

## Comparison between the American standard and the RILEM recommendation for the calculation of the porosity of mortars

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### ABSTRACT

Cementations materials have chemical, physical and mechanical characteristics that evolve over time, which explains the complexity of their behavior. The porosity accessible to water is considered as one of the major physical characteristics of the durability of concrete and mortars. To determine this, there are several methods or standards, among them American Standard ASTM C642-97 American Society for Testing Materials and Recommendation RILEM-49TER International Meeting of Laboratories and Materials Experts. The difference between these two methods is in the saturation mode of the sample. The first is based on the boiling of the sample in hot water for 5 hours and the second uses the saturation under vacuum for duration of 4 hours. The mortars which were the subject of this study were made from mixtures containing variable contents in siliceous additions (silica fume). These additions are added in substitution to the cement (6, 9 and 12%) with a constant E / L ratio. The study focuses on the binding contribution of silica fume on the physical properties (density, porosity) and mechanical properties (dynamic modulus of elasticity) of mortars, and secondly, a comparison between the two standards. The results show the beneficial effect of silica fume on the physical and mechanical properties and for what it the appearance is, the ASTM standard is considered more effective than the RILEM standard.

#### Keywords:

Correlation, mortar, silica fume, porosity, ASTM, RILEM

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## 1. Introduction

The incorporation of mineral additions is now an important technique in improving the properties of cementitious materials. These mineral additions significantly affect the rheology of fresh cement materials, which is directly related to the development of strength and durability of cured materials [1]. Among these mineral additions, silica fume plays an important role in the manufacture of very high performance concretes [2].

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It is accepted in the literature that silica fumes lead to high and very high performance concretes, both at the young and at the advanced ages [2, 5]. Indeed, very fine particles of silica fume can fill the space between cement grains [6], which makes the concrete matrix more compact and thus improves their durability [2].

The porosity accessible to water is a very important parameter when interpreting durability studies such as freeze / thaw resistance and accelerated carbonation. Two methods or norms are currently used to determine this physical characteristic: American standard ASTM C642-97 [7] and recommendation RILEM-49TER [8, 9]. The only difference between them is in the saturation mode, the first uses the boiling technique of the samples for 5 hours and the second the saturation takes place under vacuum for a duration of 4 hours. This study is based on the influence of the substitution rate of silica smoke on the density, porosity, propagation velocities of ultrasonic waves and the dynamic modulus of elasticity of mortars in the state hardened. As well as the effect of saturation mode on the results of density and porosity.

## 2. Experimental Plan

### 2.1 Materials Used

#### 2.1.1 Cement

The cement used in this study is CPA-CEM I 42.5N, from the cement plant in Zahana (Algeria), chemical compositions of cement and mineralogical clinker and physical characteristics is summarized in Table 1, 2 and 3.

**Table 1**  
 Basic chemical composition of CPA-CEM cement I 42.5

CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	MgO	RI*	PAF**
63,89	21,35	4,59	5,52	2,72	0,41	0,13	1,37	0,22	2,47

\*: Insoluble Residues, \*\*: Fire Loss.

**Table 2**  
 Mineralogical composition of the CPA according to Bogue

Elements	C <sub>3</sub> S	C <sub>2</sub> S	C <sub>3</sub> A	C <sub>4</sub> AF
Content (%)	53,55	20,34	2,17	15,68

**Table 3**  
 Physical characteristics of the CPA

Characteristics	Values
Specific surface area Blaine (cm <sup>2</sup> /g)	3304
Bulk density (g/cm <sup>3</sup> )	1.02
Absolute density (g/cm <sup>3</sup> )	3,12
Start time (minute)	110
End of setting (minute)	220
Normal consistency (%)	25

#### 2.1.2 Sand

The sand used is a standardized sand conforming to standard NF P 15-403 which is a natural sand, siliceous in particular in its finest fractions. It is clean, the grains are generally isometric and rounded.

It is dried, riddled with all guarantees of quality and regularity, controlled by the testing laboratory of materials of the city of Paris (L.E.M.V.P), made in plastic bags of net weight equal to  $1350 \pm 5\text{gr}$ .

### 2.1.3 Smoke of silica

The smoke of silica used is the smoke (SILTEK POWDER), in the form of a powder of gray color, consisting of 93 to 98% of amorphous  $\text{SiO}_2$  spherical particles, its particle size varies from 0.05 to 0.15  $\mu\text{m}$ , is about 220,000  $\text{cm}^2 / \text{g}$  (Blaine). Its apparent density is 300  $\text{kg} / \text{m}^3$ . Its chemical composition is given in Table 4.

**Table 4**  
 Chemical composition of SILTEK POWDER silica fume

CaO	$\text{SiO}_2$	CaO	MgO	$\text{Fe}_2\text{O}_3$	$\text{Al}_2\text{O}_3$
$\geq 95$	$\leq 0.5$	$\leq 0.5$	$\leq 1$	$\leq 1$	$\leq 0.5$

### 2.1.4 Water for mixing

For the manufacture of the various mortars, drinking water was used, distributed by the public service network of the town of Mascara (Algeria). The chemical analysis of this water was carried out in the laboratory of Algerian water. The results are presented in Table 5. They meet the requirements of XP P 18-303.

**Table 5**  
 Chemical analysis of the mixing water

Ca	Mg	Na	K	Cl	$\text{SO}_4$	$\text{CO}_3$	$\text{NO}_3$	Fe	Organic material	pH
32,86	51,36	38,00	0,00	113,60	65,46	368,44	12,22	0,03	0,18	7,88

## 2.2 Procedure

### 2.2.1 Sample preparation

Mortars are made in accordance with EN 196-3 from mixtures with variable silica fume contents (6, 9 and 12%) and a plasticizer with a 2% cement weight dosage. The addition of silica fume is achieved by substituting part of the cement for the same weight of smoke.

In this study, we chose to consider the constant E / C ratio (= 0.5) and not the workability of the mortar. Each mixture makes it possible to manufacture simultaneously three cubic specimens ( $50 \times 50 \times 50 \text{ mm}^3$ ) of mortar. Table 6 gives the different constituents of each mixture and the notations used.

**Table 6**  
 Formulation of different mixtures of mortars

Component	MP0	MP6	MP9	MP12
Sand (g)	660	660	660	660
Cement (g)	220	206.8	200.2	193.6
The silica fume (g)	00	13.2	19.8	26.4
Water (g)	110	110	110	100
E/L	0.50	0.50	0.50	0.50

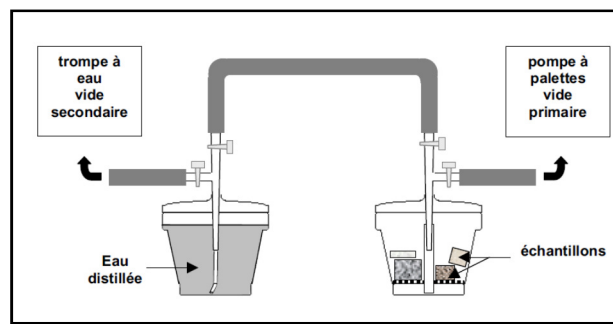
The concrete test specimens are stored in their molds for 24 hours at a temperature of  $20 \pm 1^\circ \text{C}$  and a relative humidity of  $55 \pm 5\%$  and then immersed in water at  $20^\circ \text{C}$  until the specified time.

### 2.2.2 Absolute density and porosity accessible to water

The bulk density and the porosity accessible to water were determined on cubic samples according to the two techniques;

The recommendations RILEM (49TER) [8]. The procedure adopted is as follows: The samples are first kept under vacuum for 24 hours in a vacuum bell, they are then immersed in water and kept under vacuum for 48 h. The volume of the sample is then determined by weighing in air and in water by means of a dispositive de pose hydrostatic.

To obtain the dry mass, the samples are dried at  $105^\circ \text{C}$ . to a constant mass (drying is achieved when the difference in mass obtained between two successive values (24 hour interval) does not exceed 0.05%).



**Fig. 2.** Experimental device for the measurement of porosity by imbibition of vacuum water

The American standard (ASTM C642-97) [7]: in this technique it is first necessary to dry the samples at  $105^\circ \text{C}$ . until the mass is constant ( $M_{sec}$ ). Subsequently, after final drying and cooling, the sample is immersed in water at about  $21^\circ \text{C}$  for at least 48 hours and until two successive values of the sample mass at 24-hour intervals do not exceed Not 0.5% ( $M_{humide}$ ). For determination of the mass of test piece in saturated state ( $M_{sat}$ ), in a suitable container boil the samples in hot water for 5 h. Then allow to cool for at least 14 hours to a final temperature of 20 to  $25^\circ \text{C}$ . At the end, after immersion and boiling, the apparent mass in water ( $M_{eau}$ ) is determined by hydrostatic weighing. The porosity accessible to water is calculated according to the formula

$$\varepsilon = \frac{M_{sat} - M_{sec}}{M_{sat} - M_{eau}} \times \rho_{eau} \quad (1)$$

The bulk density is calculated according to the formula:

$$\rho_{app} = \frac{M_{sec}}{M_{sat} - M_{eau}} \times \rho_{eau} \quad (2)$$

where

$M_{sat}$  : is the mass of the sample saturated in air

$M_{eau}$  : is the mass of the sample saturated in water

$M_{sec}$  : is the mass of the sample at the end of drying

### 2.2.3. Ultrasonic measurements

The measurement of the compression wave velocities ( $V_p$ ) is carried out using an ultrasonic apparatus [10]. The measurement of  $V_p$  on mortar samples consists of emitting an ultrasonic signal in the form of a pulse and analyzing its propagation in the sample. The travel time of the ultrasonic wave is measured between two piezoelectric sensors, a transmitter and a receiver, placed in contact with the sample facing each other; The division of the distance traveled by the wave over the travel time makes it possible to determine the speed of propagation. This type of non-destructive measurements in a material makes it possible to obtain information on the porosity, the state of cracking, and the elastic properties.

The dynamic modulus of elasticity, among others, is calculated from the velocity of the P waves ( $V_p$ ), the density of the hardened concrete ( $\rho$ ) and the fish coefficients ( $\nu$ ) of the material by the following formula:

$$E_d = \rho V_p^2 \frac{(1+\nu)(1-2\nu)}{(1-\nu)} \quad (3)$$

## 3. Results and Discussions

### 3.1 Porosity

Figure 3 shows the evolution of the porosity accessible to water of the samples after 28 days of curing in water at 20 ° C., as a function of the silica smoke content in the cement. Each value in this figure corresponds to the arithmetic mean of the results obtained on three test pieces. There was a decrease in porosity for 6% and 9% levels, either by the RILEM or ASTM method, and a slight 12% increase in content. These results are explained by the beneficial effects of silica fume on the microstructure of cementitious materials [11]; the rapidity at which the pozzolanic reaction develops and the particular physical effect of the silica smoke particles known Filler effect name. These two effects result in both a great increase in compactness and an improvement in mechanical strengths due to the pozzolanic reaction of the silica fumes. On the other hand, the 12% increase may be explained by the fact that the specific surface area of the silica fumes is larger than that of the cement, so that more water is added to the mortar if we want to have the same fluidity as the control mortar. In fact, we have fixed the ratio E / L equal to 0.5, consequently the fluidity of the mortar decreases according to the addition content.

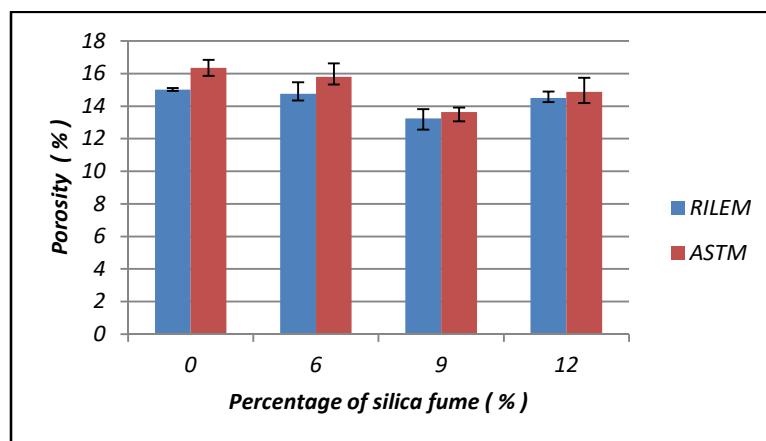
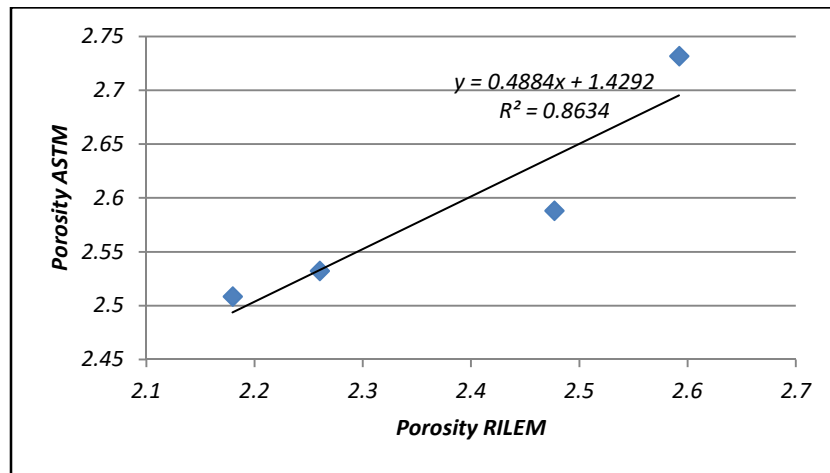


Fig.3. Effect of silica fume on the porosity of mortars

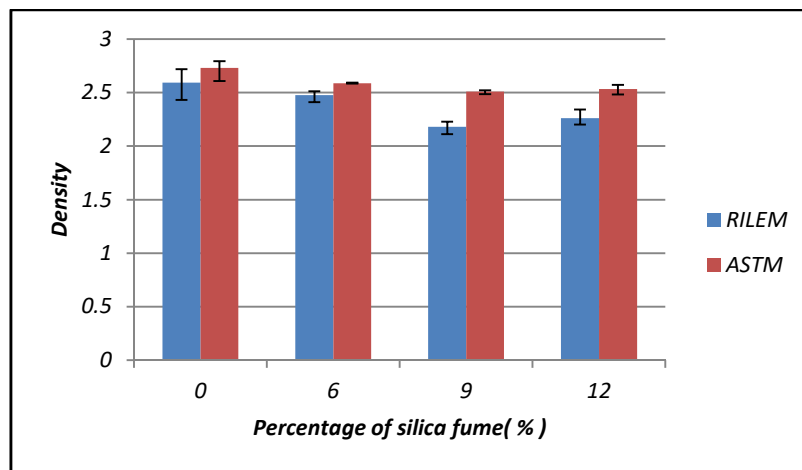


**Fig. 4.** Correlation between RILEM porosity and ASTM porosity

Furthermore, it is noted that the porosity values determined by the ASTM standard always give values higher than those calculated by the RILEM recommendations. It can be said that when the open pores are filled by boiling only by vacuum saturation.

### 3.2. Density

Figure. 5 shows the evolution of the density of the mortar specimens after 28 days of curing in water at 20.degree. C., as a function of the silica smoke content in the cement. These are the same samples that we used to determine the porosity. It is found that the density evolves in a similar way as the porosity. And it is still the ASTM standard that gives the highest values compared to the RILEM recommendations.

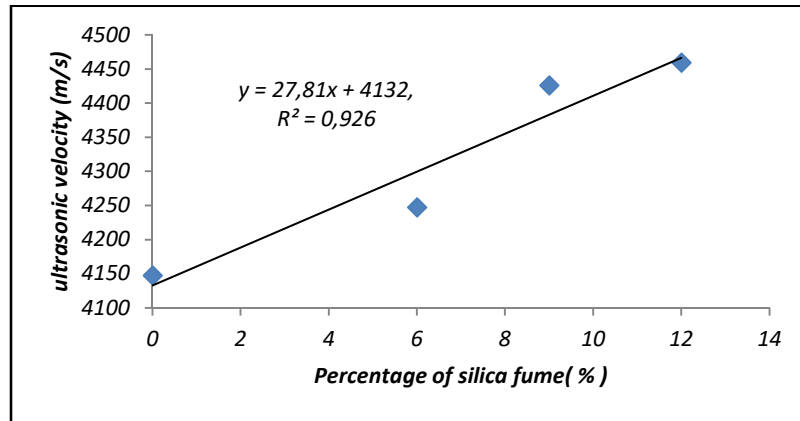


**Fig. 5.** Effect of silica fume on the density of mortars

### 3.3 Speed of Sound Propagation

Ultrasonic methods are frequently used for non-destructive characterization of materials. The ultrasonic signals are very sensitive to the heterogeneity of the medium. Depending on the state of the material, the waves change in speed, the propagation of the waves is perturbed by the

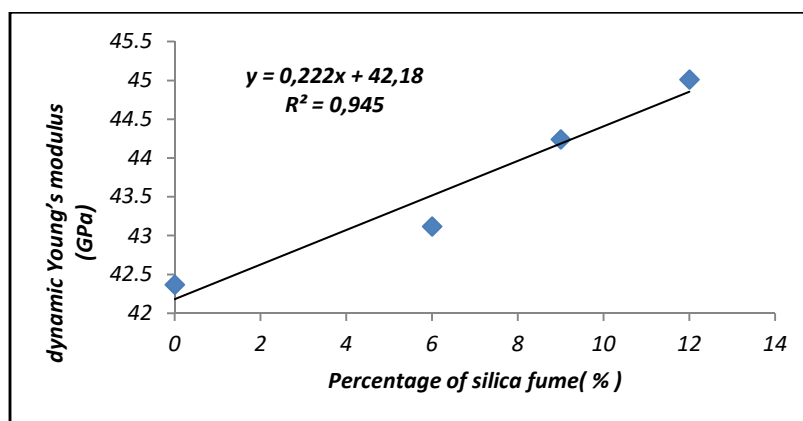
discontinuities. When the specimen contains many pores or the cracking and alteration of the material increase, the propagation velocity of the waves is decreased while the attenuation of the waves increases [12, 13]. Figure 6 shows the evolution of the propagation velocity of the ultrasonic waves. The speeds are measured on the samples in the dry state. It is easy to see that the velocity of ultrasonic waves increases markedly, ranging from 4147 to 4459 m / s. This increase in ultrasonic velocity is certainly due to the increase of the volume of the solid phases and the decrease of the porosity.



**Fig. 6.** Influence of the silica fume content on the speed of sound propagation

### 3.4. Dynamic Elasticity Module

From the curve shown in FIG. 7, there is a remarkable increase in the dynamic modulus of elasticity of the samples produced with the addition of the silica fume with respect to the control sample. The kinetics of rise of the modulus of elasticity observed is faster, this is due to the dual role of silica fume: the pozzolanic effect and the filler effect. The fact that the propagation velocity of the sound and the modulus of dynamic elasticity increases even at a content of 12%, where an increase in porosity has been observed, which is contrary to the scientific literature [14, 15, 16]. Can be explained by the fact that in both techniques only the open porosity of the mortar is measurable.



**Fig. 7.** Variation of the dynamic modulus of elasticity of mortars as a function of the silica fume content

#### 4. Conclusion

The porosity accessible to water is a very important parameter when interpreting durability studies such as freeze-thaw resistance and accelerated carbonation. In this study, it should be noted that the partial substitution of the cement by the silica fume in the mortars causes a decrease in the porosity accessible to the water and at the same time a decrease in the density.

The substitution of 9% cement with silica fume allowed us to find an optimum for which the open porosity is the lowest. Beyond 9%, the porosity increases slightly without affecting the mechanical characteristic (the dynamic modulus of elasticity), and this is due to the filler effect of the silica fume powders which will clog the space between the cement grains, which makes the concrete matrix more compact. The ultrasonic wave propagation test allows us to prove that there are closed pores that the water can not penetrate it, whether the saturation is by boiling or under vacuum. The high capacity capillary filling of the mortar by boiling with respect to vacuum saturation allowed us to conclude that the ASTM standard gives results that are more reliable than the RILEM standard.

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