A New Method to Determine the Density and Water Absorption of Fine Recycled Aggregates

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The construction industry keeps on demanding huge quantities of natural resources, mainly minerals for mortars and concrete production. The depletion of many quarries and environmental concerns about reducing the dumping of construction and demolition waste in quarries have led to an increase in the procuring and use of recycled aggregates from this type of waste. If they are to be incorporated in concrete and mortars it is essential to know their properties to guarantee the adequate performance of the end products, in both mechanical and durability-related terms. Existing regulated tests were developed for natural aggregates, however, and several problems arise when they are applied to recycled aggregates, especially fine recycled aggregates (FRA). This paper describes the main problems encountered with these tests and proposes an alternative method to determine the density and water absorption of FRA that removes them. The use of sodium hexametaphosphate solutions in the water absorption test has proven to improve its efficiency, minimizing cohesion between particles and helping to release entrained air.

Keywords: fine recycled aggregates, density, water absorption

1. Introduction

By the year 2010, construction was responsible for consuming annually about 37.4 billion tons of natural aggregates and it is expected that by the year 2015 those figures can reach about 48 billion tons1. The sustainable development demanded by Humanity at this stage requires a clear reduction of that figure. Reusing coarse recycled aggregates (CRA) from construction and demolition waste (C&DW) has been successfully implemented in road pavements2,3. In concrete production it is common practice today as a partial replacement of natural aggregates since various studies have demonstrated that its performance is satisfactory4-9. Also, the use of recycled aggregates on mortars has been studied with very interesting results10-12. Several countries already enforce regulations that encourage its use and define the percentage of natural aggregates to be replaced by recycled aggregates13.

The initial results for the fine fraction of these recycled aggregates (size below 4 mm) were not promising, which led some codes and technical specifications to strongly advise against the use of such materials in concrete because of the problems it may cause during mixing and in the hardened state14. One of the properties that most distinguishes fine recycled aggregates (FRA) from fine natural aggregates (FNA) is water absorption; this is substantially higher in the former with damaging consequences for the workability, strength and durability of the concrete15-17. Also the fact that FRA have higher length to width ratios and are more irregularly-shaped than FNA15,18, which was confirmed by the higher specific surface the former19, leads to a higher water demand in order to maintain workability.

As the availability of FNA and dumping grounds for the unused fine fraction of the C&DW decreased, the study of FRA gained new momentum, with various researchers obtaining satisfactory results14,20-22. To obtain concrete with good characteristics made with FRA it is paramount to know their properties in detail before use.

Water absorption and density are properties prior knowledge of which is decisive to defining the aggregate’s quality23. These properties are presently determined in FNA using the procedures set out in standard EN 1097-624. Although suitable for FNA this standard does not contemplate certain peculiar features of FRA: their origins vary, making them a material of great heterogeneity; they contain a non-negligible amount of ultra-fine particles (below 0.063 mm) possibly including cohesive clays and partially hydrated cement particles25. Furthermore, the existing test does not determine the amount of water absorbed by the aggregates as a function of time. Other European norms, such as BS812: Part 2 26 used for coarse aggregates, also have problems when used to determine the water absorption of recycled aggregates.

Studies have been published that determine this property more accurately for CRA27. But none were found that address the problems of applying EN 1097-6 to the fine fraction.

This paper presents a new method to measure the density and water absorption of FRA with the goals of overcoming the problems of the lab measurements yielded by the existing standard and determining their water absorption during the test.
2. The Importance of Water Absorption by Aggregates

The quality of the aggregates plays a fundamental role in the quality of concrete. This is a reflection of the geometrical, physical and chemical properties of the aggregates. One of the most important factors in concrete’s performance is the mix’s water/cement ratio. Water is essential to the cement hydration that gives concrete its mechanical strength, but it must be dosed correctly. Too much water increases the concrete’s porosity, thus decreasing its mechanical performance and durability. A shortage of water in the mix will lead to incomplete cement hydration reactions and a reduction of the fresh concrete’s workability.

When added to the mix, very porous aggregates such as FRA absorb part of the mixing water before adequate cement hydration occurs. Besides being high, the water absorption of FRA shows considerable scatter with values ranging from 4% to 17% in selected research works. This absorption may be dealt with in two ways: pre-saturating the aggregates or compensating with the mixing water. Previous studies have demonstrated that the pre-saturation technique negatively affects, albeit very moderately, the concrete’s properties, and it is preferable to compensate with the mixing water. Furthermore, pre-saturation is impracticable with FRA because of the small size of the particles. The compensating process requires adding enough extra water to the mix to account for the water absorbed by the aggregates during mixing. For this, correct knowledge of the water absorption of the aggregates both before they are added to the mix and after saturation is essential.

For natural aggregates this is usually not a problem because they are not particularly porous and so have low water absorption potential. But recycled aggregates absorb far more water and it is imperative to compensate for this during concrete production.

3. Methods to Determine Density and Water Absorption

3.1. Determination of the density and water absorption according to EN 1097-6

This standard establishes two distinct tests for three size ranges of aggregates. The pycnometer method was applied to the aggregates under analysis (size below 4 mm). The procedures used in these experiments are the ones presented here. They follow the standard, but in a slightly different sequence that has no effect on the final result and makes the test much simpler and more efficient.

According to the standard a sample of at least 1.5 kg must be prepared using the procedure described in EN 932-3 standard. The sample must be washed and then submerged for 24 hours to saturate. Then it is air-dried until reaching the saturated surface-dry condition. After the mass of a fraction of the aggregate is registered it must be introduced in the pycnometer and the remaining space is filled with water to the brim, and the sample is then weighed. Then the water is removed and the aggregate particles are filtered through a sieve covered by a filter paper. The particles are then placed in a ventilated oven for at least 24 hours to constant mass and registered as the oven-dried aggregate mass.

With these data it is then possible to determine the density of the particle material (oven dried, saturated surface-dry and relative), as well as the total water absorption.

3.2. Problems with the test according to EN 1097-6

When performing the test according to the European standard a number of factors were observed that prevented a reliable result:

- When the sample is placed in the pycnometer and filled with water the finer particles tend to settle and fill all the voids (Figure 1). This stops the occluded air between the particles from rising to the surface, leading to incorrect results. Even though most of the fine particles are removed by washing, the porous and pulverulent nature of the FRA allows many particles to remain after washing (some clayish with cohesive characteristics) and others to be produced by the disaggregation of bigger particles during the drying process;
- The drying process generates small lumps of particles that prevent the proper drying of each individual particle, because of the clay particles that remain after washing and their cohesive nature (Figure 2); and

Figure 1. Settling of the finer particles.
It is not possible to know how the absorption increases over time since the test only provides the overall/potential water absorption. The amount of water absorbed at the beginning of the test, which is important for the correct dosage of the concrete mixing water, remains unknown.

3.3. Hydrostatic scale test

To determine the FRA’s water absorption over time a test initially described by Leite\(^1\) was used, in which a few small changes were made. In this test the sample is submerged and the evolution of the hydrostatic mass is measured using a scale. It takes 24 hours and the increase of the hydrostatic mass during that period corresponds to the aggregate’s water absorption.

A sample of at least 1 kg is dried in an oven to constant mass after being washed over a 0.063 mm sieve. After removal from the oven the sample’s mass is registered and it is placed at the bottom of the set of sieves prescribed in EN 933-2 standard\(^2\). The base’s lid is the 0.044 mm sieve and the set is connected to a hydrostatic scale and then submerged. From this point on the gain in hydrostatic mass is monitored at regular intervals that increase as the test proceeds. The data obtained is used to determine the overall water absorption of the aggregate during the test.

3.4. Problems with hydrostatic scale test

This test had two problems when applied to FRA:
- The water absorption relative to the initial instant is not known, since the sample has already absorbed some water when the first reading was obtained;
- Some extremely fine material left over from washing the sample, derived from the FRA’ porous and pulverulent nature, prevents occluded air from exiting the sample because of low fluidity and generates incoherent readings. The material also suffers incipient particle agglomeration, as is evident at the end of the test (Figure 3). This phenomenon is more obvious in FRA samples predominantly from crushed concrete.

4. Experimental Work

4.1. Samples

The tests were performed on samples from a Portuguese C&DW recycling plant. The material collected ranges from 0 to 8 mm. It was size graded at the lab and only the fraction from 0 to 4 mm was used for the tests. Strictly speaking the EN 1097-6 procedure analyses the water absorption of the fine aggregate from 0.063 mm to 4 mm. The fraction below 0.063 mm in FNA is usually small. But this fraction is important in FRA, specifically it is 23% of the overall weight of the sample of this research. Several attempts were made to determine the density and water absorption of the <0.063 mm fraction with the new method, but without success, as described below, due to their high clay content. Clay has cohesive characteristics that block the test procedures and prevent the use of unwashed FRA as they are collected at the plant for concrete production.

4.2. Use of sodium hexametaphosphate as particle dispersant

Bearing in mind that a good part of the very fine material (<0.063 mm fraction) in the samples collected consists of clays, efforts were made to find a way to prevent their cohesion. Sodium hexametaphosphate \([\text{NaPO}_3\text{O}_6]\) is a well-known clay dispersant used to prepare clay suspensions for various studies\(^3,4\). Its application in soil analyses is widespread and is has even been standardised\(^5\). To prevent cohesion of the particles, sodium hexametaphosphate was diluted in the demineralised water used for the test. A concentration of 1g/L of water was used, as suggested in the literature\(^3,8\). The use of this compound also allows air to be release from the samples more efficiently.

4.3. Tests performed

Initially the <0.063 mm fraction was included in the tests, according to both the EN 1097-6 standard and the hydrostatic scale test. It was soon clear that these particles prevented the normal running of the test.

In the EN 1097-6 procedures the strong cohesion caused by the clays led to a failure of the test at the drying stage.
because of the formation of lumps. The saturation of the aggregate with sodium hexametaphosphate solution did not solve the problem, though it reduced it to some extent. Since it was impossible to go beyond the drying stage no results could be obtained from this test.

In the hydrostatic scale test, even though a result was obtained at the end, it was not reliable because of the problems mentioned above, which significantly increase when the very small fines are present. It was found that when the metal frame supporting the sieves was not stirred between measurements the hydrostatic mass measurement of the unit was affected. This was due to the exiting occluded air bubbles becoming caught in the sieves. If the frame was stirred they would exit, thus changing the measurements. Another experiment involved sieving a dry sample and so partially eliminating the <0.063 mm fraction. The same problems were encountered.

It was thus decided that the aggregates had to be washed before performing the tests, which adds an extra task to the use of FRA in concrete production.

### 5. New method to determine density and water absorption of FRA

#### 5.1. Procedures

An alternative method to determine the water absorption of FRA is now presented. It combines the pycnometer method with the hydrostatic scale test but only uses the >0.063 mm fraction.

The hydrostatic scale test is used to determine the FRA’s water absorption over time, but it only measures water absorption relative to the first reading, usually 2 to 3 minutes after immersion of the FRA. The relative overall value is then supplemented by using the pycnometer method to obtain the absolute overall water absorption and the density. Knowing this value it is then possible to determine the amount of water absorbed by the aggregates between their immersion and the first reading on the scale. The procedures always consider the use of a sodium hexametaphosphate solution with the concentration stated above.

#### 5.1.1. Stage 1: Pycnometer method

A sample is prepared according to EN 932-1, from which a specimen weighing at least 1.5 kg and representative of the material under analysis is taken.

The specimen is washed on top of a 0.063 mm sieve to remove the finer particles.

Five litres of sodium hexametaphosphate solution of 1 g/L are prepared.

Distilled water is used to prepare the solution. The specimen is immersed in the solution for 24 hours to guarantee it is completely saturated.

Most of the solution covering the aggregate is decanted and the specimen is dried using a hot air stream.

The specimen is stirred often to ensure homogeneous drying.

To check when the specimen reaches the saturated surface-dry state the cone test is used in accordance with EN 1097-6.

As soon as the specimen reaches that state a sample is collected (no lighter than 1 kg) into a tray and its mass is registered as M1.

The material is placed in the pycnometer which is filled with the solution to the brim.

The pycnometer is placed in lukewarm water (22 °C) for 24 hours.

After that measurements are taken to make sure all the remaining occluded air bubbles come out.

The pycnometer is dried on the outside and its mass is registered as M2.

A 0.063 mm sieve and a paper filter are weighed and their mass registered.

The paper filter is positioned underneath the sieve and adapted to its circular shape. The entire contents of the pycnometer are emptied through the sieve, with the paper filter thus collecting all aggregate particles within it.

The sieve and its contents are placed in an oven at (110 ± 5 °C) to constant mass.

The pycnometer is now filled with the solution alone and dried on the outside and its mass is registered as M3.

The sieve is removed from the ventilated oven and weighed.

The mass of the sieve and the paper filter is deducted and the result is registered as M4.

The particle densities ($\rho_a$, $\rho_{sd}$, $\rho_{mm}$), in mega grams per cubic meter, and the water absorption ratio are determined according to the following equations where $\rho_a$ is the density of the sodium hexametaphosphate solution (assumed approximately equal to that of water) at 20 °C: relative particle density (1); saturated surface-dry density (2); oven-dried density (3); water absorption (as percentage of dry mass) after immersion for 24 hours (4).

$$\rho_a = \frac{M_4}{\left[ M_4 - (M_2 - M_3) / \rho_o \right]}$$  \hspace{1cm} (1)

$$\rho_{sd} = \frac{M_4}{\left[ M_1 - (M_2 - M_3) / \rho_o \right]}$$  \hspace{1cm} (2)

$$\rho_{mm} = \frac{M_1}{\left[ M_1 - (M_2 - M_3) / \rho_o \right]}$$  \hspace{1cm} (3)

$$WA_{sd} = \frac{100 \times (M_1 - M_4)}{M_4}$$  \hspace{1cm} (4)

#### 5.1.2. Stage 2: Hydrostatic scale

A specimen of at least 1 kg and prepared according to the EN 932-1 procedures is dried in a ventilated oven to constant mass.

The sample is removed from the oven, weighed and the result is called $M_{sd}$.

The specimen is placed at the bottom of a set of sieves mixed in a sodium hexametaphosphate solution.

The chronometer is switched on and the specimen is stirred in the solution with a trowel.

As soon as possible a 0.044 mm sieve is put in place as a lid and the unit is mounted on the metal frame that connects the set of sieves with the hydrostatic scale.
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After connection the set is submerged in a tank containing a sodium hexametaphosphate solution with 1 g/L, and the gain in hydrostatic mass is then monitored for 24 hours as follows:

- First reading after stabilisation of the scale, registered as \( M_{\text{sub,0}} \);
- One reading every 2 minutes for first 10 minutes;
- From 10 to 30 minutes, one reading every 5 minutes;
- From 30 minutes to 1 hour, one reading every 10 minutes;
- From the first hour to the second, one reading every 15 minutes and from then on one reading per hour until 9 hours has elapsed; and
- One final reading after 24 hours, registered as \( M_{\text{sub,24h}} \).

The frame is removed from the tank; it is completely cleaned of aggregate particles, and is immerged again. The value on the scale is registered as \( M_{\text{eq}} \).

The hydrostatic mass of the specimen in the first and the final readings is given by (5) and (6):

\[
M_{\text{sub,0}} = M_0 - M_{\text{eq}} \\
M_{\text{sub,F}} = M_{24h} - M_{\text{eq}}
\]

where: \( M_{\text{sub,0}} \) - hydrostatic mass of the specimen in the first reading; \( M_{\text{sub,F}} \) - hydrostatic mass of the specimen in the final reading.

The water absorbed by the immersed specimen between the first and the final readings of the test is given by (7):

\[
W_{A_{\text{sub}}} = \frac{M_{\text{sub,F}} - M_{\text{sub,0}}}{M_{\text{sub,0}}}
\]

Between readings the container must be carefully stirred to facilitate the exit of the occluded air.

5.1.3. Calculation and expression of the results

The pycnometer stage of the test provides the absolute overall water absorption of the aggregate and its density. The hydrostatic scale stage of the test gives the overall water absorption from the first reading, usually 2 to 3 minutes after immersion of the aggregates, as well as its progress over time.

It is thus possible to know the water absorption of the aggregates on the first reading of the hydrostatic scale by determining the difference between these two figures. It is given by expression (8).

\[
W_{A_{\text{initial}}} = W_{A_{24h}} - W_{A_{\text{sub}}}
\]

The progress of water absorption must be presented graphically. The water absorption relative to any time when a reading has been made as described can also be determined using Equation (9).

\[
W_{A_{\text{sub},t}} = \frac{M_{\text{sub,t}} - M_{\text{sub,0}}}{M_{\text{sub,0}}}
\]

where: \( W_{A_{\text{sub},t}} \) - water absorption between the first reading and instant \( t \); \( M_{\text{sub,t}} \) - hydrostatic mass of the specimen at instant \( t \); \( M_{\text{sub,0}} \) - hydrostatic mass of the specimen in the first reading.

5.2. Experimental results

Tests were conducted to confirm the viability of the method presented. Two types of recycle fines were used: one specimen of C&DW from a Portuguese recycling plant, consisting of a mixture of concrete and masonry, and one sample from recycled concrete. In the first stage no cohesion was observed during drying of the aggregate. The air occluded between the particles when the specimen was in the pycnometer with a sodium hexametaphosphate solution exited notably faster than in all previous tests, in which the solution was not used and the aggregates were not washed. Although no absolute evidence was found that entrained air bubbles were completely removed, visual observation of the apparatus seems to indicate that.

Then the hydrostatic scale was used, leading to the graphs in Figure 4 (average values and standard deviations). The values obtained are shown in Table 1. The absorption given by the hydrostatic scale is slightly lower than that from the pycnometer. Since the same sample was tested in both methods and under the same conditions it is assumed that the difference is the water content that the aggregate absorbs between being submerged in the hydrostatic scale test and the first reading taken after 3 minutes. The initial water absorptions were 2.58 and 1.62%, respectively.

It is thus proved that the water absorbed by FRA in the first instants of this test (which goes undetected in the conventional test) is important since it accounts for a considerable amount of the water absorption capacity.

5.3. Advantages of this method

The benefits of using the alternative test presented here instead of the conventional procedures in EN 1097-6 are:

- The problems with the cohesion of clays observed during the test cease with the use of sodium hexametaphosphate. The exit of occluded air bubbles within the specimens is easier because of the low resistance offered by the particles. The prior mixing of the solution with the specimen, in the hydrostatic scale test, also substantially improves the exit of the air in the specimen after immersion, leading to more realistic results of the test;
Table 1. Results of the hydrostatic scale test after washing the aggregates, and using a sodium hexametaphosphate solution.

|                         | C&DW   | Concrete |
|-------------------------|--------|----------|
| Hydrostatic mass of the set ‘metal frame + sieves’ (g) | 2017.3 | 2080.0   |
| Initial hydrostatic mass of the sample (g)              | 557.8  | 637.3    |
| Final hydrostatic mass of the sample (g)                | 575.9  | 688.8    |
| Final water absorption relative to the submerged mass in the first reading (%) | 3.25   | 8.08     |

- The water absorption can be known as soon as the FRA are submerged, when it occurs faster and is most important since it coincides with the mixing period of concrete or mortars; and
- The water absorption relative to any chosen period can be known (thus enabling the compensation method to be adapted to the duration of the mixing process), as well as the overall absorption and the densities.

6. Conclusions

It is a known fact that water absorption of FRA is significantly higher than their natural counterparts. In addition, FRA tend to present cohesiveness or binding properties, which make standard test procedures difficult to implement. To mitigate these specificities, a new test procedure was developed, which uses sodium hexametaphosphate as particle dispersant.

From the tests that were conducted it can be concluded that the new proposed method is effective on the elimination of particle cohesion and helping the release of entrained air, because of the low resistance offered by the particles.

This methodology also allows to know the water absorption as soon as the FRA are submerged, when it occurs faster and is most important, since it coincides with the typical mixing period of concrete.

The water absorption relative to any chosen period can be known, as well as the overall absorption and the densities.

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