A COMPARATIVE STUDY ASSESSING THE VALIDITY OF WATER ABSORPTION METHODS WHEN APPLIED TO SECONDARY FINE AGGREGATES

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Abstract

The valorization of secondary sands is crucial for fostering a circular economy within the construction sector. One significant obstacle in this endeavor stems from the high water absorption (WA) rates commonly reported for secondary sands. This high WA leads to increased water and cement demand in cementitious mixes and elevates costs. The only widely standardized method to measure the WA of fine aggregates is the cone method, which has proven ill-suited for fine aggregates with angular particles or a high content of fines. This study undertakes a comparative evaluation of the cone method and seven alternative methods: immersion, centrifugation, paper sheet, colorimetric, volumetric flask, continuous drying, and electrical conductivity. Tests were performed on natural river sand and three types of secondary sand. Findings reveal that the continuous drying and electrical conductivity methods demonstrates the greatest potential in terms of reliability and applicability. Also the volumetric flask and centrifugation methods show some potential, provided that critical refinements are made to the corresponding test protocols.

Keywords: secondary sand, water absorption tests, fine recycled aggregate, cone method, electrical conductivity method, continuous drying method

List of notation and abbreviations

RCA recycled concrete aggregates

CRCA coarse recycled concrete aggregates

FRCA fine recycled concrete aggregates

NA natural aggregates

FNA fine natural aggregates

SSD saturated Surface dry condition

WA water absorption

w/c water-to-cement ratio

1. Introduction

Understanding the physical properties of aggregates is essential for producing concrete mixes that meet specific criteria. In recent years, various studies have focused on design

methods to incorporate recycled aggregates into concrete, considering factors such as sustainability, concrete properties, and statistical analysis [1-5]. These methods along with the most conventional methods [6-7] emphasize the importance of characterizing aggregates. Water absorption (WA) is a key parameter in this characterization process.

The effects of one of the most studied secondary aggregates (i.e., any aggregate different from natural sand requiring processing for use in concrete production), recycled concrete aggregate (RCA), are primarily and indubitably governed by its high WA compared to natural aggregates (NA). This is due to the residual cement paste adhering to the particles. The WA of RCA is influenced by the properties of the parent concrete, i.e., compressive strength level, mineralogy of NA used, and the crushing process by which RCA is produced [8-9]. Notably, a much wider range of WA values is generally reported for fine (<4 mm size) RCA (FRCA), with values of 2.38-19.3%, than for the coarse RCA, ranging from 3.10 to 8.34% (Figure 1). Both of these ranges are considerably higher than those reported for most NAs: 0.3-3.0%, which are largely dependent on their mineralogy [10]. The interval for FRCA is 2.27 times wider than that for CRCA. The impact of the adhered cement paste appears to be much higher for the fine fraction than for the coarse fraction. The wider range also suggests a larger variability in the WA of FRCA than for the coarse fraction, possible due to less precision or accuracy in the experimental measurements.

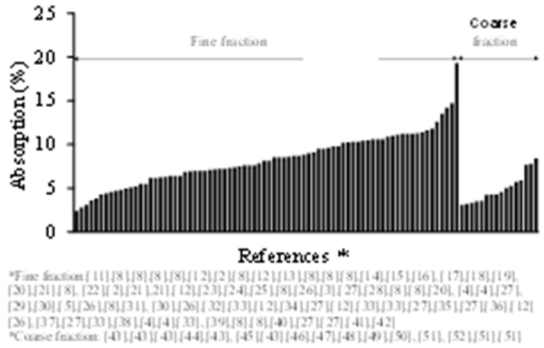


Figure 1. Water absorption of RCA

In general, standards from across different countries employ similar methodologies to determine the WA value for fine natural aggregates (FNA). The cone method is the most widely used technique worldwide. The suitability of this method for secondary sands, such as FRCA, has been strongly questioned [8,53]. Therefore, the features of secondary sands make the determination of their WA more challenging. For this, more than eight alternative methods have been proposed and cited in the literature. Discrepancies between WA values derived from the cone method and those from alternative methods can be significant in some cases (Figure 2.a). Typically, the WA values from alternative methods are higher than those obtained using the cone method (Figure 2.b). This observation seems to be a reflection of over-drying needs to achieve the shape indicated in the standard and interpreted as the saturated surface-dry (SSD) condition [8,53-55]. However, there is a notable lack of correlation between the cone method and other techniques, suggesting that the difference between the cone method and the other methods would have a significant non-systematic component.

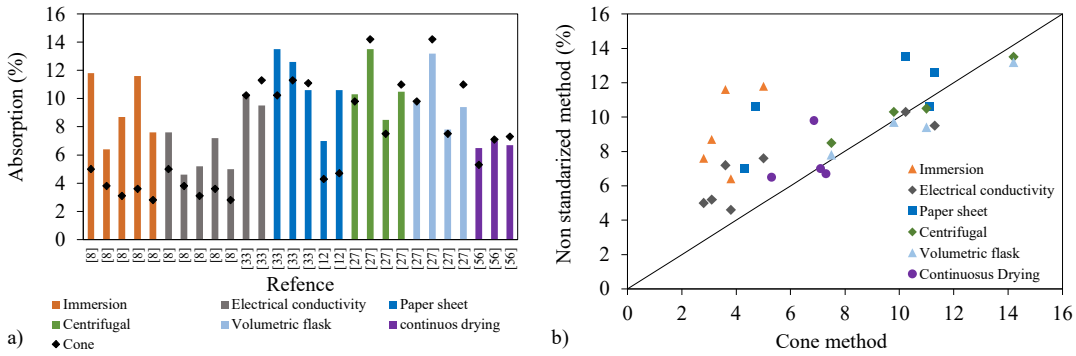


Figure 2. a) Water absorption measured by different methods. b) correlation between WA rates by the cone and other methods.

The higher WA of FRCA compared with FNA is often incorrectly addressed, and it impacts negatively on concrete performance when FRCA is used [29,57-58]. A recent study [59] found that an incorrect calculation of the WA value may have led to the lower compressive strength of some FRCA concrete mixes compared to regular mixes.

The accurate determination of WA greatly affects the properties of concrete in which FRCA is used. This is because it dictates the amount of water added to the mix to compensate for the water uptake from the unsaturated FRCA and avoid a change in the w/c ratio. When FRCA is used pre-saturated with the water quantity corresponding to its absorption, the w/c is not modified. However, this is also miscomputed if the wrong WA value is used for this. The subjective nature of the cone method for the determination of the WA randomly changes the w/c ratio of recycled aggregate concrete. This means that comparisons with conventional mixes reflect not only the effects of FRCA but also the deficient quantification of the WA of FRCA and consequent variations in the w/c ratio.

The water absorption of FRCA is an extensively studied topic. More than 8 methods have been proposed to determine this property, but the results obtained from the various methods are seldom compared with others in the literature. Duan et al [60] provides a state-of-the-art review on the various methods and discusses some of their potential limitations. The present study goes a step further by presenting a comprehensive comparison of all proposed methods for determining the WA of FRCA.

This study revisits several methods proposed in the literature for determining the WA of secondary sands and FRCA in particular. The primary variables for each method are considered, and the unresolved issues, benefits, and limitations are discussed. Additionally, this study investigates the extent to which various experimental methods produce different WA values. The aim is to contribute to discussions on best practices and to offer insights on the genuine impact of FRCA on concrete properties.

2. Methods for determining WA in FRCA

2.1. Cone method

It is the method widely used to estimate the SSD condition of FNA and is still the most used in the literature for any other sort of sand, including secondary sands and FRCA. The method is based on the cohesion developed between particles when a film of surface water is present on their surface. As particles are subjected to progressive drying, this film of water eventually disappears, and cohesion reduces until the material adopts the repose

angle (maintained only by its internal friction). The ASTM C 128 standard [61] mentions the additional difficulties of applying this method to manufactured (i.e., angular) sands or sands with a high content of fines <63 μm, but they do not indicate clear procedures to deal with these difficulties. While applying progressive drying, the method defines the SSD condition for the sample when it takes on a defined shape after removing the cone mold in which it was previously placed and compacted.

In spite of the subjective assessment by the operator following the description in the standard (which is actually slightly different in the various international standards), this method has proved to be effective in determining the WA in FNA with a rounded shape and low content of fines. Conversely, it has been several times questioned for aggregates with high internal friction and/or high content of fines [8,33,53-55,62-64]. Even in one of the standards (ASTM C 128 [61]), the precision bias (0.31%) does not apply when the WA of the aggregates is higher than 1% or for manufactured aggregates. Additionally, for aggregates with a high content of fines, the standard suggests alternative and complementary methods to determine the SSD condition, but these are also subjective to the operator's appraisal [8].

Manufactured aggregates with a high content of fines or high internal friction seem to be the most challenging for the cone method. In aggregates with a high content of fines, cohesion increases due to the finest particles. A high specific surface to volume ratio maximizes the surface phenomena because small particle size generates more menisci for a certain volume of surface water. This is therefore the main driver for interparticle cohesion. When achieving the shape established in the standard for the SSD condition, a differential drying according to the size of particles is eventually playing a role. Aggregates have a higher internal friction when particles are highly angular and with a rough texture. The contact points between particles define the flow mechanism [65] of loose particles. In such cases, cohesive forces play a secondary role. Therefore, these two features reduce the suitability of the cone method.

2.2. Immersion method

This method, originally proposed for RCA with particle size > 44 μm [58], was applied by various authors [37,57,67]. The method involves preconditioning the sample by washing-off the particles below 44 μm, drying the sample, and then immersing it in water to continuously register the weight increase as it absorbs water. The sample must be carefully agitated to release occluded air bubbles before each measurement. The WA is computed as the average between WAd (with the dry weight as a reference) and WAi (with the immersed weight as a reference), calculated according to Eqs. 1 and 2, respectively.

$$\text{WAd (\%)} = \frac{Wif - Wii}{Wd} \times 100 \qquad \text{eq(1)}$$

$$\text{WAi (\%)} = \frac{Wif - Wii}{Wii} \times 100 \qquad \text{eq(2)}$$

Where:

WAd = absorption of dry material

WAi = absorption of immersed material

Wif= sample weight in immersion at the end of the test

Wii= sample weight in immersion at the beginning of the test

Wd= sample dry weight

It is important to mention that the average value obtained as the outcome does not strictly adhere to the definition of WA. Equation 1 represents the water present in the pores relative to the dry mass of aggregate. This corresponds to the definition of WA, with the limitation that Wii is excluding a minimal amount of water absorption occurring before this first reading is complete. Conversely, Eq. 2 computes the water present in the pores with respect to the initial immersed weight of the 'dry' sample, i.e., [Wd – weight of displaced fluid by the 'dry' sample]. As it takes some time to complete this first reading (for the stabilization of the scale), the sample is no longer fully dry at the time of the reading. Equation 2 is an expression with no clear physical meaning. Thus, the calculation of WA as an average between both values does not have solid ground. Djerbi [68] proposes the same method as Leite [58] to measure the WA of coarse recycled aggregates, but using only Eq. 1 for the calculation. They report values in almost perfect correlation to those obtained by applying EN 1097. For this reason, it seems convenient to consider both the WA values calculated according to Eq. 1 and the WA values derived from the average of Eqs. 1 and 2.

Some additional problems have been reported with this method. First, the challenge of eliminating air occlusion causes inconsistent measurements and the agglomeration of particles in the sample, which subsequently increases the air occlusion [66]. To improve this, Rodriguez et al. [66] proposed using sodium hexametaphosphate as a particle dispersant. Second, it is impossible to register the weight in the first minute immediately after immersion, as the scale requires some time to stabilize. During the first readings, the absorption rate is at its highest because the sample is dry. The weight registered and used as a reference is always significantly higher than the former. Even a few seconds already have a significant impact on the initial reading. This inability to register the weight at time zero (i.e., immediately after immersion) affects the accuracy of the WA value obtained. The authors use this method to describe the kinetics of the WA, but the reported WA values are obtained using the EN 1097 standard [70].

This method and the ASTM C 128 [61] methods were both used in previous work [8] to determine the WA values of five FRCAs. The immersion method produced values between 68 and 222% higher than those from the ASTM C 128 [61] method. The authors explain these differences in light of the content of fines in the sample, which is consistent with air occlusion reported in [66].

2.3. Electrical conductivity method

This method is based on the relationship between the moisture content and the electrical conductivity. As dry aggregates are not effective electrical conductors, electricity can only be transported through surface moisture. Then, the method involves determining the electrical conductivity of an aggregate sample at multiple moisture contents. After plotting the relationship between both parameters on a semi-logarithmic graph, the relationship is approximated with two lines, one corresponding to the wet zone and the other corresponding to the dry zone (Figure 3 a). The intersection of the two lines defines

the SSD condition and, consequently, the WA value. More details on this method can be found in its standard procedure for soil [71] and previous applications to FRCA [8,33,71]. This previous work showed that the electrical method confirmed the WA values determined according to ASTM C128 [61] for two rounded FNAs, but it showed a potential significant underestimation with the ASTM C 128 method (between 33 and 50% lower than those values from the electrical conductivity) when applied for FRCA (with a high internal friction and fines content of 2.7-7.2%). In addition, from measurements performed by three different operators, the variations were reflected in a standard deviation of 0.06% for the electrical conductivity method, while for ASTM C128 [61] it was 1.21%. Kim et al. [33] analyzed the influence on the WA value of the shape and size of the container used in this conductivity method. The authors concluded that the shape and size of the container are of secondary significance concerning the measured WA.

2.4. Centrifugal method

This method was initially proposed for lightweight aggregates [73]. The variability for more than 25 operators in different laboratories was established at a value of 0.45%. In this method, a centrifugal force is applied to the wet aggregate sample while measuring the water released by the sample. The amount of water released depends on the pore radius, estimated considering a hemispherical meniscus according to the relation between radius and set parameter (Eq 3).

$$r=\sqrt{\frac{6\gamma cos\theta}{R\rho\omega^2}} \quad \text{(eq.3)}$$

where:

ɣ= surface tension

ω= angular velocity

r = radius considering a hemispherical radius

R = radius of the centrifugal rotation

Θ = angular velocity of the material

ρ = density of the water in the pores

When the centrifugal force equals the capillary force inside the pores of the aggregates, only the surface water and the water in the largest pores (> the r considered) are released. The centrifugal force is applied in a horizontal centrifuge. The speed is decided based on the relationship between the water release and the centrifugation time, so that it reaches a threshold value after 10 minutes at 2000 rpm [27]. This is the same speed proposed for lightweight aggregates [73], but in that case for only 3 minutes. The relation between time and wet mass is plotted, and when the curve becomes asymptotic, the SSD condition is assumed. When compared with the cone method, relative differences of less than 12% in WA of four FRCA were reporte [27].

Duan et al. [60] emphasized that the centrifugal force is applied in a fixed direction. Then, water in the pores facing other directions could be retained, leading to an overestimated WA value.

2.5. Continuous drying

This method was originally proposed for mineral admixtures [74]. The authors postulate that surface water does not evaporate at the same rate as water inside the pores. Based on the evaporation media porous theory, the authors define three different stages: the first stage is a transition phase in which the sample is heated, followed by a second stage of drying at a constant rate in which the surface water evaporates, and a third stage in which the drying rate decreases to a value corresponding to the drying of internal water. The transition between the two later stages is the critical point at which the SSD condition is achieved (i.e., the threshold point). (Figure 3 b). The method involves continuously weighting a wet sample while it is in an oven to construct this curve. Hlawatsch et al. [56] used this method to evaluate the WA of fine and coarse RCA with various sizes. The authors reported that the threshold point defining the SSD condition is easy to determine for samples with relatively monogranular particle size. The assessment is more uncertain for samples with a high content of fines or with multiple phases with very different drying rates. Moreover, Duan et al. [60] reported that by the time aggregates in the center of the sample reach the SSD condition, aggregates at the edge are already over-drying. In addition, Hlawatsch et al. [56] reported differences lower than 3% for FRCA when comparing the WA values from the cone and the continuous drying methods.

Based on the same principle as the drying porous media theory, Rueda et al. [62] used an electronic humidity analyzer to achieve the SSD condition for fourteen FRCAs. This setup uses infrared radiation to heat up the sample. The authors pointed out the difficulties in defining the transition point between the second and third stages, even when they used the moving average of three consecutive points to eliminate the noise. The reported WA values for the fourteen FRCA were similar to those of the cone method. An analogous method was proposed for CRCA [75], but using a microwave to heat up the sample. The authors also reported issues relative to the WA value obtained depending on the amount of sample tested and the vibration of the chamber. Contrarily, Yacoub et al. [22] did not report any significant issues in determining the threshold corresponding to the SSD condition. The authors reported WA values 42% higher than the value obtained with the cone method.

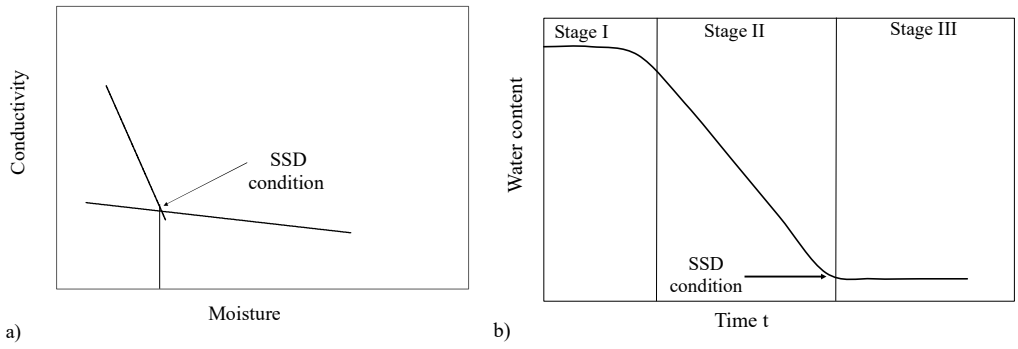


Figure 3. Curves of the a) conductivity method and b) drying porous media theory

2.6. Continuous drying by airflow

Dana and Peters [76] proposed a method using a rotating drum with slots through which a continuous current of warm air is injected. The sample is held in a 63-µm sieve to avoid loss of solids. The mass of the sample is registered at increasing time intervals, and the

mass loss is plotted over time. The SSD condition is the transition point between the second and third stages, according to the drying porous media theory (see Figure 3 b). Gentilini et al. [64] improved the setup by including temperature and humidity sensors to keep the warm air temperature constant during the test. The authors replaced the sieve with a fines collector to avoid mass loss during the test and plotted the drying gradient with respect to time to define the SSD condition. The drying gradient is defined by the temperature difference between the inlet and outlet of the air stream. Due to the evaporation of the surface water, the outlet temperature decreases with respect to the inlet temperature, and the gradient increases. The decrease in this gradient indicates the proximity to the complete evaporation of the surface water. Duan et al. [60] argued that this definition of the SSD condition could lead to underestimations of the WA. This is based on a potential delay in the threshold point of the drying gradient-time curve constructed based on temperature changes. In addition, the fines collected are not subjected to the air flow, and consequently, they contribute to the calculation of WA, but they are not involved in the same drying process as the rest of the sample.

2.7. Paper sheet method

The method involves observing traces of water on various colored paper sheets as a sample of aggregate is progressively dried. Once no trace of water is observed on the sheet surface, the aggregates are said to have achieved the SSD condition. This method is described by IFSTTAR [77] and has been applied to several FRCAs [12,26,53,78]. ASTM C 128 [61] suggests a similar methodology for aggregates with a high content of fines, but using a flat black surface instead of a paper sheet. There have been reports of differences between the WA value determined by this method and the one from the cone method of up to 187%, with an increasing gap trend with decreasing particle size [78]. The determination of the absence of traces of water involves the subjective judgement of the operator.

2.8. Volumetric flask method

This method was proposed for lightweight aggregates [62] and applied to FRCA [27]. The method involves placing a sample of FRCA in a volumetric flask, adding deionized water until reaching the mark on the flask, and then tilting 20 times or during 2-3 minutes, to eliminate the entrapped air [27,62]. Then, the flask is again filled up with water up to the mark, and the mass of the set is determined at 5 minutes. After 24 hours, the level of the water is restored, and with the additional water required to reach the mark, the WA of the aggregates is calculated as the amount of water added relative to the sample mass. An issue with this method is the 5 minutes it takes to register the first reading. In this sense, some authors have reported that the WA of FRCA in the first 5 minutes of immersion is already between 50 and 80% of the total WA [58,66,68]. Then, the first measurement registered after that time could contribute to underestimating the WA in this high range of relative values. Fernandez-Fajul et al. [79] used this method on various types of coarse aggregates, using glycerol instead of water to avoid the absorption of aggregates during the test. However, the authors reported that the absorption of NA using the glycerol to measure the initial volume was 0.5%, which was even higher than the WA determined with the EN 1097 [70] method. Duan et al. [60] reported that the impurities and bubbles can mask the meniscus at the surface of the liquid, making it difficult to judge the level of the liquid.

2.9. Extrapolation method

Zhao et al. [53] indicated that the cone and paper sheet [77] methods are not suitable for measuring the WA of FRCA containing fines of 0-0.63 mm size. For these particle size ranges, the authors estimate the WA for this fraction taking into account the WA value and composition (i.e., content of cement paste, CPc, and natural aggregates, NAc), of the size fraction above 0.63 mm. Then, the WA can be calculated according to Eq. 4. The coarse fraction serves as a proxy to estimate the WA of the cement paste, and the CPc in FRCA is determined by dissolution in salicylic acid. Other authors [12,26] proposed adaptations for Eq. 4 considering the mass loss at 475 °C or neglecting the WA of the NA (WA_{NA}). An issue with WA_{NA} determination is that these NA can be of different kinds, i.e., siliceous sand or particles from the fractured coarse aggregate contained in the source concrete. Then, the effectiveness of the WA estimation could be affected depending on the mineralogy of the coarse natural aggregates in the source concrete and its content in the coarser fraction of FRCA (normally higher than in the finer fractions that are composed mainly of cement paste). In addition, the variability of the extrapolated value magnifies the variabilities of the separate determinations and estimations for the WA of the coarse fraction, the WA of the cement paste, and the measurement of the paste content. As a result, the variability and sources of error of the method are, by nature, much larger than for the other, more straightforward methods.

$$WA = WA_{CP} \times CPc + WA_{NA} \times NAc \quad \text{eq (4)}$$

Where:

WA = water absorption

WA_{CP} = water absorption of attached cement paste

CPc= cement paste content

WA_{NA}= Water absorption of natural aggregate

NAc= Natural aggregate content

3. Methods not previously used on FRCA

The limited accuracy of the cone method is not exclusive to FRCA; it also applies to the determination of the WA of manufactured aggregates. Different methods have been previously proposed to try to solve the issue with manufactured sands. These could also be used for FRCA, but no information in this regard is yet available in the literature. Perhaps an important consideration is that these methods focus on solving the issues connected with the particle shape and fines content of secondary sands, but they do not specifically address the potential impact of attached cement paste in FRCA.

3.1. Colorimetric method

Khandal and Lee [55] proposed the use of a colorimetric method to determine the SSD condition of aggregates with a high content of fines. This method is one of the complementary methods considered in the aforementioned ASTM standard to overcome the difficulties in WA determination due to the presence of fines. The method involves immersing a sample of the aggregate in a 5 g/L cobalt chloride solution ($CoCl_2$). After 24 h, the sample is progressively dried, and when the surface moisture is lost, the surface of the particles turns blue, indicating the SSD condition is achieved. The method seems

mostly useful for angular and coarse sands that cannot be accurately tested with the cone method. It is possible that for challenging sands with a high content of fines, the color change is more difficult to appreciate for the finest fractions. As coarse particles can be dried faster, some issues associated with the homogeneity of the sample may arise.

3.2. Corelok test

This method was proposed by Hall [80] for testing blended aggregates with a specific device. The procedure involves determining the water displacement caused by a sample of dry aggregate introduced in a calibrated volumeter, which is used to calculate the dry density. Then, another sample of dry aggregate is vacuum sealed into a bag and then immersed in water in the device, and the mass of the sample is recorded. After that, the bag is cut, allowing the water to enter, the immersed weight of the sample is recorded, and the saturated density is calculated. Then, both densities are used to estimate the absorption of the aggregate. An issue with this method is that the determination of the dry density is subjective since the absorption of aggregates is happening while the weight is registered. Despite the fact that the method involves complex procedures, it does not show clear advantages in terms of accuracy over the other methods. For this reason, it was not considered in the experimental program of the present work.

4. Methodology

Based on the discussion on the various proposed methods and their principles, the most interesting methods were applied to assess the WA of four types of fine aggregate. These aggregates were natural siliceous sand (SS), manufactured quartzite sand (MQS), and two fine recycled concrete aggregates obtained from demolished old pavement (FRP) and from laboratory concrete specimens (FRL). A comparison among the WA values registered for each method and aggregate is included in the discussion of results.

Except for those cases in which it is specifically stated otherwise, the immersion and drying procedures, as well as the sample mass, followed the specifications outlined in EN 1097.

The physical properties of the four aggregates are shown in Table 1, photos and size particle distribution in Figure 4 (a and b respectively). Triplicate measurements were made for all tests and samples. The WA test methods were cone, immersion, electrical conductivity, centrifugal, continuous drying, paper sheet method, volumetric flask, and colorimetric methods. The test program does not include the method of continuous drying by air flow or the corelok test because they require complex setups not clearly justified by advantages over the other simpler methods.

Table 1. Physical properties of aggregates

Aggregate	Dry Density*	Content of fines (material ≤ 75 µm) (%)	Fineness Modulus	Compressive strength of source concrete (MPa)
SS	2.64	0.3	1.50	---
MQS	2.58	1.5	3.18	---
FRL	2.49	2.7	3.36	35
FRP	2.41	7.3	3.03	50

* Specific gravity according ASTM C 128

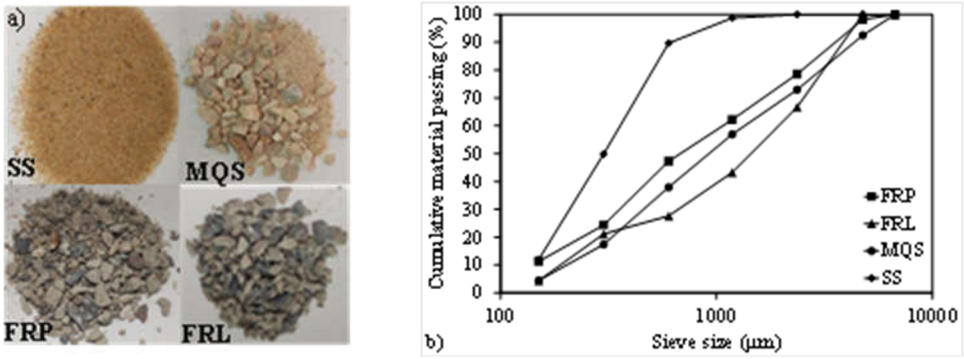


Figure 4. Aggregate features: a) pictures; b) particle size distributions

5. Results

5.1. Continuous drying method

Figure 5 shows the relative water content over time for a) SS, b) FRL, c) MQS, and d) FRP. In all cases, stabilization stage 3, as earlier explained, was achieved within an interval of 0-300 minutes. This naturally depends on the amount of free water in the sample at the beginning of the test, and it is not related to any property of the material. The drying was performed at (100±5) °C in an oven with forced ventilation. Some noise and breaks are observed in the registers, probably caused by vibration, a problem that was also reported by other authors [56,64,74]. Because of that, the determination of the threshold point is somehow subjective. In fact, taking into account variable threshold points within the deceleration period for the drying rate (outlined areas in the subfigures) already causes a significant variation of the WA value (Table 2). The reported WA values were computed as the midpoint within the range of values mentioned in each case. A better way to define the threshold point is needed to reduce the subjectivity in the computation of WA. Gentiline et al. [64] suggested the use of the moving average for a better determination of the threshold point. Although this procedure diminished the noise of the curve, the determination of the threshold point was not improved. Moreover, the homogeneity or heterogeneity of sample moisture was not possible to observe since this implies an interruption in the test.

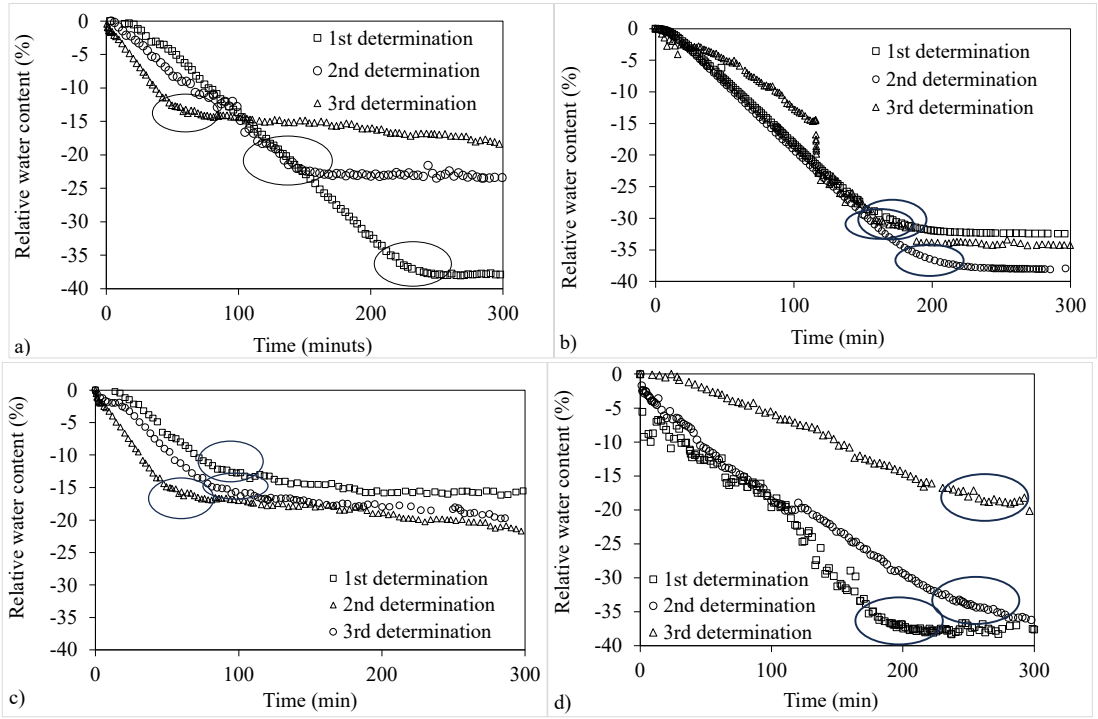


Figure 5. Continuous drying curves for a) SS, b) FRL, C) MQS and d) FRP

Table 2. Intervals for the WA values as determined from the plots relative water content versus time, for the continuous drying method

Aggregate	1st determination	2nd determination	3rd determination
	WA range (%)	WA range (%)	WA range (%)
SS	0.82 - 2.74	1.13 - 2.60	2.52 - 3.93
FRL	3.48 - 8.66	3.25 - 5.47	3.41 - 5.87
MQS	1.99 - 3.49	2.22 - 3.00	2.16 – 3.59
FRP	4.57 - 6.84	4.89 - 7.84	6.27 – 9.63

5.2. Colorimetric Method

In Figure 6, the progressing colorations of a) FRL, b) SS, c) FRP, and c) MQS with increasing drying from left to right are presented. To obtain sufficient intensity so that the change in color is more easily detectable, a higher $CoCl_2$ concentration of the solution (15%) was used than that one indicated in [55] . For FRL and FRP, the large particles turned blue earlier than the small particles. Therefore, an over-drying of large particles was necessary to achieve a homogeneous coloration. A consequent underestimation of WA can be expected. For SS and MQS, only some particles turned blue, while others remained the characteristic amber color of these sands. Moreover, the determination of the homogeneous blue color is subjective to the operator, and some agglomeration is evident as drying increases. Then this method does not represent an improvement compared to the cone method.

Figure 6. Colored aggregate samples for the colorimetric method for a) FRL, b) SS, c) FRP and d) MQS

5.3. Electrical conductivity method

For this study, 2.5 kg of sample was used. A prismatic container with right-angles was used, with dimensions of 10 cm long, 5 cm high, and 4 cm of spacing between electrodes. This shape slightly differs from the one used in [8], but such differences are not expected to have a significant impact on the results. Figure 7 shows the conductivity-moisture curves for a) MQS, b) FRL, c) FRP, and d) SS. For MQS and FRP, pictures of the shape obtained with the cone method at several points are included. When results from the electrical method indicated that aggregates were near the SSD condition, the corresponding shape for the cone method was still dubious (reaching or not the required shape for the SSD condition depending on the operator experience and the standard followed). While the condition was in many cases reached according to the description in ASTM C 128 [61] and NMX C 165 [81], it was still far from the one described in EN 1097-6 (Annex e) [70], NTC 238 [82], IRAM 1520 [83], and NTP 4000.022 [84]. In previous research, it was established that the differences between the electrical method and the cone method are related to the high content of fines and the angularity of particles, which lead to over-drying of the sample, so in the cone test procedure, it collapses and achieves the shape indicated in the standards [8,53].

Only three points in each of the dry and wet zones are required by the JSCE standard to construct the moisture content-conductivity relationship for soil. These seem insufficient in the case of the studied sands, and more than six determinations in each zone were necessary to obtain a reliable construction of the curve. Therefore, the several measurements needed to build the plot between moisture and conductivity become a

disadvantage of this method, requiring a higher mass of the sample and, hence, a high time and effort consumption.

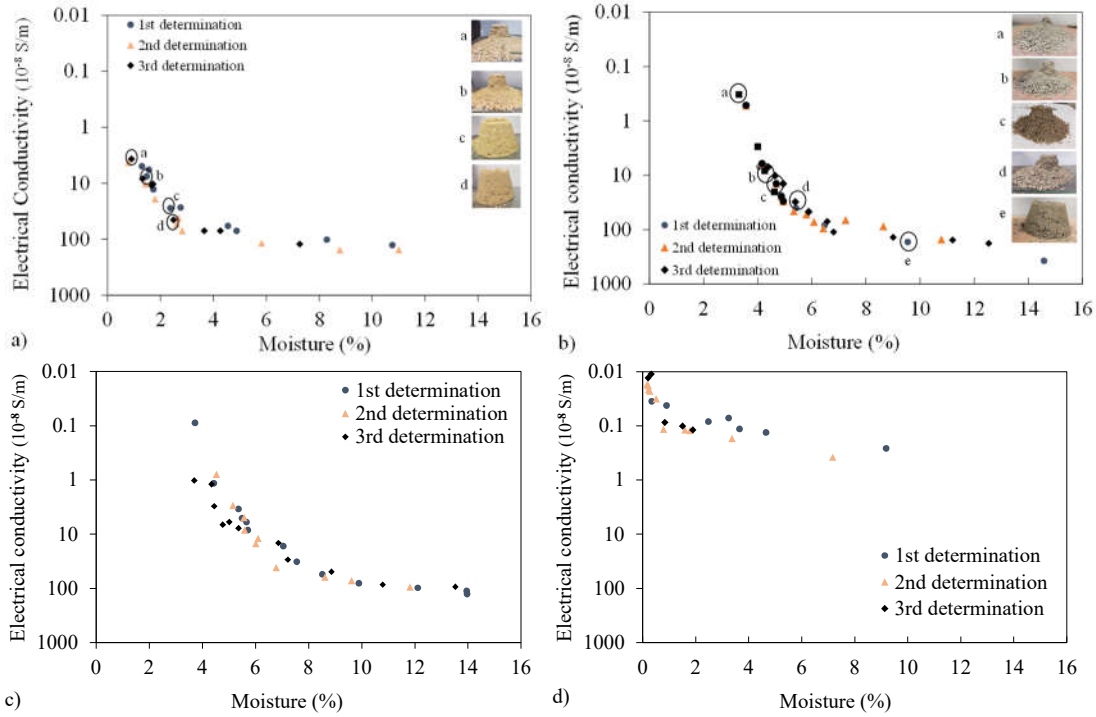


Figure 7. Curve moisture-conductivity from applying the electrical conductivity method for a) MQS, b) FRL, c) FRP, and d) SS

5.4. Paper sheet method

Figure 8 shows the paper sheet after the tests for a) SS and b) MQS. Moisture traces were clearly present on the paper sheet when the aggregates were notably wet. However, as particles only started to lose surface moisture, the traces on the paper were already hard to notice, even when visual inspection of the samples revealed notably moist surfaces. A lot of subjectivity by the operator is therefore involved, and the high variability in the SSD condition seems intrinsic to the method based on the visual inspection criteria. In search of an objective method to demonstrate the surface moisture, the cone method was applied when the samples had reached the SSD condition according to the paper method (last photo in Fig 8). The cone shape showed that there was surface moisture even for SS, for which normally the cone method is highly reliable to determine the SSD condition. Moreover, the fact that the shape of the cone for aggregate SS preserves even above the threshold moisture content as defined by the paper method clearly connects to the presence of surface moisture that gives cohesion among particles. It seems that the determination of the SSD condition according to the paper sheet method still requires additional research to achieve reliable and reproducible results.

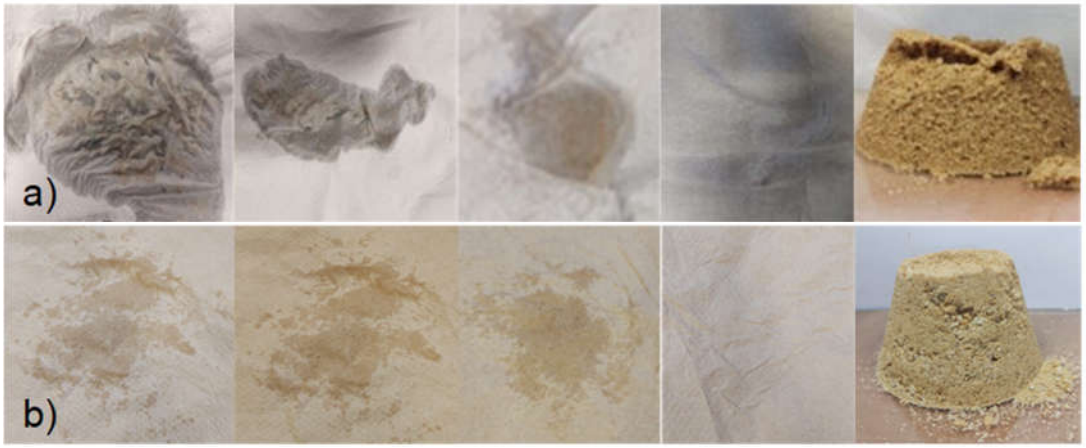


Figure 8. Paper sheet after test and respective cone test shape when not moisture trial is observed for a) SS, b) MQS.

5.5. Centrifugal method

Figure 9 shows a) the moisture-time curve from measurements done in a horizontal bowl centrifuge for all aggregates under study, and b) the cone shape for the moisture contents corresponding to the point where the asymptotic value in moisture-time curves was achieved. Contrary to results from [27,73], the moisture loss in the samples was not happening at a constant rate. There were rapid initial moisture losses, followed by more gradual reductions. A threshold point was not clear in the curves, as no abrupt deceleration of the moisture loss was happening. The centrifugation time used to estimate the SSD condition was 10 minutes, as recommended by [27]. Slight further moisture losses were noticed from 10 to 80 minutes of centrifugation, but in absolute terms, the impact on the computed WA value is very minimal, with the exception of MQS aggregate. From visual inspection and the correlation with the cone test (Figure 9b), it was very evident that the aggregates remained with a significant content of surface moisture even after 80 minutes of centrifugation. Comparable results were reported by [73] (although using a different centrifuge setup and aggregate types), with similar WA values for the paper sheet and centrifugal methods. From our analysis, it appears that both methods overestimate the WA of aggregates to a comparable degree.

In addition, the water recovered in the device had fine particles in suspension, which implies that a part of the solids in the sample was lost. This not only leads to inaccuracies in the WA calculation, but it also proves that the surface moisture present in the samples is even greater than detected (i.e., moisture content is partially masked by the loss of solids in the sample).

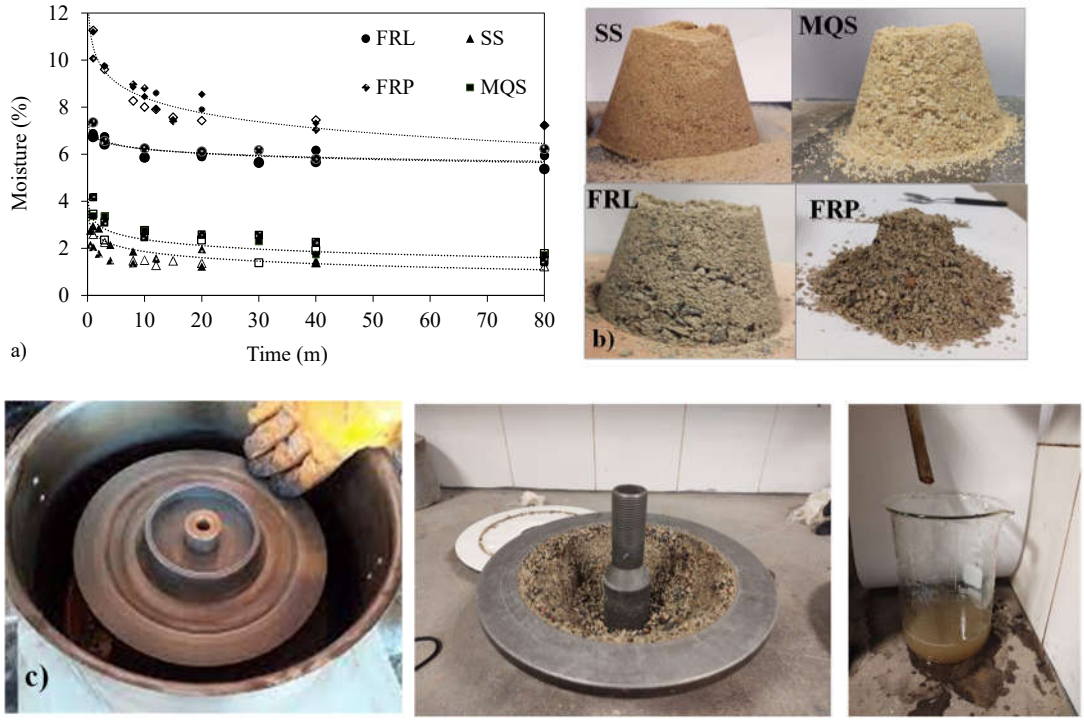


Figure 9. a) Moisture-time curve for the horizontal bowl centrifugal method, b) correlation with the cone shape in the cone method and c) Centrifugal set and water recovery

5.6. Volumetric Flask

As earlier mentioned, an unresolved issue with this method is the impossibility of having an initial reading at time 0, i.e., when the sample comes into contact with water. The first reading could be completed, on average, after 147 seconds (87 seconds spent filling the flask with water precisely up to the mark and 60 seconds spent releasing the bubbles). These first minutes are critical because the initial velocity of absorption is the fastest due to the highest humidity gradient. However, this amount of water cannot be computed in the WA as long as it cannot be measured. Another issue is the foam that forms at the meniscus due to bubbles and fine material. This foam makes it difficult to define the precise level of the liquid and causes a substantial source of error in the reading (Figure 10 a). To solve this issue, isopropyl alcohol was used to eliminate the foam (Figure 10 b). However, this prolonged the time required for the first reading up to 178 seconds, increasing even more the amount of water absorbed by the sample prior to the initial reading. Taking this situation into account, two methodologies were applied for MQS to estimate the relative benefit of using isopropyl alcohol. The initial reading was made before and after adding the isopropyl alcohol, with the first reading done at the level at the bottom of the foam. For the readings prior to and after adding the isopropyl alcohol, the WA values were 1.68% and 0.63%, respectively. Although the second methodology requires additional time to complete the first reading (with a greater impact on the reference ‘dry’ state), the certainty of the registry is greater. Although the time spent to take the first reading has a significant impact on the WA calculation, when isopropyl alcohol is used, the subjectivity of the operator decreases substantially. Thus, the second procedure was used in this study as it can provide more consistent results.

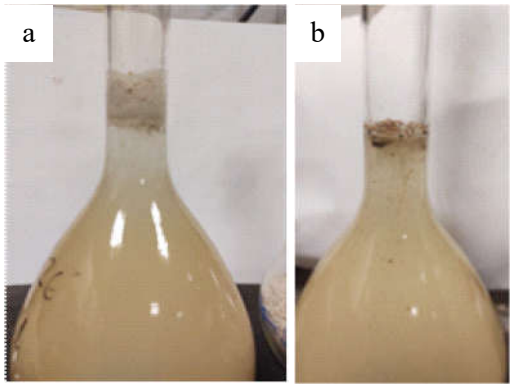


Figure 10. Foam in the volumetric flask method: a) after eliminating air bubbles in the bulk sample; b) after adding isopropyl alcohol

5.7. Immersion method

Figure 11 shows the absorption kinetics for FRP and MQS: a) within 24 h, and b) within the first 30 minutes. In this method, as for the volumetric flask, the first reading cannot be completed at time 0, as the scale needs a short stabilization period after hanging the sample. The average stabilization time spent in the experiments was 163 seconds. Before each measurement, a gentle stirring should be applied to allow the occluded air to be released. As reported by [66], the movement of the setup required to release the air bubbles produce immediate incoherent readings. In agreement with previous research [58,66,68], the first ten minutes after immersion showed the highest absorption rates, followed by a long stabilization period at a slow absorption rate. However, for the different readings and aggregates, the saturation degree at 10 minutes ranges from 60 to 95%. This ample range is slightly higher than the one reported in the literature of between 50 and 80% [57,68].

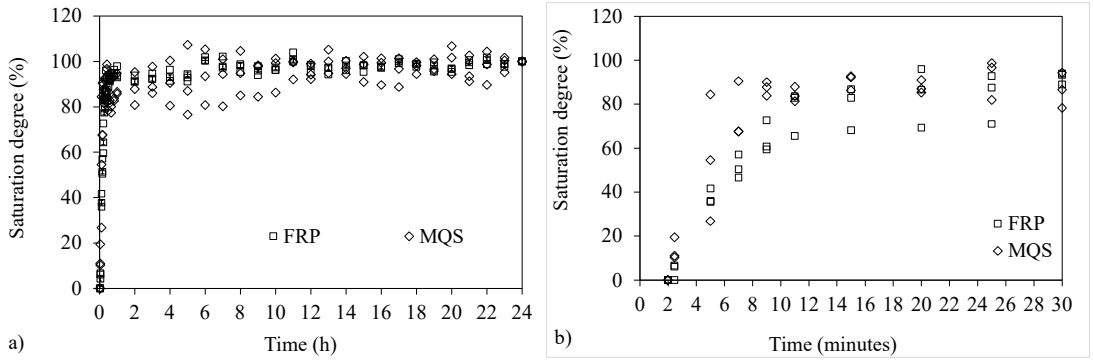


Figure 11. Absorption-time curve from the immersion method for FRP and MQS aggregates: a) measurements up to 24 hours; b) initial measurements up to 30 minutes.

Table 3 presents the WA values obtained with the formulas suggested by [58,69]. Using Eq. 1 only, values are significantly lower than those using the proposal from Leite (i.e., the average of Eqs. 1 and 2). In addition, WA values using solely Eq. 1 are more consistent with the outcomes from the other methods.

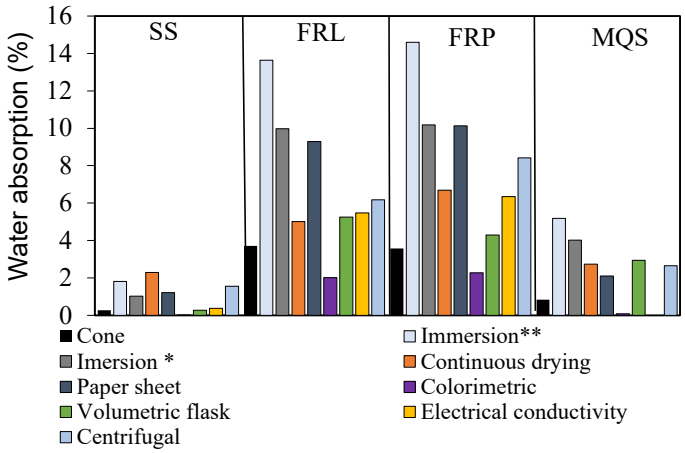
Table 3. WA values from immersion method using average of Eqs. 1 and 2 [58] and using only Eq. 1 [68]

	Computation method	WA Value (%)

Aggregate		1st determination	2nd determination	3rd determination
FRL	Average of Eqs. 1 and 2	11.38	16.24	13.32
	Eq. 1	8.76	11.32	9.83
FRP	Average of Eqs. 1 and 2	12.27	15.24	16.30
	Eq. 1	8.50	10.97	11.11
MQS	Average of Eqs. 1 and 2	4.25	5.18	6.14
	Eq. 1	3.28	4.07	4.67
SS	Average of Eqs. 1 and 2	1.21	2.08	2.16
	Eq. 1	0.91	1.05	1.12

5.8. Summarized water absorption results

Figure 12 shows the average WA for each method. Significant variations in the WA value can be observed across the different methods, even for the SS aggregate. For all methods, SS (natural aggregate) exhibits the lowest absorption values. However, the relative differences vary depending on each of the methods. The relative differences do not show a clear trend. The discrepancies in the WA values across the various methods may be attributed to the specific factors affecting each one of them.



* Calculation suggested by [69]; ** Calculation based on [58].

Figure 12. Average WA values from the studied methods.

Table 4 shows the standard deviation (SD) and the coefficient of variation (CV) for each method. Both parameters exhibit inconsistent variations without a discernible trend. For instance, the results of the immersion method have lower CVs for FRL and FRP, yet they are notably high for MQS and SS. The CV for the volumetric flask method is low for MQS aggregate but the highest among all the methods for SS, FRL, and FRP. The CV of the cone test is similar to those of other methods for SS, FRL, and FRP aggregates, but it is one of the highest for MQS. With the exception of the centrifugal method, CV shows high variation depending on the method and aggregate considered.

Table 4. Standard deviation (SD) and coefficient of variation (CV) for the various methods and aggregates evaluated; values are in percent points for SD and % for CV.

	SS	FRL	FRP	MQS

Methods	SD	CV	SD	CV	SD	CV	SD	CV
Cone	0.04	16.10	0.52	13.93	0.53	14.93	0.49	58.68
Immersion*	0.53	29.00	2.45	0.18	2.09	0.14	0.95	14.93
Immersion **	0.38	35.41	1.32	0.13	2.59	0.25	1.13	0.28
Continuous drying	0.81	35.41	0.92	18.42	1.15	17.2	1.15	0.42
Centrifugal	0.14	8.79	0.29	4.74	0.40	4.76	0.16	6.21
Colorimetric	0.17	14.19	1.05	11.3	0.96	9.43	1.83	86.7
Volumetric flask	0.14	53.30	2.59	49.35	2.75	64.07	0.12	4.07
Electrical conductivity	0.06	15.02	0.82	14.95	0.40	6.23	0.08	2.42

* Calculation suggested by [68]; ** Calculation based on [58].

6. Discussion

In the centrifugal method, it was not possible to determine the moisture threshold point. The centrifugation time of 10 minutes (as recommended by [55] to achieve the SSD condition) was achieving relatively stable moisture content for three of the four analyzed sands. However, after an extended centrifugation time of 80 minutes, surface moisture was visually detected in all the studied aggregates. Furthermore, the cone shape developed by the SS sample (i.e., the natural sand normally not giving issues for the cone method) also demonstrated surface moisture. The centrifuge force of the device was insufficient to eliminate the free water from the sample, with the possibility that the formation of interparticle menisci helps to retain water. This explains why this method seems to overestimate the WA still slightly, as shown in Figure 13 b). It is interesting to see that for the cases of MQS and FRL, the WA values obtained with the centrifugal method were still very similar to those obtained with the other non-deviant methods. A direct comparison between the obtained results and those reported in the literature is not possible because the features of the centrifuges differ from each other. It still appears that the setup features play a main role in the water absorption determination, even when the involved principles are the same.

One possible reason why WA values for the centrifugal method are similar to those of the other methods (despite the obvious presence of surface moisture) may be the loss of fines detected during the centrifugation. The increased weight due to the water excess due to surface moisture is (at least partially) compensated by the decreased weight due to the loss of fines. Both sources of error tend to compensate each other, but the fact that this is a random situation reduces the convenience of this method.

The conductivity method advantages the cone method, especially for very angular aggregates. This is because the former establishes a more objective definition of the SSD condition than the interpretation of the cone shape, which relies on the operator and standard considered. The CV and SD for the electrical method are comparable or lower than those for the cone test (see Table 2). In terms of practicality, even though the setup for the electrical method is easy to construct and low-cost, the procedure is time-consuming. The required amount of sample and the effort of the operator are considerably

greater than those in the cone method. The sample mass needed for the cone test is around 1 kg, while for this method it should be at least 2.5 kg. The Japanese standard [71], which is used for soil, establishes the need for at least six determinations (three in the dry zone and three in the wet zone) to construct the curve. However, these seem insufficient for fine aggregates, and at least 10 points are advisable. This is even more true as, at the moment of making the measurements, it is not possible to clearly preview whether the measurement is in the dry or wet zone. Moreover, if the measurements are too close to each other with little difference in moisture content (especially for the dry zone), the variations in the slope are very sensitive to small errors in the determinations, and the curve might not be sufficiently representative and affect the accuracy of the method. To solve this, a well-detailed procedure to acquire and interpret the data and specific training for the operator are required.

The colorimetric and paper sheet methods do not demonstrate improvements in terms of objectivity compared to the cone method. The three of them show significant operator subjectivity as they consist of a visual inspection of the sample to establish the threshold that defines the SSD condition. Moreover, based on the correlation between the result of the paper sheet method and the cone method for SS, it seems that the paper sheet overestimates the WA even for the least complex sample. Contrarily, the colorimetric method appears to underestimate the WA value since over drying is necessary to obtain a homogenous coloration.

The immersion and volumetric flask methods have the common issue that the reference initial state is not actually in the dry state but after a pair of minutes of exposure to the water. The time required for the first record can be as long as 4 minutes, during which the material has already absorbed water. This missed absorption is not contemplated in the calculation of the WA by these methods. Additionally, in the flask method, the fines floating on the surface of the meniscus make it difficult to define the liquid level, and this involves some subjectivity. Isopropyl alcohol can solve this last issue, but it lengthens the time required for the first record. Another issue common to both methods is the presence of occluded air. There is no full certainty that the stirring or shaking is able to completely eliminate bubbles from the sample. It has been reported that the remaining bubbles may cause a difference of around 30% for the WA of CRCA [49]. The effect is maximized for the fine fractions, as bubbles are trapped not only in the pores of particles but also in between particles. Furthermore, the shaking procedure to release air bubbles affects the reading on the scale, potentially causing incoherent values and increasing uncertainty about the result. By applying vacuum, the occluded air may be eliminated faster and more efficiently, and future research in this regard seems valuable.

The continuous drying method shows great potential for the true determination of the WA. However, the setup hanging down from the scale and over the oven is subject to vibration from the motor of the oven. This arrangement causes noise in the register. The uncertain determination of the threshold point needs to be improved since it can cause a high standard deviation and uncertainties about the WA measurement. The homogeneity of the moisture content across the sample (questioned already by [60]) may be improved by using a small amount of material to form a single layer of particles. The requirement for an exclusive oven and scale per sample for more than one day may imply a significant limitation for the adoption of the method.

The centrifugal method demonstrated a particularly low standard deviation for the WA values compared with the other methods. However, from the comparison with the shape of the cone test, it is clear that it is not suitable to achieve the SSD condition of aggregates. As was reported in the literature [73], similar WA values were obtained using this and paper sheet methods. Both demonstrate that there is still surface water for the condition indicated as corresponding with the SSD condition. Despite the expected overestimation with the centrifugal method, the loss of solids through the filter partially compensates for the result, reducing or eliminating these overestimations. However, this increases the uncertainty of the method.

The absorption capacity of sand and secondary sands is a very important aspect that still acts as a barrier to the adoption of circular practices in the construction industry. Secondary sands normally demonstrate higher WA values, but there is still uncertainty about the accuracy of the relevant determination methods. Our research seems limited to being able to recommend a single method, but it was able to reduce the list of methods of interest from 8 to 4. On this basis, two recommendations may be made:

- Literature describing the application of secondary sands characterized by one of the various WA methods must be considered with caution. This is especially important for the four methods that consistently demonstrate aberrant WA values: immersion, colorimetric, paper sheet, and cone method. From these, the most alarming is the conventional use of the cone method for the characterization of secondary sands with an angular shape or high content of fines. This is still the only standardized method that can be implemented by the industry nowadays. This leads to a second recommendation, as follows.
- Significant and strategic effort needs to be invested in one or more new WA methods for secondary sands with an angular shape and/or high content of fines. The cone method has proven to be unreliable, and four methods show sound grounds to be established as more appropriate standardized methods: centrifugation, volumetric flask, continuous drying, and electrical conductivity. The first two still show clear inconveniencies, such as the loss of solids (centrifugation) or the effort intensive and time-consuming elimination of air bubbles (volumetric flask). It seems convenient that efforts are concentrated on improving and standardizing one or two of these methods instead of designing new methods or creating additional fabrications to adapt the cone method.

7. Conclusions

From the analysis of various WA methods and their application to river sand, manufactured sand, and two types of fine recycled concrete aggregate, the following conclusions are drawn:

- The paper sheet and colorimetric methods demonstrate no practical advantages over the cone method, mainly due to their inability to address operator subjectivity in assessing the SSD condition. They also provide much higher values compared to the other methods.
- The centrifugal and paper sheet methods are prone to overestimating the WA. They indicate the SSD condition despite the obvious presence of surface moisture on all tested aggregates. This visual observation was corroborated for the natural

rounded sand by the cone method, which also detected surface moisture for those conventional SSD conditions.

- In the volumetric flask method, the determination of the accurate level of water is hard when no isopropyl alcohol is used. However, the main limitation comes from the impossibility of recording the initial measurement immediately after submerging the sample. The water absorbed by the sample in these first few minutes is one of the major sources of inaccuracy of the method. This is also a result of the significant effort needed to eliminate air bubbles in the mass of the submerged sample.
- The electrical conductivity and continuous drying methods highest the highest potential for accurately determining the SSD condition and computing the WA. Both of them need additional improvements. The continuous drying method needs improvements in the definition of the threshold point. This lack of precision can potentially represent a major source of inaccuracy in the WA value. The electrical conductivity method demonstrates its robustness in the determination of the WA. The effort, time consumption needed, and the number of points required to plot a curve that permits an easy definition of the intersection point are the main limitations of this method.
- The analysis of literature reporting the WA of secondary aggregates as determined by one of the various methods requires extreme caution. The inaccurate determination of the WA by unsuitable methods (including the standardized cone method) leads to biassed conclusions on the influence of secondary sands (e.g., manufactured sands, recycled sands from construction and demolition waste, recycled sands from excavated material) on the design and performance of mortar and concrete. The main concern is that the misestimation of the WA of the secondary sand makes it impossible to reliably estimate the effective w/c ratio in the cementitious mix.

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CRediT

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