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ORIGINAL ARTICLE

Measuring packing density and water demand of Portland cement and SCMs by the mixing energy method

Medindo densidade de empacotamento e demanda de água do cimento Portland e MCSs pelo método da energia de mistura

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Abstract: Wet packing methods evaluate the packing density of fine materials through the determination of the apparent density and voids content of pastes with different water to solids (w/s) ratios. Its goal is to estimate the minimum water demand to achieve the maximum solids concentration in the mixture, a parameter applied to the mix design of cementitious composites based on particle packing theories. Since most methods based on apparent density are time-consuming and require a high volume of materials, this paper aims to evaluate the mixing energy method as an alternative for the wet packing method and to adapt it to be used for SCMs (supplementary cementitious materials). With a reduced time and material to perform the test, results demonstrate a better precision of the mixing energy due to its discrete measurement. The ideal water flow and initial volume of materials to perform the test on cement and SCMs are discussed.

Keywords: particle packing, fine powder, compressive packing model, Arduino.

Resumo: Os métodos de empacotamento via úmida avaliam a densidade de empacotamento de materiais finos através da determinação da densidade aparente e teor de vazios de pastas com diferentes relações água/sólidos (a/s). Seu objetivo é estimar a demanda mínima de água para atingir a concentração máxima de sólidos na mistura, um parâmetro aplicado à dosagem de compósitos cimentícios utilizando teorias de empacotamento de partículas. Como a maioria dos métodos baseados na densidade aparente são demorados e requerem um grande volume de materiais, este trabalho visa avaliar o método de energia de mistura como uma alternativa ao método de empacotamento via úmida e adaptá-lo para ser usado para MCSs (materiais cimentícios suplementares). Com um tempo reduzido e menor quantidade de material realizar o teste, os resultados demonstram uma melhor precisão do método da energia de mistura, devido à sua medição discreta. O fluxo de água ideal e o volume inicial de materiais para realizar o teste em cimento e MCSs são discutidos.

Palavras-chave: empacotamento de partículas, pó fino, modelo de empacotamento compressível, Arduino.

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1 INTRODUCTION

Concrete is a building material made from a mixture of aggregates of varied sizes embedded in a binding matrix (cement paste) that is responsible for filling the voids between the aggregates [1]. According to this concept, the

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concrete mix design can be optimized based on particle packing theories to achieve more sustainable mixtures by reducing Portland cement consumption or replacing it with supplementary cementitious materials - SCMs [2]. In particle packing theory, particle fractions of different sizes are used to fill the voids between the larger particles generating a system with a reduced voids volume [3]. The water added to the cement will fill the voids between the particles and the excess water will lubricate the surface of the particles, giving the desired flowability to the mixes [4].

To design concrete based on particle packing theories, the packing density of Portland cement and other granular materials is one of the main parameters required for the application of several particle packing models such as the Furnas [5], Stovall et al. [6], and De Larrard [7] models. Over the years, several methods have been proposed to determine the packing density and/or water demand of fine particles, as shown in Table 1.

Table 1. Packing density methods available for Portland cement and fine materials.

Method	Evaluation criteria	Reference
Water demand France	Visual assessment of paste consistency	[7]
Water demand Germany	Visual assessment of paste saturation point	[8] apud [9]
Proctor test	Oven-dry humidity in compacted mixture	[10], [11]
Centrifugal consolidation	Excess water after centrifugation	[12], [13]
Water demand – Japan	Slump flow test results	[14]
Rheology – Krieger and Dougherty	Viscosity of the mix	[15]
Vicat test	Needle penetration	[16]
(Gas) pressure filtration	Pressure variation	[17]
Wet packing method	Apparent density of the paste	[11]
Water demand / mixing energy	Energy consumption during the mixture	[18] apud [9] [19] apud [20]

It can be observed in Table 1 that all the methods presented consider the presence of water for the measurements. Measuring the packing density of fine particles such as Portland cement in dry systems is a difficult task due to the agglomeration of particles smaller than 100μm [21]. In very small particles, due to their low mass and high surface area, Van der Walls forces, electrostatic charges and chemical bonds are higher than the forces that can separate the particles (shear gravity), and this effect causes the particles to agglomerate [22]. This determination on fine powders needs to be performed in the presence of water and superplasticizers to ensure powder dispersion [23]. Hence, dry packing methods such as [24]–[26] are not advised, because they tend to overestimate the voids content and underestimate the packing density of fine particles [11]. Furthermore, when these fine particles are used to produce concrete, water and chemical admixtures will be present in the mix, which makes measuring the packing density under these same conditions a more reliable parameter to be used for concrete mix design.

Most of the methods given in Table 1 have been evaluated by Fennis [9], who states that all tests give reasonably accurate results (the standard deviation obtained in each test is low), although they do not all comply with each other since the different techniques will lead to different values for the packing density. Therefore, studies comparing different methodologies are fundamental to prove the effectiveness and reliability of those techniques and their adaptations. Fennis also suggested that the water demand test from France [7], [27] and mixing energy test [18] are the ones that produce better results of packing density for powders. However, the water demand test from France is based on visual analysis, which makes the results highly dependent on the operator. The centrifugal consolidation test [28], [13] is also an accurate method, according to the author [9], as long as enough measurements are performed (at least three). Nevertheless, the centrifugal consolidation test demands special equipment for test performance, which is not commonly found in construction materials laboratories. Furthermore, the compaction energy is limited to the centrifugal energy used. The wet packing method [11] has been successfully used for measuring the packing density of cement pastes with a wide range of w/s ratios [29], [30] and it has also been successfully applied to the design of eco-friendly concretes [31]–[33] using particle packing theories, resulting in mixtures with low cement consumption.

The advantage of the wet packing method [11] relies on the use of regular laboratory equipment, and the application of similar conditions to which the concrete will be exposed during mixing and compaction, such as the use of chemical admixtures and the casting method (compacted, vibrated or gravity). On the other hand, it requires several pastes to be produced to obtain the results, which can be a downside regarding both time and the amount of material required for testing. This can become a concern when a combination of several binders and supplementary cementitious materials needs to be evaluated repeatedly. It is also not viable to produce the mixtures for the wet packing method in small batches, since the authors recommend using a minimal paste volume of 181ml or similar for the test. Since changing the mixing procedure could influence the results, its adaptation requires further investigation.

The mixing energy method also has the advantage of not relying on human judgment for the results to be obtained. Plus, it requires less material to run the test. Although the method allows a discrete measurement and high precision, its application depends on extra equipment to perform the energy monitoring of the mixer. Some low-cost approaches such as the use of a PC-connected multimeter and programmable microcontrollers are possible options, which will be used in the present work. Additionally, for the application of the mixing energy method for SCMs, adjustments in the procedure may be required.

The purpose of the present work is to compare the mixing energy method as an alternative to the wet packing method to measure the packing density and water demand of Portland cement. Additionally, the mixing energy method was adapted to be used for SCMs (supplementary cementitious materials). The monitoring of power consumption was successfully performed with a low-cost system attached to the standard mixer. Both wet packing and mixing energy methods will be further explained and detailed in the following sections, as well as the proposed adaptations.

From the proposed methods, a faster evaluation of the packing density of Portland cement and other fine materials is possible. Therefore, the development of eco-friendly mortars and concretes can be achieved based on particle packing theories, allowing a reduction in cement consumption, optimizing the use of SCMs, reducing carbon footprint, and increasing the durability of composites.

2 EXPERIMENTAL PROGRAM

2.1 Materials

In the present work, pastes were mixed using a single binder (Portland cement or SCMs (fly ash, limestone filler, metakaolin and densified silica)), with chemical admixture and water. The specific gravity of the powder materials was evaluated according to ASTM C188 [34].

A Brazilian high early strength Portland cement (CP V – ARI) [35] was used. The cement is equivalent to ASTM type III Portland cement [36]. All the SCMs used were commercialized in Brazil for concrete production. The particle size distribution of the materials was obtained by laser diffraction in a particle size analyzer (CILAS 920) using an 850 nm diode laser. The dispersion of the materials was performed by dissolution in water without any dispersing agent and ultrasound for 60 seconds during testing [3], [37]. For the densified silica, due to its smaller size, the particle size distribution was obtained by the Dynamic Light Scattering (DLS) technique (Microtrac Nanotrac), using a 785nm laser in reflected measurements. The dispersion was performed by dissolution in distilled water with 5% dispersant to the solvent volume (polycarboxylate base). The sample was diluted at a ratio of 2mg/ml in 50ml of the solution and dispersed in an ultrasonic washer (Schuster L-100, 160W, 42000 Hz) for 15 minutes.

A third-generation superplasticizer chemical admixture composed of polycarboxylate polymers was also used. The chemical admixture meets the requirements of ASTM C1017 [38] and presented a 1.12 g/cm^3 specific gravity.

2.2 Particle size distribution and specific gravity

The particle size distribution (PSD) of Portland cement and SCMs can be found in Figure 1. The specific gravity and characteristic average diameters (D_{50}) of the materials are presented in Table 2.

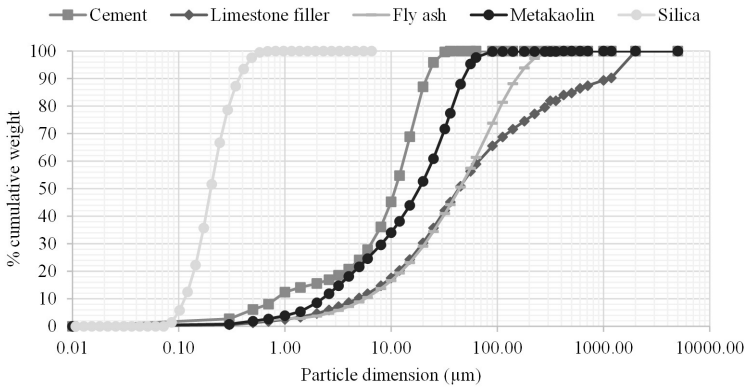

Figure 1. PSD of cement and SCMs

Table 2. Specific gravity and average diameter of fine powders

Material	Specific gravity (g/cm³)	D_{50} (µm)
Portland cement	3.13	10.99
Fly ash	3.00	44.14
Limestone filler	2.78	43.55
Metakaolin	2.49	18.45
Silica	1.97	0.201

From the results presented in Figure 1, it can be noticed that all materials have a continuous distribution. Based on their PSDs, fly ash and the limestone have particles sizes with similar average diameter, while the silica presented the smaller average diameter. Additionally, the limestone filler presented about 20% of retained material in the 355 µm mesh sieve, hence with some coarse particles. Along with the particle morphology, those physical properties will directly influence the obtained results of water demand and packing density.

2.3 Mini-slump test

The saturation dosage test for the superplasticizer was performed using Kantro's cone test [39] for the Portland cement, by measuring the paste spreading diameter in the fresh state. During the test, the w/s ratio was kept constant at 0.3 for all mixes, and chemical admixture contents tested were 0.25%, 0.5%, 0.75%, 1.0%, 1.5%, 2.0%, 2.5% and 3.0% (by cement weight). The saturation point found was used in all mixes, including the SCMs. Due to the small particle size, the SCMs may demand higher dosages of chemical admixtures, but large dosages may result in a mechanical behavior different from when realistic dosages are used in the field [40].

The results of the chemical admixture saturation point test with Portland cement obtained in the Kantro test are shown in Figure 2. The pastes produced without chemical admixture did not provide the required consistency for the test, therefore, its results were considered zero.

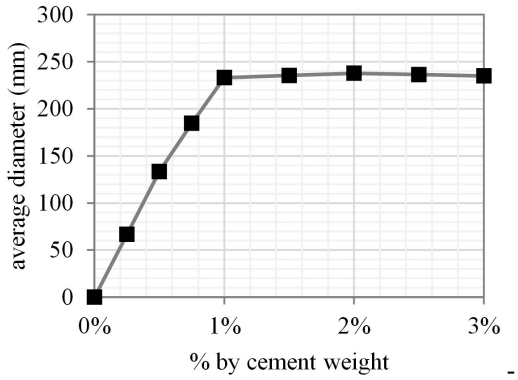

Figure 2. Superplasticizer saturation dosage test results

It can be observed in Figure 2 that the opening diameter obtained in the Kantro test increased significantly when the admixture content was increased up to 1.0%. For higher contents of the admixture, no significant increase in the fluidity was observed, although the manufacturer suggests ratios up to 5.0%. Therefore, 1.0% of the chemical admixture by cement weight is the saturation dosage for the superplasticizer tested and it was used to perform all the packing density tests in the present study. The saturation point of 1% by mass found was used in all mixes henceforth, including the SCMs mixtures.

2.4 Wet packing method

The first test performed to determine the packing density of Portland cement followed the procedures proposed by Wong and Kwan [11]. The method consists of producing cement pastes with different w/s ratios and determining their apparent density. The water-to-solids (w/s) ratio of the first cement paste produced was 0.35. After that, several other mixes were produced with smaller w/s ratios, which varied from 0.35 to 0.13, by weight. It corresponds to 1.08 to 0.40, by volume.

The mixing method consisted of initially adding 50% of the cement to the mixer, followed by 80% of the water and the chemical admixture, mixing at low speed (140 rpm) for 3 minutes. Then, the remaining materials were added in 4 equal parts, mixing at low speed for 3 minutes after each addition. After the mixing process, the paste was placed in a cylindrical container in three layers, each layer being rod-compacted 30 times. Finally, the paste weight to fill the container was registered (the apparent density measurement) and the solids concentration and the voids ratio were calculated according to Equations 1 to 3.

$$V_s = \frac{M}{p_w.u_w + \sum_{i=1}^{n} p_i.R_i} \quad (1)$$

$$\mathrm{u} = \frac{(V - V_s)}{V_s} \quad (2)$$

$$\phi = \frac{V_s}{V} \quad (3)$$

Where: V_s is the volume of solids in the mix (cm^3); M the mixture mass (g); p_w the water density (g/cm^3); u_w the w/s ratio, by volume (-); p_i the material i specific gravity (g/cm^3); R_i the material i volume in relation to the total solids volume (-); u the voids ratio (-); V the container volume (cm^3) and; ϕ is the solids concentration (-).

The test was repeated following the same procedures previously described, but the volume of the containers used for the apparent density measurements was changed to 200ml. The original test procedure used a container with 181ml, and it is indicated that the paste volume produced should be 50% higher than the container's volume. A regular mortar mixer (140 rpm) was used, and the test was performed twice for each w/s ratio. A summary of the mixing procedure is presented in Table 3.

Table 3. Test conditions evaluated for the wet packing method.

Mixing speed	**140 rpm**
Container	200ml
w/s ratio (by mass)	0.13 – 0.15 – 0.17 – 0.20 – 0.25 – 0.35
Mixing procedure	50% cement + 80% water + 100% superplasticizer Mix for 3min Add 4x (12.5% cement + 5% water) and mix for 3 min

Using several w/s ratios for testing through the wet packing method is necessary to reproduce the different conditions of grain-water interaction in mixtures, as presented in Figure 3. From Figure 3c it can be observed that when the w/s ratio is high, there is water between the particles, resulting in a high voids ratio. This situation is associated with a saturated mixture, where the particles are spaced apart from each other by excess water, hence the mixture presents a high fluidity. By reducing the w/s ratio, the voids ratio decreases, and the funicular state is reached as shown in Figure 3b. In this state, the granular particles remain surrounded by the water, but the water is not enough to fill all the voids between the particles, which will approximate the grains because of the capillary forces and generate the minimum voids ratio. When the voids ratio is minimum, the solids concentration is maximum, which corresponds to the packing density of the material in the wet condition. It is important to notice that under this circumstance the voids ratio cannot be further reduced, since the particles are already in contact with each other. If the w/s ratio is decreased beyond this point, the system enters the pendular state as presented in Figure 3a, when the amount of water is not sufficient to wet all the grains, forming ‘‘water bridges’’ in the points of contact between particles. The voids ratio will be increased in this situation and the particles will be pushed away from each other due to the water surface tension [17], [41], [42].

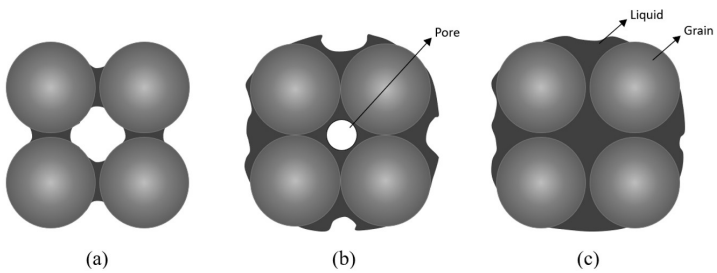

Figure 3. Different conditions of grain-water interaction in mixtures: (a)Pendular state, (b) Funicular state and (c) Saturated mixture.

2.5 Mixing energy method

The packing density and water demand can also be evaluated by monitoring the energy spent to mix the paste, as proposed by the mixing energy method ([18] apud [9]; [43] apud [20]). When a mix is being produced and water is added slowly to the mixer bowl, the paste goes from the pendular to the funicular state, and if water keeps being added, the saturated state is reached, as described in Figure 3.

Thus, when performing the test, initially a small amount of water is added to mix the dry paste and low power consumption is registered since the mix is not yet homogeneous. By adding water to the system, it becomes more difficult for the mixer blade to rotate and homogenize the paste because even though more water was added, the mix is still dry. When the funicular state is reached the energy consumption is expected to be the maximum obtained since the mixture will reach higher shear stress due to the particle's proximity [20]. After this point, it is expected a decrease in power consumption due to the increase of the water layer surrounding the particles. Therefore, the packing density and the minimum water demand occur when the highest power consumption is registered, at the funicular state. With the increase in the amount of water, the thickness of the water layer surrounding the grains increases, pushing the particles away from each other, resulting in a reduction of the shear stress. A typical graphical result obtained in the test is given in Figure 4.

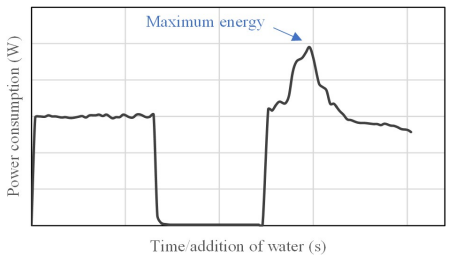

Figure 4. Representation of the mixing energy test result, with increasing energy consumption as a function of water addition over time

The test was performed twice, and the packing density was calculated by Equation 4 [44]. Where: ϕ is the solids concentration (-); V_s is the volume of solids (cm^3); V_a is the admixture volume (cm^3) and u_w is the w/s ratio, by volume (-), at the maximum power consumption.

$$\phi = \frac{V_s}{V_s + V_a + u_w} \tag{4}$$

The determination of the water demand by the mixing energy method ([43] apud [20]) was performed considering some adaptations. Fennis [9] suggests the use of a 1500g of cement along with 264g of water and superplasticizer chemical admixture, resulting in an initial w/s ratio of 0.176. After mixing for 60 seconds and an extra 60 seconds interval without mixing (resting and scraping), a constant water addition of 1.5 ml/s occurs using an intravenous (IV) drip while mixing until liquefaction of the mix. For the continuous additions of water, the IV drip was attached to the mixer as shown in Figure 5a and 5b, and placed as close as possible to the paddle center. The water drops should not touch the bowl, to ensure a proper incorporation of the water. The flow regulator was previously adjusted for the required water flux.

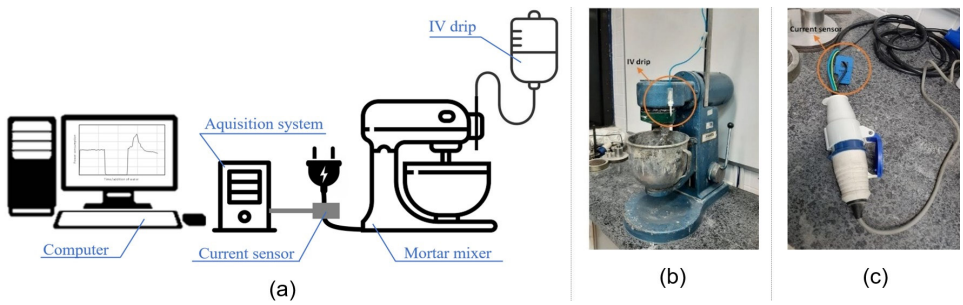

Figure 5. Mixing energy test: (a) set up schematic illustration (b) IV drip flow regulator and (c) current sensor

During the experiment, the mixing energy test was also performed reducing the cement to 1kg due to the high torque of the mixer that was causing paddle locking and energy peaks. Two water additions, 1.5 and 0.5 ml/s were also evaluated by the authors since a reduction in water flow will facilitate the energy peak identification. Hungers and Browers [20] suggest using 10 ml additions every 20 seconds, since there is a delay in the water effect when continuously added, hence, this water addition was also tested. Table 4 shows a summary of the mixing energy adaptations performed in the present study. The test was performed twice for each water addition.

Table 4. Adapted mixing energy test procedure

Initial w/s ratio	**0.176 (0.55 in volume)**			
Cement consumption	1.5 kg		1.0 kg	
Water addition rate	1.5ml/s	1.5 ml/s	0.5 ml/s	10ml/20s

During the mixing process, the current was monitored every 0.1s using a low-cost system with the aid of an open-source electronic prototyping board. For this, an Arduino Nano with SCT013 current sensor (±1% accuracy within 0-100A range) was used on the mixer power supply connections (single phase wiring), as shown in Figure 3a and 3c. Ohm's law was used to calculate the equivalent power consumption during the test. The use of sensors attached to Arduino systems has been successfully used in recent published cementitious composite studies [45]–[51].

For the proper application of the test procedure, it is essential to use a standard planetary mixer according to ASTM C305 with controlled rotation speed. In order to keep a constant rotation speed, a build-in frequency inverter in the equipment will adjust the applied current over time. Therefore, a variation in the measured current is expected when water is added. After establishing the reliability of the mixing energy method when compared to the wet packing method developed by Wong and Kwan [11], and the water addition of 0.5ml/s in 1.0kg of cement as the best setup for the test procedure, the method was applied to SCMs with some adaptions on the initial volume and water ratio were established experimentally during testing.

The initially proposed mass of fine material, 1 kg, proved to be inadequate to perform the method in silica fume. This mass presented a very large volume, inadequate to the capacity of the mixer bowl, and for this reason, the initial mass of silica fume was reduced by 25%. For the other materials, the initial mass of 1 kg allowed the normal performance of the test.

The initial water/solids (w/s) ratio was also adjusted to meet the particularity of each material analyzed. The adjustment on the initial volume of dry material and water/solids (w/s) for SCMs was required to prevent the mixer paddle from jamming and avoid potential dead zone problems during mixing, which may affect mixing curves. For metakaolin and silica fume, the ratio initially proposed was low, which resulted in a very long testing time. Thus, the water/solids ratio was increased for these mixtures to allow an adequate test time. For the limestone filler, the ratio originally proposed produced a very fluid mixture (with excess water) and was reduced by 50%. Table 5 summarizes the parameters used for the tested SCMs.

Table 5. Parameters used for each material when performing the mixing energy test

Material	**Initial dry mass (kg)**	**Initial w/s ratio (weight)**
Portland cement	1.00	0.176
Limestone filler	1.00	0.088
Fly ash	1.00	0.176
Metakaolin	1.00	0.264
Silica	0.75	0.606

To assess whether the parameters are adequate, the test results (water demand) between two consecutive measurements, and the increase in power consumption over time should present similar behavior among the tests, indicating a good dispersion of the water in the mixture.

3 RESULTS AND DISCUSSIONS

3.1 Wet packing method

The packing density of the cement was first determined by the wet packing method [11]. The voids ratio (u) and the solid concentration (ϕ) of the cement pastes are shown in Figure 6.

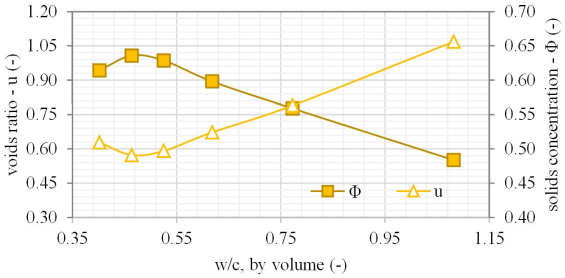

Figure 6. Void ratio and solids concentration of Portland cement

The maximum solid concentration is considered equal to the packing density of the cement particles in the wet condition. The value of packing density found was 0.636 and the minimum w/s ratio needed to produce a homogeneous paste (needed to reach the highest solids concentration) was 0.464 (by volume) or 0.150 (by weight).

3.2 Mixing energy method

3.2.1 Portland Cement

The results of the mixing energy test with different setups are given in Figure 7. It can be seen in the graphics an initial energy increases relative to the starting of the equipment torque at time zero. After this initial moment, energy consumption remains constant for the first 60 seconds of the mixture, while the amount of water is constant (w/s ratio equal to 0.176, as previously stated in Table 3). After that, the mixer is turned off for 60 seconds, during which there is no record of power consumption, and the amount of water remains unchanged. After this period the bowl is scraped, the mixer is turned on again and water is added according to Table 3. With the addition of water in the mix, there is an increase in the shear stress as the mix goes from the pendular to the funicular state and, consequently, an increase in the power consumption is observed. At the funicular state, the maximum energy consumption is reached when all particles are in contact with each other, and their surfaces are wet and connected by water films [20]. By adding more water, the particles start to move away from each other due to the increase of the water layer around them (saturated state), hence, the shear stress and the power consumption decrease. The test stopped after the power consumption dropped and reached a stable condition.

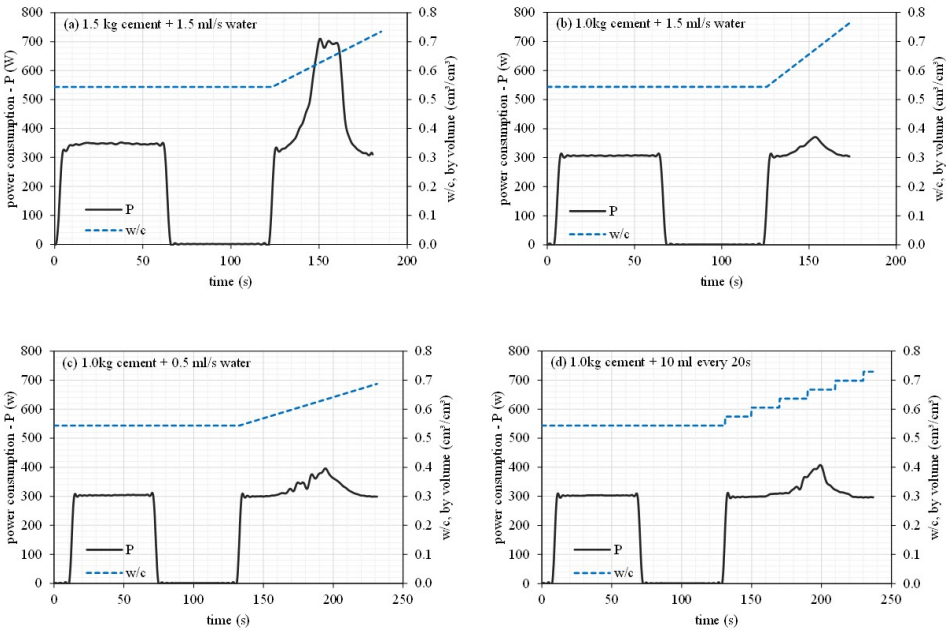

Figure 7. Mixing energy test results with a water addition rate of: (a) 1.5 ml/s with 1.5 kg of cement, (b) 1.5 ml/s with 1.0 kg of cement, (c) 0.5 ml/s and (d) 10 ml every 20 s

Based on the results shown in Figure 7, it is possible to observe that the Arduino Nano with SCT013 current sensor was able to perform a discrete measurement of the power consumption with good precision. From the obtained data, the use of 1.0 kg of cement reduced the power consumption during the initial mixing by 13% and also reduced the energy peak at the funicular state, as can be seen when comparing Figure 7a and Figure 7b. This reduction allowed a better measurement, since the use of 1.5 kg of cement generated power oscillations at the peak due to the high shear stress of the paste, while 1.0 kg of cement prevented the paddle jamming and promoted better data acquisition. While the wet packing method evaluated the water demand for every 0.05 w/s interval, the mixing energy test allowed the estimation of the water discretely every few milliliters of water, hence, with better precision.

Using 10 ml additions every 20 seconds generated an asymmetrical energy peak, as seen in Figure 7d, and the water effect became harder to identify. Besides, errors in the water additions could have happened due to the difficulty of adding small amounts of water precisely every 20s. Table 6 shows the packing density and water demand calculated using the mixing energy method.

Table 6. Packing density and water demand results obtained from the mixing energy method

Method for adding water to the mix	Packing density (-)	Minimum w/s, by volume (-)	Minimum w/s, by weight (-)
1.5 ml/s with 1.5 kg of cement	0.604	0.628	0.203
1.5 ml/s with 1.0 kg of cement	0.574	0.700	0.227
0.5 ml/s with 1.0 kg of cement	0.597	0.634	0.205
10 ml/20 s with 1.0 kg of cement	0.591	0.652	0.211

The packing density results shown in Table 6 varied from 0.574 to 0.604. The original mixing energy method performed with 1.5kg of cement and 1.5ml/s water additions presented a higher packing density (Φ=0.604). When the amount of cement was reduced from 1.5kg to 1.0kg, the packing density was reduced by 5%, while the w/s ratio increased to 0.700 (by volume), possibly because of the high-water flow used in a smaller amount of cement. Reducing the cement to 1.0kg and water additions to 0.5ml/s did not affect the results (variations below 1% from the original method). While the test performed with 1.5kg and 1.5ml/s took 26s to reach its peak, the test with 1.0kg and 0.5ml/s increased that time to 65s. This increase in time to reach the energy peak allowed a better estimation of the energy peak position and gave more time for the added water to interact with the cement, reducing any delay in its effect - mentioned as a downside of the method by Hunger and Browers [20]. Regarding the addition of 10ml every 20s, this setup presented similar packing density results to the original method, but increased the water demand by 4%, possibly due to the asymmetrical behavior of the energy consumption and delay in the water effect. A comparison of the results obtained from the wet packing and the mixing energy methods is illustrated in Figure 8.

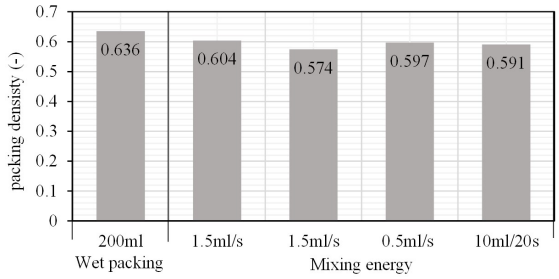

Figure 8: Packing density with the wet packing and the mixing energy methods

When observing the results presented in Figure 8, it can be noticed that both methods were able to measure the packing density of the cement pastes with a small deviation between the test results. The slightly higher packing density results obtained from the wet packing method are a consequence of the compaction step applied when measuring the apparent density. It induces higher energy to the pastes and, therefore, particles get closer to each other due to compaction. It also reduces the water demand, since the volume of water between the grains is smaller. When producing mortars and concretes this compaction energy is different from those used during casting, hence, the mixing energy method probably allows a better estimation of both the packing density and the water demand, since a discrete

measurement is performed, and every water addition can be evaluated during the mixing process. The mixing energy method also demands a smaller volume of material to perform the test. A reduced time to perform the mixing energy test is also positive since one mix is needed for the measurements, while the wet packing method requires at least five.

3.2.2 Supplementary cementitious materials

Figure 9 shows the results of the mixing energy test performed on the SCMs, and Table 7 presents the summary results of the packing density and water demand for those materials.

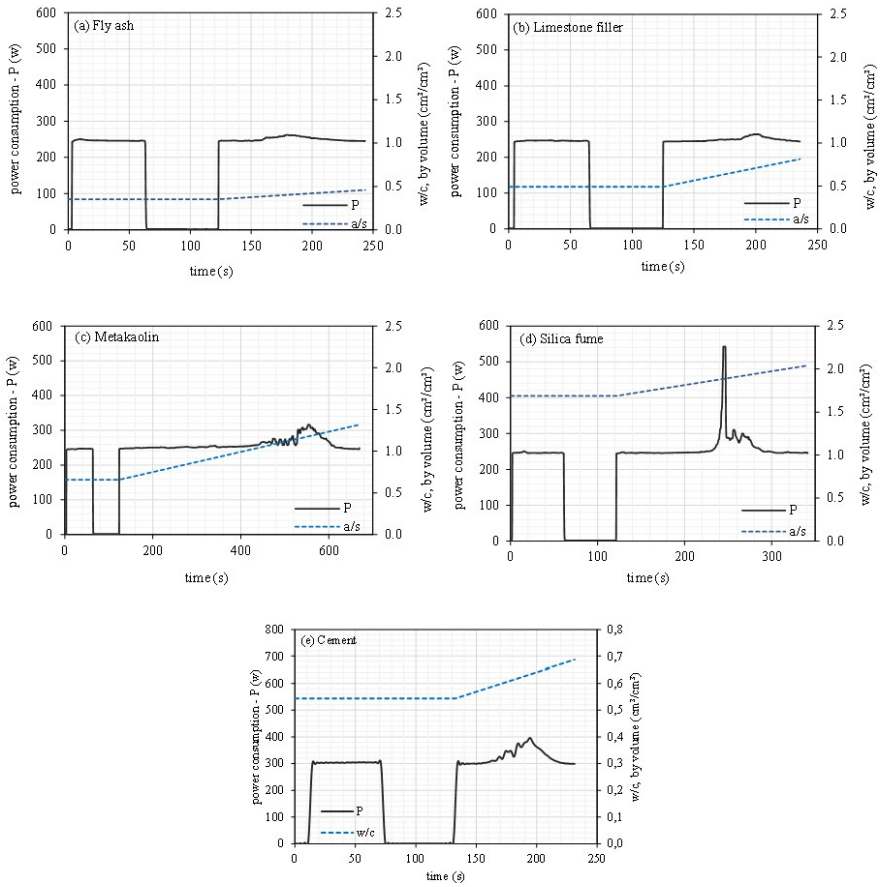

Figure 9. Mixing energy test results, with 0.5ml/s water additions for (a) Fly ash, (b) Limestone filler, (c) Metakaolin, (d) Silica and (e) Cement

Table 7. Packing density and water demand of Portland cement and mineral additions

Material	Packing density (-)	Minimum w/s, by volume (-)	Minimum w/s, by weight (-)
Portland cement	0.597	0.619	0.194
Fly ash	0.704	0.402	0.200
Limestone filler	0.578	0.704	0.126
Metakaolin	0.455	1.177	0.472
Silica	0.344	1.885	0.677

In the mixing energy test for the SCMs, the same behavior observed for the Portland cement was found. With the continuous addition of water, there was a gradual increase in power consumption, relative to the approximation of the particles, until the funicular state (maximum power consumption) was reached, where the particles are in contact, the packing density and the minimum water demand were set.

Regarding the energy consumption for mixing the pastes, it can be seen in Figure 9 that cement and metakaolin showed similar behaviors, with intermediate energy peaks. It is noteworthy that these materials had an average diameter of 11 and 18μm, respectively. The fly ash and the limestone filler (with diameters around 40 μm) required a low energy consumption at their peak. The silica fume (with an average diameter of 0,2 μm) presented an intense and well-defined peak of energy consumption during mixing. The peak energy consumption is more intense the smaller the diameter of the particles since smaller diameter particles will exert higher forces of attraction when compared to the test performed on coarser grains.

Comparing the materials presented in Table 7, it is observed that the highest value of packing density was obtained for fly ash, the lowest was found for silica fume, while the cement, limestone filler and metakaolin presented intermediate values. These differences are expected due to the differences in particle size distributions of each of the materials.

Values of the packing density of Portland cement are similar to those found in the literature: Wong and Kwan [11], using the originally proposed method, obtained a packing density of 0.622 in the presence of a chemical admixture [29]. Similar values for packing density were founded by Campos et al. [52] for Portland cement (0.610) and silica fume (0.354) using the wet method in the presence of chemical admixtures.

The packing density of silica fume is lower than the other SCMs. On the other hand, the water demand for this material was the highest among those studied. This fact demonstrates the need for a higher amount of water (w/s) to obtain the maximum solid concentration. Figure 10 shows a relationship between the water demand and packing density from the mixing energy test.

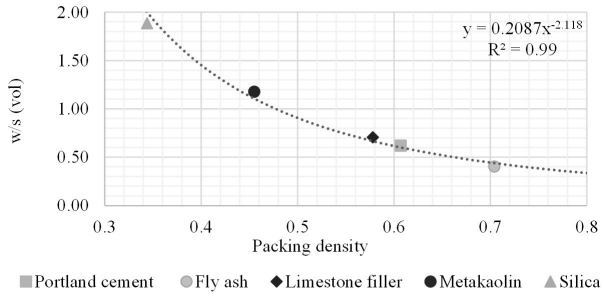

Figure 10. Packing density and water demand correlation from the mixing energy method

Since the packing density is based on a direct measurement of the solid concentration and water content in the mixture, the relationship presented in Figure 10 is expected and demonstrates that only with a low water demand is possible to achieve a high packing density. It was not possible to establish a direct relationship between the mixing energy test results and the average diameter of the particles since the PSD, specific surface, contact angle, water absorption and grain morphology will also influence the test results.

Although this paper evaluated single binders (cement or SCMs) aiming the determination of the packing density of each separate material, future applications of the mixing energy test method could also be performed on binary or ternary mixtures of binders.

4 CONCLUSIONS

The present work addresses the comparison of packing density results measured by the wet packing method and the mixing energy method. From the experimental program developed and the results obtained, the following conclusions can be taken:

- The packing density results achieved with the mixing energy method were like those achieved by the wet packing method in a 200ml container. Considering the best setup for each test (packing density of 0.597 in the mixing energy method with 0.5ml/s in 1.0kg of cement and packing density of 0.636 in the wet packing method with 200ml), the relative difference between the techniques is 6.1%. This variation is relative to the compaction applied to the wet packing test. The mixing energy method also consumed almost seven times less cement and was carried out in less than 15% of the time required for the wet packing method (since it requires only one mix for the measurements to be performed).

- In the mixing energy method, reducing the amount of cement to 1.0 kg avoided the paddle locking during mixing, and the continuous addition of water of 0.5ml/s proved to estimate the funicular state with better precision, giving the system more time to identify the energy peak and eliminating any delay on the paste rheology due to the water addition. While adding 10ml every 20s caused asymmetrical energy consumption that could be related to a delay in the water effect and influence the determination of the packing density and water demand.
- The mixing energy method allows a better estimation of the water demand and packing density since a discrete measurement can be performed with a small amount of cement without the influence of evaporation and compacting processes.
- The mixing energy test was effective in measuring the packing density and water demand of SCMs with a wide range of particle sizes. Some adaptations to the initial weight and water were required to properly measure the power consumption. A relationship between solid concentration and water content was found in the samples.

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