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Additional Information

Design and Quality Control of Fresh Concrete Using Halogen Moisture Analyzer for Calculating the Amount of Retained Water in Fine Aggregates

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Abstract

Measuring the rate of water absorption of the aggregates used in mortars and concrete mixtures is critical for the production and application of the final product. This is because it determines the amount of water required to achieve proper cement hydration and thus ensure the appropriate consistency. This paper focuses on the study of a new technique for measuring retained water in fine aggregates as an alternative to other conventional techniques specified in several international standards. In order to obtain results, the procedure described in the existing standards takes more than 24 hours. Additionally, it involves high consumption of heat energy due to the use of drying ovens. Furthermore, the results obtained remain highly variable and therefore discussed by the scientific community. In this research, a novel technique based on a halogen moisture analyzer was implemented. The technique was assessed using an experiment design with a surface response model. The most important factors and levels were identified together with the interactions between them. Finally, the model was validated and the results obtained with the proposed technique were compared with those obtained via conventional techniques in order to verify that they were equivalent.

Keywords:

Fine aggregate, absorbed water, adsorbed water, characterization technique, concrete, mortar, thermal analysis

Introduction

Water retention is defined as the amount of water required to fill the pores of the aggregate completely -absorption- and to cover the surface of the particles -adsorption-. The amount of retained water by an aggregate affects the properties of the resulting fresh concrete, particularly its workability which according to the technical standard of the American Society for Testing Materials (ASTM C 125), this property (workability) is related to the effort required to manipulate a freshly mixed quantity of concrete with minimum loss of homogeneity. Also retained water can reduce mechanical strengths (compression and flexo-traction) in a hardened state. On the other hand, the inclusion of saturated aggregates in the mixture acts as a water reservoir that prevents the reduction of the relative moisture into the cementing matrix. This method is known as "autogenous" or "internal" curing. Recent studies have established that this kind of curing could be successfully applied to produce high-resistance concrete by significantly reducing cracking [1, 2]

Aggregates account for 60% to 80% of the total volume of the concrete's mass and are its most

porous components. Therefore, they retain the highest amount of water in this composite material [3]. The amount of water retained depends mainly on the amount and continuity of the pores in the particles, their size and shape and, to a lesser extent, their mineralogical constitution [4]. The more precise the results for absorption would have as a consequence the better the design of the mix and, consequently, the properties of the concrete.

Some practices propose saturating the aggregates with water before preparing the mixture. Consequently, the amount of water required for the cement hydration is not absorbed, thus improving the cement-aggregate interface [5]. Conversely, if dry aggregates are used, the water required for densifying the cement-aggregate interface will be absorbed. Other studies [4] establish a relationship between cement paste porosity and the initial amount of water in the aggregates: dry aggregates (0% of saturation) produce higher macroporosity and lower resistance in the concrete. When concrete is made with saturated aggregates (100% of saturation), it tends to show higher mesoporosity and less resistance than partially saturated aggregates (50% of saturation).

According to the ASTM 128 standard, absorption is measured by calculating the increase in mass of a solid porous body. This increase results from the penetration of a liquid into the permeable pores of said body. The NTC 237 standard prescribes the method for determining the density and absorption rates of a fine aggregate. The process described in the NTC 237 is equivalent to that of other international norms and shall be referred to as "the conventional technique" throughout this paper.

According to the conventional technique, 1 kg of material is required for preparing the test sample. Similarly, a mechanical splitter or the quartering method can be used to obtain the necessary amount of material. Then, it is dried at a temperature of 110 ± 5 °C until a constant mass is obtained. Next, it is cooled until it can be handled and then it is immersed in water for $24 \text{ h} \pm 4 \text{ h}$. After the saturation time has passed, excess water is removed, the material is placed on a flat surface, exposed to a stream of warm air and mixed to ensure even drying until it reaches a saturated surface dry state (SSD). SSD is defined as the condition in which the permeable pores of the aggregate particles are completely filled once immersed in water for a given amount of time. Likewise, there is no free water on the surface of the aggregate.

To guarantee that the material is in a saturated surface dry state, a mold in the form of a cone frustum is filled to the brim with the aggregate. Then it is compressed 25 times using a ram. The height of each fall must not exceed 5 mm. After this, loose sand is removed from the base and the mold is raised vertically. If there is still surface moisture, the aggregate will retain the shape of the mold. Furthermore, the fine aggregate settles slightly when it reaches the saturated surface dry state. On the other hand, if the material collapses completely, then the aggregate is too dry; thus, it is necessary to moisten it again for 30 minutes and then repeat the process.

When the SSD state is reached, the test sample is dried at a temperature of $110 \text{ °C} \pm 5 \text{ °C}$ until a constant mass is obtained. Then it is cooled at room temperature for $1 \pm \frac{1}{2} \text{ h}$ and its mass is determined. Finally, absorption is calculated as follows:

$$\text{Absorption, \%} = \frac{(S-A)}{A} 100 \quad (1)$$

Where A is the mass (g) of the oven-dried sample.

And S_s is the mass of the saturated surface dry sample (SSD).

R.Kasemchaisiri and S.Tangtermsirikul [2] demonstrated that the conventional method for obtaining the SSD is not effective due to the high degree of inter-particle friction existing in the aggregate. They [5] developed a new method based on the concept of free water under gravity conditions. They subsequently compared it with the method established by the ASTM C 128. The results regarding the appearance of moisture in the sample when the cone collapses (NTC 287 and ASTM C 128) indicated that the portions were very dry as some of them took a white color due to excessive drying. In contrast, the portions used in the proposed method retained the shape of the cone. The values for the standard deviation of the proposed method were also lower than those obtained with the method proposed in the ASTM C128. This indicated that collapsing cone criterion specified in the standard for finding the SSD was not suitable for aggregates with fine pores and a high degree of inter-particle friction. The reason for this is that this condition would allow the material to retain its cone shape even when it was highly dry.

Many other alternative methods for determining the aggregates' dry surface have been researched [2]. One of such is based on the water displacement process throughout the dry aggregate. In this method, absorption can be calculated by measuring the original apparent volume of the dry aggregate as well as by measuring the decrease in the apparent volume due to water being absorbed into the aggregate's pores. Another method [5] [6] involves forcing saturated moist air through the compacted particles of the aggregate. A dry surface is thus defined as the change in the slope of the curve that relates drying time and aggregate weight. For light aggregates [7] the dry surface of an initially moist aggregate can be obtained by centrifuging the aggregate for a fixed amount of time. The centrifugation method is theoretically recommended for light aggregates, but it is too sophisticated for field use.

V.W.Y.Tam, X.F.Gao, C.M.Tam and C.H. & Chan [8] proposed another method for obtaining the absorption rates in recycled aggregates. This technique is called Real-time Assessment of Water Absorption (RAWA), and provides an easier way to obtain water absorption at different time intervals without having to immerse and dry the recycled aggregate, thus obtaining a more accurate result. When using the RAWA technique, the mass of the dry aggregate is obtained by putting the sample in an oven at 75 ± 5 °C. In this way, free water is removed from the aggregate without losing any crystallized water (which is found in the cement based residues). The temperature must be kept for $24 \pm \frac{1}{2}$ h or longer, until a constant mass is reached. After this, the aggregate is placed in a pycnometer and filled with distilled water. Any water levels resulting from the T1, T2, T3... Ts time intervals as well as any other noticeable drop in the water levels are caused by the aggregate as it absorbs water and by the evaporation occurring inside the room. Then, the pycnometer is filled again with water and weighed to obtain the values for initial mass (M_i) and initial time (T_i). The difference between the mass of T_i and $T_i - 1$ equals the amount of water absorbed by the aggregate (M_i, M_{i-1}). This procedure is repeated at different times until there are no more changes in mass or no decrease in water levels, which indicates that the saturated condition of the aggregate has been achieved in T_s . In this study the authors conclude that the BS:812 standard for assessing the water absorption of recycled aggregates is not suitable. The patches of cement paste adhering to the surface of the aggregate affect water absorption. This is why the standard 24 h for saturation is not adequate. The RAWA method avoids removing the cement adhered paste during the soaking and

drying phases. Furthermore, this is a simple and more precise method for measuring the true water absorption value of recycled aggregates.

Djerbi Tegguer.A, [1] determined the water absorption of recycled aggregates using hydrostatic weighing. This method consists in observing the aggregates' rate of capillary rise over time. Water absorption is measured gravimetrically, using hydrostatic weighing to measure changes in the mass of a sample that has been immersed into a thermo-regulated bath. The aggregate sample is dried in the oven at 110 ± 5 °C until the mass difference is less than 0.1%. After drying, the sample is placed in a basket and hung in a scale. The mass of the system is registered as the value of the mass of the oven-dried aggregate when measured outside the oven M_s . Then the thermostatic bath is moved until the sample is completely immersed in distilled water at a temperature of 20 °C. It is assumed that the first value observed after immersing the sample is the mass of the oven-dried aggregate in water. This value is called M_{ws} . Due to the capillary effect, the percentage of water absorbed is calculated with the following equation:

$$100 \frac{M_{wa} - M_{ws}}{M_{ws}} \quad (2)$$

Where M_{wa} is the saturated aggregate sample in water.

Similar to the previously cited study, this work confirms that the procedure described in the NF EN 1097-6 standard (which establishes a saturation time of 24 h) is not suitable for recycled aggregates. In the case of the granulometric fractions of 12.5 - 20 mm and 5 - 12.5 mm, the water absorption coefficient of the recycled aggregates which have been soaked for 24 h generates around 60% and 70% of the total amount of absorbed water obtained after 85 h and 110 h of soaking, respectively.

In the present study, the authors propose a new and easy method for determining the amount of water retained in fine aggregates. The technique, that uses the halogen moisture analyzer, could decrease the time in which results are obtained as well as the amount of energy consumed and the probability of human error associated with experiments conducted using the conventional technique.

Theoretical Concept

Thermogravimetry is the process through which the loss/gain of mass of a sample is determined via direct and/or indirect heating. This technique makes it possible to obtain the percentage of moisture [9] in a material by regulating the temperature in values below 100 °C.

There are several sources of infrared radiation (IR) used to generate heat. One of such is halogen light as a near infrared source of heat. This technique generates heat faster than traditional systems. Others like Mendoza and Tobón [10] have studied the chemical reactivity of pozzolan with lime through thermogravimetry in a halogen moisture analyzer.

By using the halogen moisture analyzer, continuous water loss measurements can be obtained. These measurements may be associated with adsorbed and absorbed water in a material. Fig. 1 represents the state of the particle under different moisture conditions. SSD is considered the most suitable state since particles in this condition will not absorb, thus leaving the properties of the concrete

unaffected, which in turn leads to a good mix design. When the state of particle changes from SSD to dry when heating, the rate of de-absorption (rate of mass loss) is defined.

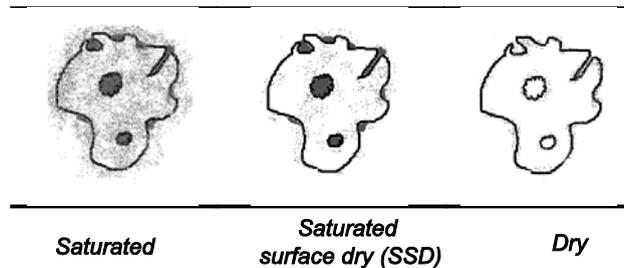


Fig. 1 Moisture conditions in a particle of the aggregate

The hypothesis used in this research for calculating absorption rates is based on the change observed in the slope of the curve which, under isothermal conditions, may be representative of water loss versus time (Fig. 2). It is thus a method based on the kinetics of water evaporation. The first stage is characterized by adsorbed-water loss. The particles are initially in a moist state but eventually reach the SSD condition. The second stage is characterized by water loss associated with small fissures and porosity. It is thus assumed that the speed with which water is lost in those cases is different.

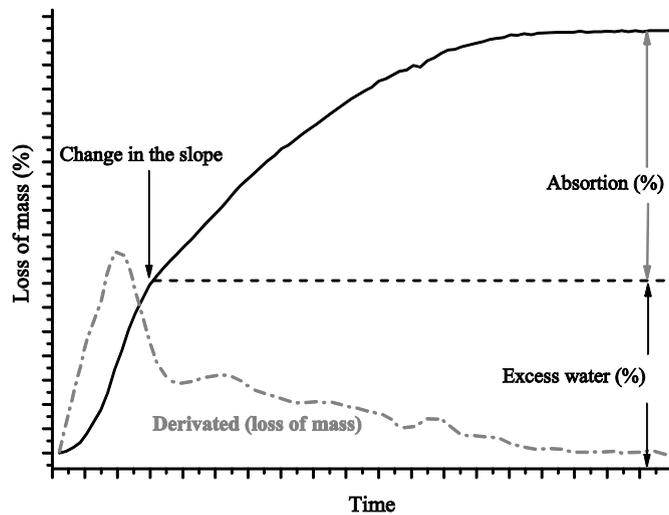


Fig. 2 Loss of mass in relation to time.

In order to identify when the absorbed water begins to be lost, it is first necessary to obtain data regarding the rate of water loss in relation to time and also to identify the point where the slope changes or the corresponding derivative curve . It is also worth noting that this absorbed water is strongly linked to the particles.

Equipment

An halogen moisture analyzer (Precisa XM 60 moisture analyzer) with a maximum load capacity of 120 g and a sensitivity of 1 mg was used. The halogen light ensures quick and uniform sample heating for reproducible results within minutes. The operation data used for plotting the mass loss vs. heating time curve were extracted through a cable using the analyzer's USB output port.

Test Materials

The sand used in this research was obtained from a quarry. Its shape was angular and its texture vitreous, as seen in the microscopic image shown in Fig. 3. The granulometry complied with the requirements specified in the ASTM C 33 standard or in its equivalent NTC 174. The granulometric curve is shown in Fig. 4. As for mineral composition, it has a high content of silica with traces of feldspar. Furthermore, its genesis is sandstone.

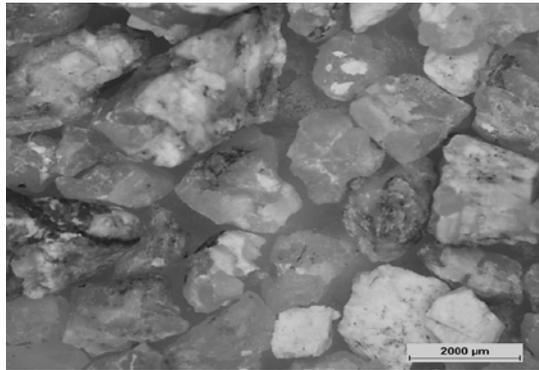


Fig. 3 Microscopic picture of the sand used for this research project.

In order to identify the effect of the granulometry on the water absorption rate, three fractions of sand were used: fraction 1 was defined as the sand for the whole series, i.e. sand without any modifications; fraction 2 was sand retained on a sieve with a mesh aperture of 0.6 mm; this sand was labeled coarse sand. Finally, fraction 3 corresponded to the sand passing through the same mesh and retained on the mesh aperture of 0.15 mm; this sand was labeled as fine sand.

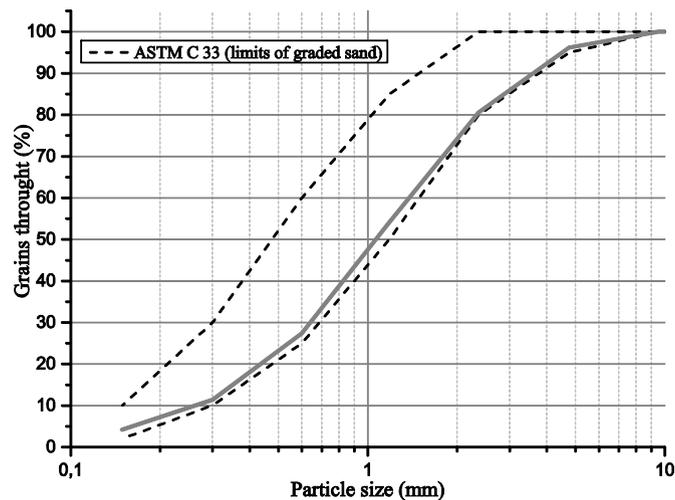


Fig. 4 Granulometric curve for the sand used in the study

The three fractions belong to the same type of sand, but their granulometric distribution is different. This makes possible to establish the effect of particle size on the rate of absorption using both the conventional method described in the ASTM C 128 and the halogen moisture analyzer method.

Testing procedure

The experiment consisted in using the quartering method to choose 1 kg of sand from each fraction. Then, the samples were immersed in water for 24 h to guarantee complete saturation.

After 24 h, the material was extracted and exposed to water suction in a funnel with filter paper. This process enabled us to extract large amounts of excess water from the surface of the particles. Then, a portion of the material was put into the sample holder of the halogen moisture analyzer in order to obtain the data regarding the rate of mass loss vs. time.

The experiment design for studying the absorption rate took into account the following factors:

- A: Test temperature (around 80 °C)
- B: Test mass (around 42.5 g)
- C: Time spent in the vacuum pump (approximately 80 seconds)

Previous studies carried out in this research have shown that the recommended test temperature had values around 80 °C. Any lower values induced longer test times, while higher values caused rapid water evaporation, which in some cases made it more difficult to identify the change in the slope of the curves displaying moisture loss vs. time (or the corresponding derivative curve).

Test mass was defined as an important factor because a large mass can hinder water loss for the particles located in the bottom of the litter, which also causes the particles located in highest levels to start losing the water that is most strongly bound.

The last factor was the time spent in the vacuum pump. It was established to standardize the process and thus eliminate large amounts of moisture (superficial water) from the test mass.

Experiment Design

To determine the best process conditions, we used an experiment design based on the response surface methodology using the three factors described above and identifying the central points. The response surface design was presented with a replica and applied for each of the three sand fractions. Table 1 shows the matrix of experimental runs.

In order to validate the results, we compared the data obtained via this method with those from the conventional technique (ASTM C 128).

Table 1. Experimental design matrix

Temperature (°C)	Mass (g)	Time spent in the vacuum pump	Response variable (Absorption %)		
			Coarse	Whole series	Fine
80.0	42.5	12.7	1.46	1.13	2.50

80.0	13.1	80.0	2.30	2.52	3.30
80.0	42.5	147.3	1.37	1.36	1.90
90.0	25.0	120.0	1.86	1.80	2.10
80.0	42.5	80.0	1.30	1.65	1.40
80.0	71.9	80.0	1.02	1.12	1.30
90.0	60.0	120.0	0.85	0.96	1.40
70.0	25.0	120.0	1.61	1.59	3.10
80.0	42.5	80.0	1.61	1.46	2.30
80.0	42.5	80.0	1.25	1.66	2.00
96.8	42.5	80.0	1.26	1.77	1.80
80.0	42.5	80.0	1.15	1.60	1.70
63.2	42.5	80.0	0.86	1.09	1.30
70.0	25.0	40.0	1.75	1.72	1.70
90.0	60.0	40.0	0.96	1.16	1.60
70.0	60.0	40.0	0.90	1.19	1.50
80.0	42.5	80.0	1.54	1.72	2.20
90.0	25.0	40.0	1.29	2.25	3.10
80.0	42.5	80.0	1.12	1.76	1.80
70.0	60.0	120.0	1.03	0.77	1.50

Analysis and Results

Table 2 shows the p values obtained for each of the factors as well as their interaction. For the proposed ANOVA the null hypothesis states that the factors temperature, test mass and time spent in the vacuum pump have no significant effect on the water absorption values for the assessed fractions of sand.

For a significance of $\alpha = 0.05$, i.e. with an associate confidence of $\beta = 95\%$, we can say that, if the p value is less than α , then the null hypothesis is rejected.

Table 2. p values of the ANOVA for each fraction of the sand

Variable	Coarse sand	Whole series	Fine sand
Temperature (T)	0.386	0.009	0.002
Test mass (m)	0.544	0.000	0.421
Time spent in the vacuum pump (t)	0.416	0.224	0.000
Double interaction (T*T)	0.669	0.079	0.213
Double interaction (m*m)	0.593	0.285	0.211
Double interaction (t*t)	0.405	0.007	0.355
Double interaction (T*m)	0.167	0.255	0.355
Double interaction (T*t)	0.343	0.839	0.024
Double interaction (m*t)	0.207	0.935	0.586

Since the p value did not show any level of significance for the coarse sand fraction, the factor *time spent in the vacuum pump* was considered as part of the error and the ANOVA was evaluated again (see Table 3)

Table 3. p values of the ANOVA for the coarse sand fraction

Variable	<i>p</i> value
Temperature (T)	0.040
Test mass(m)	0.150
Double interaction (T*T)	0.036
Double interaction (m*m)	0.080
Double interaction (T*m)	0.871

Upon obtaining the ANOVA, the data were analyzed to determine a statistical model. Similarly, this model was plotted as response surface using isolines in the contours system. The contours plots represent the test conditions (mass and temperature) which could yield different results for the rate of absorption of aggregates using a halogen moisture analyzer.

The results of the statistical models are described below:

Sand for the whole series: Refined model with an adjustment of 73.85%

Fine sand: Refined model with an adjustment of 64.02%

Coarse sand: Refined model with an adjustment of 73.53%

The refined models were named that way because they made it possible to consider one of the assessed factors as part of the error. The factor "time spent in the vacuum pump" was considered as part of the error for both coarse sand and the sand for the whole series. This was not done for fine sand because, in this case, the time spent in the vacuum pump showed very significant values in the ANOVA.

Analysis for the coarse sand fraction using a halogen moisture analyzer. Table 3 shows that temperature is the main factor in the response variable. It also indicates that there is a marked effect of curvature as can be observed in Fig. 5.

Fig. 5 shows the contour isolines. These indicate the rate of absorption associated with the proposed experimental methodology in which a value of approximately 80 °C is considered the maximum for concavity.

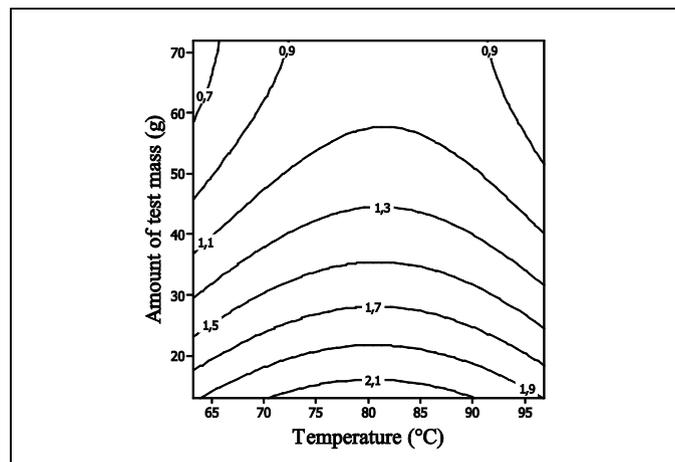


Fig. 5 Contours for coarse sand

The absorption values obtained using this methodology did not exceed 2.1%. This is consistent with [11], who reported that the absorption rate of silica sand usually ranges from 0.4% to 3.8%. In addition, these studies also found that, when particle size increases, the rate of absorption tends to decrease. This is due to the surface/volume ratio, which is higher for fine aggregates and allows water to be in contact with a larger surface.

Analysis of the fine sand fraction using the halogen moisture analyzer technique. The results show that mass was not significant in the response variable. In contrast, the time spent in the vacuum pump was highly important. This is mainly because the fine sand was distributed uniformly on the paper filter, thus generating high suction pressures and allowing the extraction of higher amounts of superficial water. The temperature also had an effect on the response variable as shown in Fig. 6.

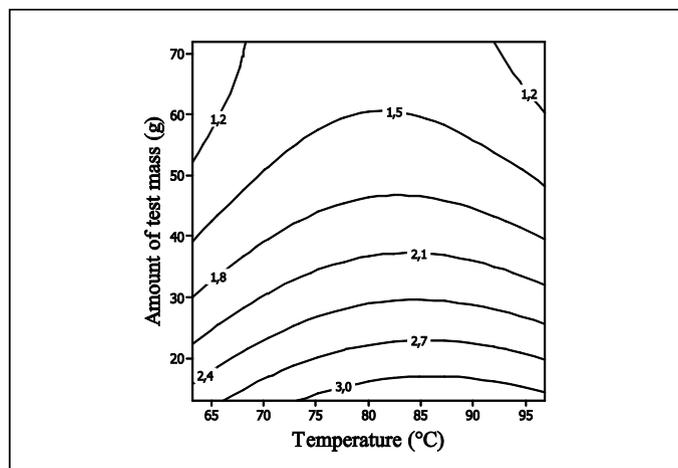


Fig. 6 Contours for the fine sand fraction

Although the contour lines also indicate an effect of curvature in relation to the temperature. This was not as marked as in the case of the experiments with the coarse sand fraction. Instead, we have the effect of the interaction between time spent in the vacuum pump and the temperature.

Analysis for the fraction of the whole series using a halogen moisture analyzer. It was observed that the test mass showed the highest level of significance and that the temperature continued to show an effect of curvature as seen in Fig.7.

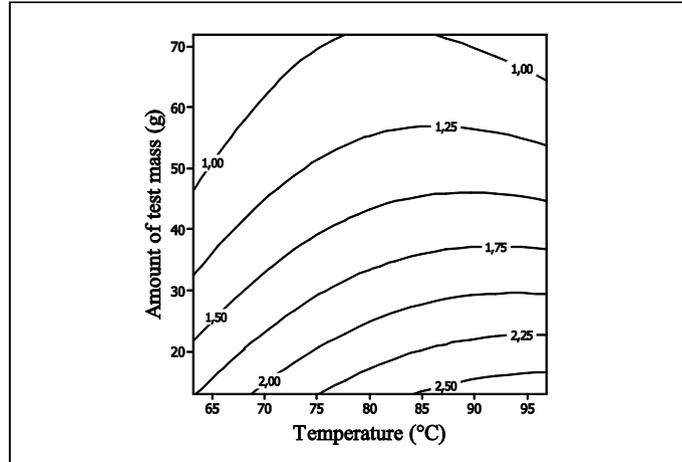


Fig. 7 Contour for the fraction of the whole series

Analysis for the fraction of the whole series using the conventional technique (ASTM C 128) . According to Table 4, the results obtained show high variability: there are two standard deviations that are equivalent to a confidence interval of 95%. In addition, we observed that the standard deviation for the tests conducted on all fractions was very similar. It is thus possible to establish that the tests were correctly conducted. There was a variation of 25% in the mean for both the highest and lowest values of all the tests.

From the results observed in the previous table it can be established that the absorption rates for fine sand and coarse sand are respectively 23.3% and 17.0% higher than the absorption values for the fraction of the entire series. Thus, it is important to have an appropriate granulometric distribution in the mixtures in order to obtain lower absorption rates.

Table 4. Absorption rate under the ASTM C128

	Mean	Standard deviation	Min.	Max	Tests (random)
Fine sand	3.34	0.37	2.60	4.09	6
Sand for the whole series	2.71	0.40	1.91	3.51	6
Coarse sand	3.17	0.38	2.40	3.93	6

Comparison of the two techniques. The technique described in the ASTM C 128 standard was compared with the halogen moisture analyzer methodology through contours plots. The white region in Fig. 8 and 9 is the area where the two assessed techniques intersect. Thus, it is defined as the region of confidence under which the results of both techniques are statistically equal.

When comparing the two methodologies, the use of coarse sand yielded no equivalent results in both techniques. Most likely, this is because, in order to reach the SSD condition for coarse sand, the conventional technique (ASTM C 128) requires a higher degree of surface moisture. This moisture compensates for the lack of fine particles and thus makes it possible to reach a level of cohesion that makes it possible for the material to retain the shape of the cone.

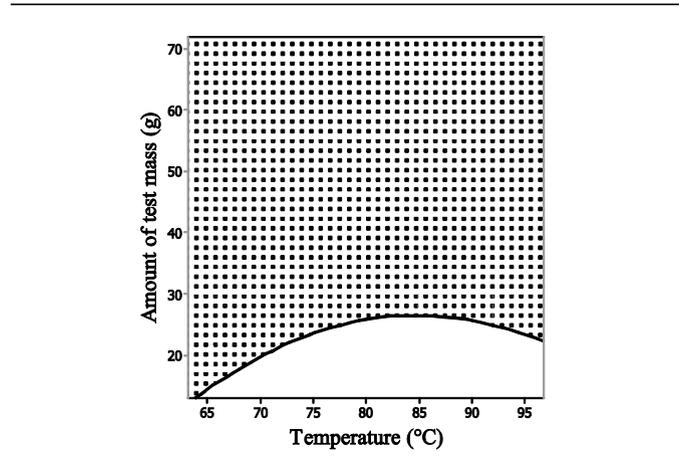


Fig. 8 Region (white zone) where the two techniques intersect for the fine sand fraction.

Fig. 8 shows the region where the rates of absorption observed with both techniques intersect when using fine sand. It can be observed that results are comparable for masses below 25 g and at a temperature ranging from 65 °C to 95 °C approximately. This is most likely a result of the homogeneous distribution in the sample holder of the halogen moisture analyzer, which guarantees uniform water evaporation. Greater masses showed a less uniform loss of mass, which makes it difficult to interpret the loss of water vs. time curve.

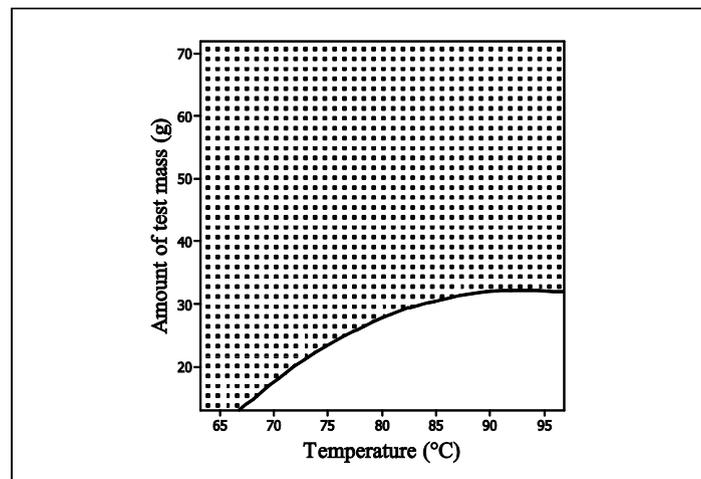


Fig. 9 Region of intersection (white zone) of the two techniques in the sand fraction for the whole series

For the sand fraction of the whole granulometric series, we observed that the confidence region is wider than that of the fine sand series in terms of test mass, and very similar in terms of temperature (See Fig. 9). This result confirms that the fraction whose results for absorption rate are most comparable with those of the conventional technique is the fraction containing a more continuous and better-distributed granulometry (i.e. a richer granulometry).

Conclusions

The halogen moisture analyzer technique can be used to determine the absorbed water of sand if there is a good granulometric distribution in the range between meshes No. 4 and No. 100.

The halogen moisture analyzer technique let to yield absorbed water results that are comparable to those obtained by means of the conventional technique: a mass of approximately 25 g and a heating temperature of approximately 85 °C should be used.

For the coarse sand fraction, the halogen light technique underestimates the values of the water absorption assessed using the conventional technique.

A very similar trend was observed for the temperature and for the test mass required in the determination of the absorbed water for the fine sand and for the whole series.

The technique which uses the halogen moisture analyzer reduces the time required to obtain accurated results, the amount of energy consumed and the probability of human error associated with the experiments conducted using the conventional technique.

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