properties, it was always considered that the independent variables were the property of RF (because there is no precedent in the area of study), leaving the density and the w/c relationships as dependent variables, allowing the regression equations to be established by the mathematical formulation that best predicted the dataset of the dependent variable. In the case of the air content property the regression was not performed because of failure to comply with some of the necessary validations indicated above; however, this property is presented in graphs with the average value obtained for every family of study (referred to the w/c ratio, by more uniform behavior).

The trend lines are determined by calculating the points of study (set of independent and dependent variables) by the method of least squares adjustment, and using the following Eq. 2 as the structure of its formula:

$$y = mx + b$$  \hspace{1cm} (2)

Where:

$x$ = Value $x_i$ of the independent variable  
$y$ = Value $y_i$ of the dependent variable  
$m$ = The slope of the straight line  
$b$ = The intersection of the straight line with the vertical axis

| Classification | Materials | M-25 | M-25-10 | M-25-20 | M-25-30 | M-25-50 | M-25-100 | M-400 | M-400-100 | M-400-200 | M-400-300 | M-400-500 | M-400-1000 | M-475 | M-475-20 | M-475-30 | M-475-50 | M-475-1000 |
|---------------|-----------|------|--------|--------|--------|--------|---------|-------|-----------|-----------|-----------|-----------|--------------|-------|--------|-------|--------|-------------|
| AU** (g)      |           |      |        |        |        |        |         |       |           |           |           |           |              |       |        |       |        |              |
| <sieve 30    | 781       | 672  | 588    | 500    | 331    | 0      | 800     | 780   | 610       | 521       | 348       | 0          | 811           | 711   | 633    | 526   | 343    | 0           |
| >sieve 30    | 781       | 672  | 588    | 500    | 331    | 0      | 800     | 780   | 610       | 521       | 348       | 0          | 811           | 711   | 633    | 526   | 343    | 0           |
| CA** (g)     |           |      |        |        |        |        |         |       |           |           |           |           |              |       |        |       |        |              |
| <sieve 30    | 0         | 60   | 118    | 171    | 265    | 418    | 0       | 69    | 122       | 179       | 278       | 517        | 0              | 63    | 127    | 180   | 274    | 639         |
| >sieve 30    | 0         | 90   | 176    | 257    | 397    | 627    | 0       | 104   | 183       | 268       | 417       | 775        | 0              | 95    | 190    | 271   | 412    | 959         |
| Water (g)    |           |      |        |        |        |        |         |       |           |           |           |           |              |       |        |       |        |              |
|               | 327       | 338  | 361    | 346    | 393    | 353    | 334     | 390   | 355       | 373       | 397       | 476        | 334            | 366   | 378    | 386   | 400    | 586         |

** Dry condition.
4 Results and discussion

Below are the obtained results relevant to the tests of consistency (quantity of water required), air content, density and final w/c relationship obtained from the CeRM of study (see Table 3).

| Material    | Density (g/cm$^3$) | Air content (%) | w/c final |
|-------------|--------------------|-----------------|-----------|
| UM-3.25     | 2.18               | 3.7             | 0.68      |
| CeRM-3.25-10| 2.15               | 4.0             | 0.74      |
| CeRM-3.25-20| 2.12               | 4.0             | 0.80      |
| CeRM-3.25-30| 2.09               | 3.5             | 0.79      |
| CeRM-3.25-50| 2.05               | 3.7             | 0.96      |
| CeRM-3.25-100| 1.96              | 3.5             | 1.10      |
| UM-4.00     | 2.16               | 3.8             | 0.84      |
| CeRM-4.00-10| 2.13               | 3.8             | 0.90      |
| CeRM-4.00-20| 2.12               | 3.9             | 0.93      |
| CeRM-4.00-30| 2.09               | 4.2             | 1.00      |
| CeRM-4.00-50| 2.05               | 3.8             | 1.14      |
| CeRM-4.00-100| 1.95              | 3.7             | 1.48      |
| UM-4.75     | 2.15               | 3.8             | 0.98      |
| CeRM-4.75-10| 2.10               | 5.1             | 1.10      |
| CeRM-4.75-20| 2.09               | 4.5             | 1.13      |
| CeRM-4.75-30| 2.06               | 4.2             | 1.22      |
| CeRM-4.75-50| 2.00               | 4.3             | 1.39      |
| CeRM-4.75-100| 1.91              | 5.1             | 1.74      |

The coefficients of the regression equations were obtained from these results and a triple-entry diagram was developed with theoretical-experimental correlations for the properties in fresh condition of CeRM studied. Figure 1 shows the results of density and w/c ratio with respect to RF, for each different ratio of c/a. From the diagram it is possible to confirm that the relationship between RF and density is inverse (negative sign in equations), while the RF relationship and the w/c is direct. This indicates that an increase in RF in the CeRM causes a decrease in its density and a growth in its w/c ratio. On the other hand, the term m (slope of the line) in the regressions indicates that the change between RF and density of CeRM is smaller than the change between RF and w/c ratio. This permits the assertion that the RF has more influence on the value of the w/c ratio than on the density of CeRM; as a result the relationship between RF and w/c ratio could be considered more important than the predictive behavior in the interaction diagrams obtained.

Concerning the studied values of c/a (3.25, 4.00 and 4.75), the inter-distance between the obtained regression lines (and also of the straight lines with experimental values) with respect to density indicates that, in this case, the straight lines do not maintain between them a parallel or a radial traced to a common origin (even there are intersections for the case of c/a = 3.25 and 4.00); this could indicate the possible existence of another variable (not established in the study) that causes another ‘extra’ coefficient in the regression equations, producing an acceleration in the change of density due to the increase of the c/a ratio (it is possible that this behavior is due to the structure of the cementing matrix of the CeRM, as well as in the hydration process affected by the presence of the CA). On the other hand, for the case of the c/a ratio with respect to the w/c ratio, the straight lines are also not parallel to each other, but display a radial path intersecting at a common point, which shows that this indicator is of normal proportionality between them, providing more solidity as a tool for predicting behavior.
To validate the obtained interaction diagram and check its usefulness, a comparison is made with two other previous studies that have sufficient data to be used in the diagram. In both, the 20, 50 and 100% [29], and 5 and 10% [15] of the UA has been replaced by CA. For the specific case of predicting density by using the interaction diagram, and using the RF and the c/a ratio as input data, the trend of predictive data presents similar behavior to that indicated in the graph of Figure 2; however, they show an average approximation, or accuracy, error of between 7.3 and 5.3% respectively, these differences being greater at high RF values (in the specific case of [15]). These differences could be attributed to variables not included in this research, such as the density of aggregates (45% less in previous researches) compared to those used to validate the interaction diagrams proposed in this research; it is also possible that the w/c ratio should be considered as another factor that allows greater accuracy in predictions.

From this validation, it is possible to assert that the diagram obtained in this research can be used to predict the properties of the CeRM with similar characteristics (CA properties); and as this research progresses—when more experimental data, including more variables, becomes available—a more complete diagram involving more variables could be obtained, improving predictions and making them more widely applicable.

5 Conclusions
The following conclusions were reached in this work:

- Having information about the behavior that the CeRM may present, from the design phase of mixes of its properties in a fresh state, allows the correct adaptation to be anticipated through the specific use of mixture proportions and CA replacement, adapting these needs to the functions desired.
- The information obtained from this diagram of interaction can be used as a reference for other similar diagrams, in which, hardened state properties are included (such as resistance to compression).
- When using this diagram of interaction, it should be ensured that the data of the CeRM used have similar characteristics (density of CA and w/c relationship, among others).
- It is necessary to carry out more tests in the future by means of different variables not included in this research, in order to obtain more general diagrams with multiple options for different possible alternatives.

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