

# WATER CONTENT AND ITS EFFECT ON THE DRYING BEHAVIOR OF REFRACTORY CASTABLES

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## ABSTRACT

The fluidity of refractory castables can be modified by adding water to their formulation. However, this can impact mechanical strength and permeability because it leads to higher interparticle spacing and porosity. The present work evaluated these effects on the drying behavior of castable compositions prepared with varying water contents. The results indicated two simultaneous effects: an increase in permeability and porosity promotes higher drying rates, while a reduction in mechanical strength heightens the risks of explosive spalling.

**Key words:** refractory castables, amount of water, drying behavior, permeability, mechanical strength, porosity.

## INTRODUCTION

The addition of water to refractory castable formulations is a well-known way to adjust the flowability of these castables [1]. Higher water content can reduce the consumption of energy during mixing and increase the flow value, even under restricted volumes (such as those prevailing in pumping and shotcreting). This behavior can be better understood through an analysis of the interparticle spacing parameter (IPS).

The IPS ( $\mu\text{m}$ ) can be regarded as indicative of the closeness of the fine particles in the castables' matrix (diameter  $< 100 \mu\text{m}$ ), taking into account the solid loading ( $\phi$ ), volumetric surface area (VSA, in  $\text{m}^2/\text{cm}^3$ ) and theoretical porosity ( $P_0$ ) of the powder packing [2]. It can be calculated (in microns) by the following equation:

$$IPS = \left( \frac{2}{VSA} \right) \left[ \left( \frac{1}{\phi} \right) - \left( \frac{1}{1-P_0} \right) \right] \quad (1)$$

As the solid loading decreases through the addition of water, the IPS values increase, resulting in a thicker coating of water surrounding each particle, and a greater ability to avoid collisions between the fine particles. Physically, this phenomenon can be regarded as a lessening of the difficulties that castables face under flow [3].

The IPS effects on the castable's structure can also continue after casting, as the increase of the water content promotes a reduction of the castable's mechanical properties. Because materials of low toughness such as ceramics are very sensitive to the number and size of defects, their elastic modulus and mechanical strength are drastically reduced by porosity. Besides the mechanical strength, the water addition can simultaneously affect the permeability, and as a consequence, the drying process of the castables.

Few systematic studies have investigated the effect of the water content on the drying stage of refractory castables because of experimental difficulties and the large number of variables involved (i.e., mass and energy transfer, phase transformations, mechanical response, etc.). For this reason, the work reported here evaluated how the porosity enhances the castable's permeability and how the reduction of mechanical strength induced by the water content affects the castable's drying behavior. Castable compositions were designed with identical particle size distributions (PSD) but with different water contents. A strong correlation was found between the water content and the risks of explosive spalling.

## EXPERIMENTAL PROCEDURE

### Formulation, mixing and curing

The refractory castable composition was formulated using Andreasen's particle packing model (coefficient  $q = 0.21$ ). The matrix of the castable (particles  $< 100 \mu\text{m}$ ) contained calcined aluminas (Almatis, USA, and Alcoa, Brazil) and 2 wt % of calcium aluminate cement (CA 14 – Almatis, USA). Electrofused aluminas (Elfusa, Brazil) were used for the aggregates (particles  $> 100 \mu\text{m}$ ). Citric acid ( $0.26 \text{ mg}/\text{m}^2$ ) was used as the dispersant.

Mixing was carried out in a paddle mixer. The same amount of water was added to each batch until the turning point was reached. After that, the addition of different amounts of water resulted in distinct formulations. This method is described in the literature [4]. Table I presents the amounts of water, the mean distance separating the matrix particles (IPS) and the solid matrix loading ( $\phi_{\text{matrix}}$ ) for each composition.

After mixing, the samples were cast and cured at a low temperature ( $8^\circ\text{C}$ ) and kept in a moisture-saturated environment ( $\sim 100\% \text{ RH}$ ) for 72 hours. This temperature was selected based on previous studies aimed at increasing the likelihood of explosive spalling [5]. For the

**Table I. Water content for castable processing, mean distance separating the matrix particles (IPS) and solid matrix concentration ( $\phi_{\text{matrix}}$ ).**

Water Content		IPS ( $\mu\text{m}$ )	$\phi_{\text{matrix}}$ (Vol %)
Vol %	Wt %		
(i) 13	3.82	0.0176	75.3
(ii) 15	4.51	0.0261	72.1
(iii) 17	5.24	0.0349	69.0
(iv) 20	6.39	0.0491	64.5

thermogravimetric tests, K-type thermocouples were inserted 1 mm below the surface to evaluate the samples' temperature during the tests.

### Permeability, porosity and mechanical strength

Cylindrical samples (25 x 75 mm) dried at 8°C for 96 hours in silica gel were employed for the permeability measurements. The non-Darcyan permeability constant ( $k_2$ ), given by the Forchheimer Equation, was obtained by polynomial fitting between the airflow and air pressure values [6].

Total porosity was measured in dried samples according to the ASTM C 20-87 (immersion test), using kerosene as the immersion liquid.

The castables' mechanical strength was measured through the splitting tensile strength test (ASTM C 496-90) under a constant load rate of 42 N/s. The tests were performed on wet (immediately after curing) and dried (96 hours in silica-gel) cylindrical samples (40 x 40 mm).

### Drying tests

The drying tests were performed in equipment developed by the authors' research group [5], and the samples tested immediately after curing. A continuous heating rate (10°C/min) was applied from room temperature up to the explosion of the sample. Furnace and sample temperature, and mass loss data were recorded at 5-second intervals throughout the tests.

The mass loss rate or drying rate during the tests was identified based on the time derivation of the parameter  $W_d$  (amount of mass loss) defined in Equations 2 and 3.

$$W_{di}(\%) = 100 \times \left( \frac{M_0 - M_i}{M_f} \right) \quad (2)$$

$$\frac{dW_{di}}{dt} (\%/min) = \left( \frac{W_{d(i+10)} - W_{d(i-10)}}{t_{(i+10)} - t_{(i-10)}} \right) \quad (3)$$

$W_d$  is the water loss during heating;  $M_i$  is the instantaneous mass recorded at time  $t_i$ ;  $M_0$  is the initial mass and  $M_f$  is final one, which, due to the spalling, was calculated considering the initial amount of water added during mixing [5].

## RESULTS AND DISCUSSION

Figure 1 presents the non-Darcyan permeability constant as a function of the water content and the mean distance between the matrix particles (IPS). The increase in water content (and hence in the IPS value) increased the castables' permeability considerably. A similar effect was observed in the samples' porosity leading to an expected reduction in mechanical strength (Figure 2). As the total porosity increased, the mechanical strength of both wet and dry samples decreased.

The effect of the water content on the castables' permeability, mechanical strength and porosity relates directly to the mean distance between matrix particles (IPS), as shown in Figure 3. Higher IPS values indicate that the particles are farther apart and the inter-particle forces are reduced [2,3]. As a result, after drying, a more permeable and porous structure was generated in castables with higher water content (Figure 1 and 2).

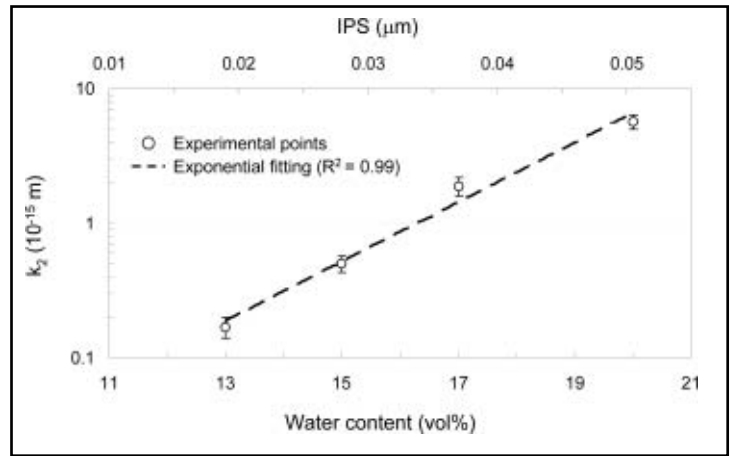


Figure 1. Non-Darcyan permeability constant ( $k_2$ ) as a function of the water content and mean distance between matrix particles (IPS).

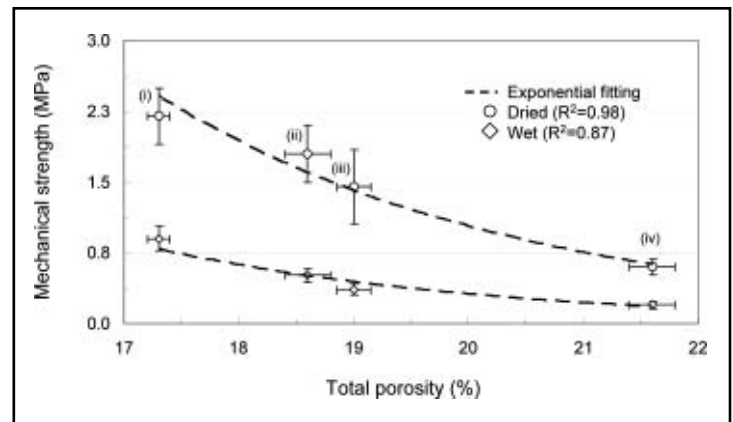


Figure 2. Mechanical strength of wet and dry samples as a function of total porosity.

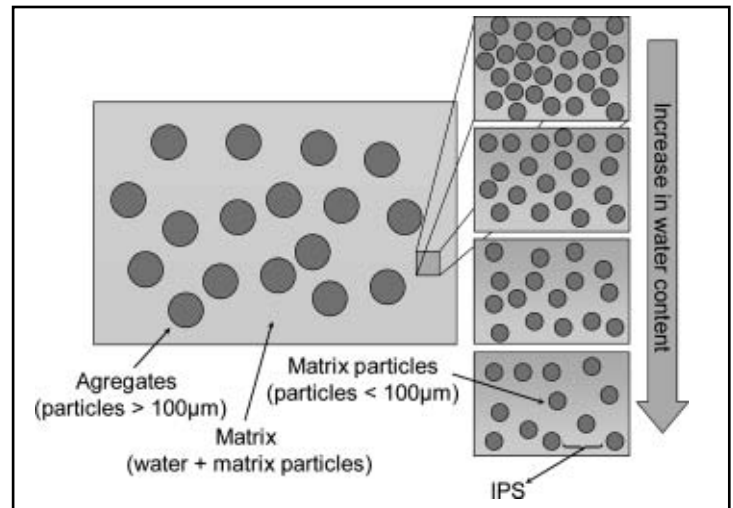


Figure 3. Effect of water content on the castable matrix.

The results presented here indicated two simultaneous effects: an increase in permeability and porosity promoted easier drying, whereas a reduction of the mechanical strength enhanced the risks of explosive spalling. An overall analysis of the data presented in Figures 1 and 2 does not indicate whether or not an increase in water content might be more detrimental to the castables' drying process.

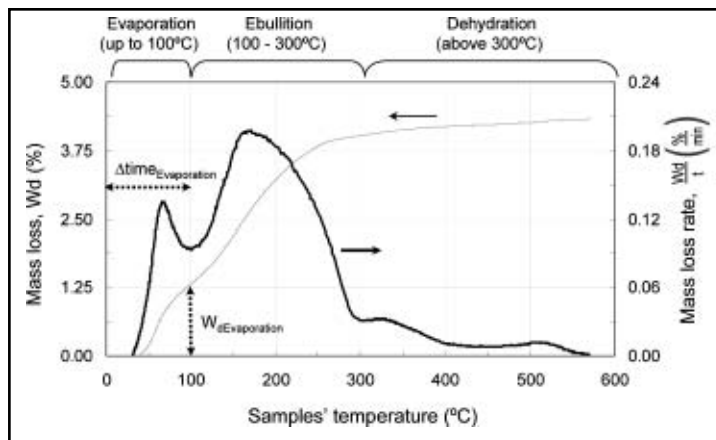
Thermogravimetric tests were therefore conducted to clarify the effect of permeability and mechanical strength on the drying steps.

The drying behavior of refractory castables was recently reported [5,7] to consist of three main stages: evaporation (from room temperature to 100°C), ebullition (from 100°C to 250-300°C) and dehydration (up to 250-350°C). In the present work, the Medium Evaporation Rate (MER) parameter was used to quantify the amount of water released as non-pressurized vapor during the evaporation stage. The MER was defined as:

$$\text{MER}(\%/ \text{min}) = \frac{W_{\text{dEvaporation}}}{\Delta \text{time}_{\text{Evaporation}}} \quad (4)$$

where,  $W_{\text{dEvaporation}}$  is the percentage of the total water content released during the evaporation step, and  $\Delta \text{time}_{\text{Evaporation}}$  is the extent of this stage (as shown in Figure 4).

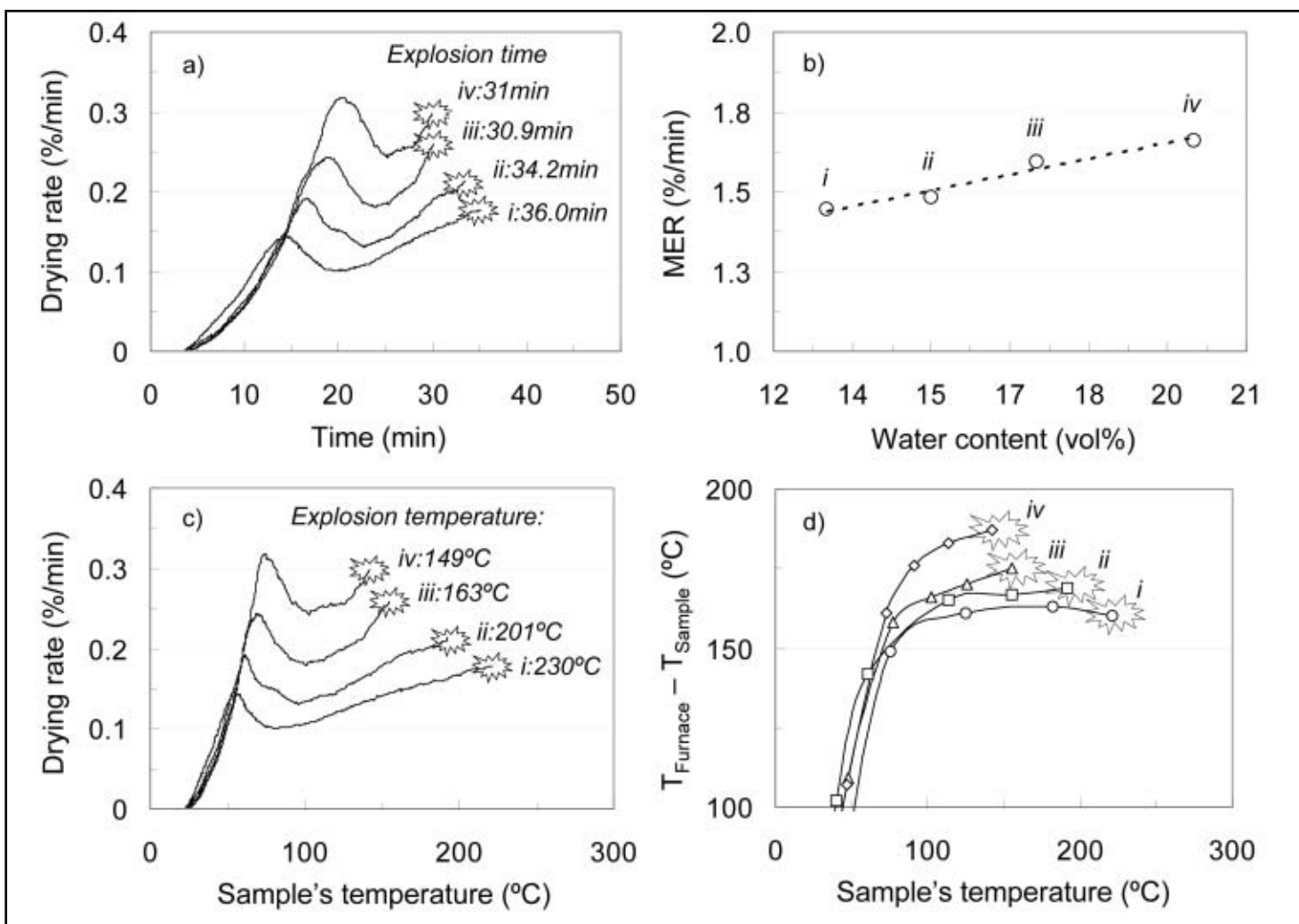
When the water boiling temperature is reached, pressurized vapor develops and the drying rate can be greatly augmented. For low permeability structures, the pressure generated can reach the material's ultimate mechanical strength and lead to an explosion. In the last drying stage, the chemically bonded water and hydroxyl-containing compounds (usually  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$  or  $\text{C}_3\text{AH}_6$  and  $\text{Al}(\text{OH})_3$  for



**Figure 4. Typical results of mass loss and mass loss rates for refractory castables [5,7].**

high alumina refractory castables) are decomposed. Figure 4 shows typical mass loss and mass loss rate curves for refractory castables, while Figure 5 depicts the results of the drying tests in samples containing different water contents.

Figures 5a and 5b indicate that the water content scales with the drying rate during the evaporation (MER) and with the time required



**Figure 5. a) Drying rate as a function of time, b) mean evaporation rate (MER), c) drying rate as a function of the samples' temperature, and d) thermal gradients between the sample and the furnace observed during drying of castables with different water contents (i = 13 vol%, ii = 15 vol%, iii = 17 vol% and iv = 20 vol%).**

to complete this stage. This implies that the larger amount of water to be removed delays the completion of the process, even though the water is released more easily due to the greater permeability. Figure 5c indicates that the increase in water content also leads to explosive spalling at lower temperatures. A reasonable explanation can be proposed based on the results illustrated in Figure 5d.

During the actual drying stages, unless the heating rate is very low or the sample too small, the castable's temperature does not follow that scheduled for the furnace [5,7]. During water removal, lower heating rates prevail at the surface of the samples due to the system's thermal inertia and endothermic liquid-vapor phase transformation. When all the water that could be removed by evaporation is eliminated, in order to reach the equilibrium, the solid is heated up faster than the furnace thus reducing the thermal gradient between them. Figure 5d illustrates how this thermal gradient increases with the water content exposing the samples to higher heating rates. As a direct consequence, a larger amount of vapor is generated, pressurizing the material. This fact, combined with the lower mechanical strength, leads to earlier explosions.


### CONCLUDING REMARKS

Increased water content in refractory castables seems to be prejudicial to their drying process. Despite their higher drying rates, castables prepared with higher water content exhibited a greater likelihood for explosive spalling due to their diminished mechanical properties and to the higher thermal gradient between the samples' surface and the furnace, a phenomenon observed at the end of the water evaporation stage. These findings strongly suggest that particle size distribution and dispersion adjustments are still the best way to increase the flowability of refractory castables.

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