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Experimental Study of the Morphogenesis Process of the Surface of a Cement + Sand Mixture During the Setting Process

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Abstract:

The morphology of the surface of the materials, can yield fundamental information that impacts the characteristics of the same, specifically in the process of setting of the binders that implies transformations in the density and viscosity. The objective of this work is to carry out an experimental study about the surface formation process during the setting of a cement-sand mixture, using the fractal dimension. The results showed that the experimental observations showed that the roughness and fractal dimension increased when the formation of pores occurs, likewise, the decrease of free water in the system responds to a first-order kinetics, while in the second stage It can be described through second-order kinetics..

Keywords **—Morphogenesis, fractal dimension, kinetics.**

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I. INTRODUCTION

The process of setting the construction materials involves changes in the density and viscosity of the suspension during the chemical crystallization reaction stage, which influence the final surface morphology. This process can be modified with external additives (Fernández, A., Morales, J., & Soto, F. 2016). The concrete can also be obtained from various recycled mixtures that are important for increasing sustainability (Fernández, A. et al. 2016). The majority of the evaluation tests require destructive mechanical processes that allow to know the characteristics of the same (Lizarazo, JM et al. 2016) and depend on both the components within the mixtures and the reactions that develop along the processes of setting; The reaction kinetics is mainly involved, for example, the first order which is the reaction where the speed depends on the concentration of the component, in this case the binder, while the second order kinetics (the one where the speed is equivalent to the square of the reagent used) (Paulini, P. 1990), (Hou, C., Zhu, W., Yan, B., Guan, K., & Du, J. 2018)

The surface of the solids is characterized by being irregular, property that can be quantified through the roughness (Ozol, MA 1978), related to the magnitude of fluctuations in the height of the interface, and the fractal dimension, which allows to estimate the specific surface area of the solid. This property depends on the individual components that have their own formal surface structure (Li, Z. Et al. 2019). The surface characteristics of the concrete may change due to the same property or the placement of surface elements among which is the Graffiti (Neto, E. et al. 2016)

In the case of cement + sand mixtures, experimental observations show that the roughness and fractal dimension increase when the formation of pores occurs, as well as with the decrease in the size of the sand particles. The models obtained and validated in this regard are based on stochastic formalism, and predict the relationship between the fractal dimension and the size of the particles in steady state, as well as the fact that the fractal dimension should decrease during the early stages of formation of the solid.

The present work aimed to carry out an experimental study about the surface formation process during the setting of a cement-sand mixture.

II. METHOD

A small portion of the sand + cement mixture was placed on a slide and photographs of the surface were taken every 5 minutes for a total time equal to 80 minutes. The photographs were taken with an optical microscope with an increase of ×1000.

The morphology of the corresponding surface each image was quantified through two fundamental parameters. The average intensity of the pixels of the corresponding 8-bit image, and the value of the fractal dimension of the average intensity profile of the pixels. The first variable was selected because the setting process involves a chemical reaction that leads to a change in surface coloration, while the second variable takes into account surface morphology.

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III. RESULTS AND DISCUSSION

When analyzing the images obtained, it was observed that during the first 20 minutes, approximately, there is a free water content on the surface that reflects the light, so these regions stand out and show an intense white color. From this time on, the content of free water is practically negligible, and the change in color, which ranges from a dark gray to a lighter color, can be attributed mainly to the crystallization process as such. For this reason, the setting process was divided into two stages, the first one related to the decrease of free water due to the chemical reaction, and the second one corresponding to the crystallization of the cement. Some of the images for each of these stages are shown in Figures 1 and 2.Figure 1. Images of the observed surface for T <20 min.

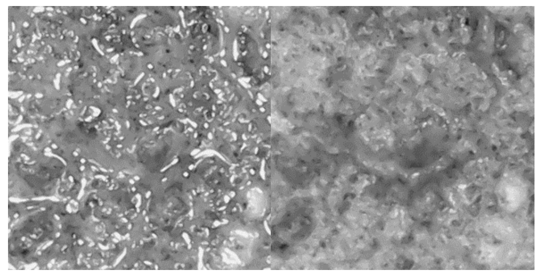

Figure 1 . Images of the observed surface for T <20 min

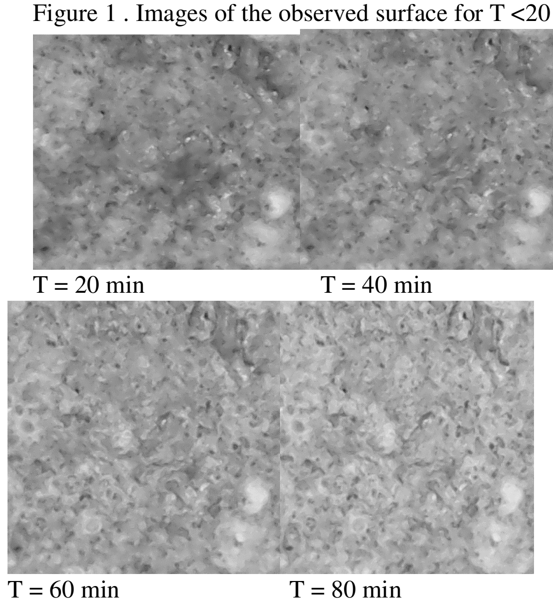

Figure 2. Images of the observed surface for T> 20 min

The fractal dimension of the profile is related to the reasonable assumption that the intensity of the pixels is proportional to the height of the surface, such that the lightest pixels are those that correspond to spatial positions of the surface where the height of the interface is bigger. For this reason, this variable is not suitable to analyze the morphology of the surface for T <20 min, since the intense white color is not due to the height of the surface, but to the reflection of the light by the free water that is found on the surface.

Figures 3 and 4 show the behavior of the intensity of the pixels and the fractal dimension with respect to the time observed experimentally.

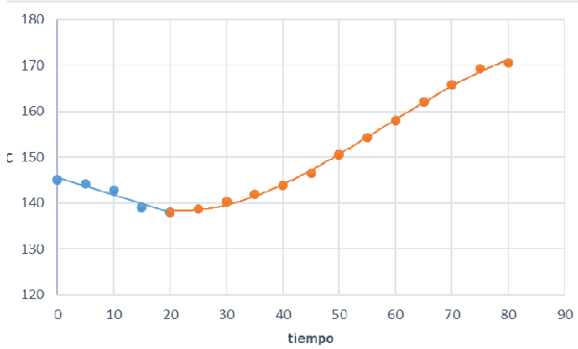

Figure 4. Behavior of the fractal dimension of profile f with respect to time

A. *Adjustment and experimental data*

The statistical adjustment of the behaviour of the fractal dimension with respect to the time made through the STATGRAPHICS program resulted in a linear model:

$$f = 1{,}26148 + 0{,}000162857t$$

With a correlation coefficient R2 = 22,06% and R2 (gl) = 14,98% y un valor de P = 0,1053 > 0,10 which indicates that there is no significant statistical relationship between both variables. This indicates that fluctuations in the fractal dimension of the profile are mainly due to the color changes caused by the chemical reaction, and not because there is a significant change in the interface's atura. This result corresponds to the results obtained in previous works, where it is considered that the height difference is caused by the distribution of size and spatial positions of the sand particles (which in this case do not change with time) and by the presence of the porosity caused by the inclusion of air, a phenomenon that was not observed in this experiment. Therefore, it is assumed that the surface area has a constant value and is quantified in this case through the average fractal dimension of the profile, which in this case is equal to 1,27.

In the case of the behavior of the average intensity of the pixels in the first stage, T <20 min , an exponential model was adjusted:

$$C = 145{,}47\mathbf{exp}(-0{,}0027t)$$

With a coefficient R2 (g.l.) = 92,79%, a standard error of the estimated 0.0059 and a value of P = 0.005 that indicates a significant correlation between both variables with 99% confidence. This is indicative that the overall kinetics related to the decrease in water in the system corresponds to a first order reaction, where the reaction rate constant is equal to 0.0027 min -1 .

The behavior of the average intensity of the pixels with respect to the time in the second stage is clearly non-linear, so a reaction kinetics of order 2 is assumed . The model adjusted by statistical techniques resulted in:

$$C = \frac{1}{(0{,}007891 - 0{,}000025t)}$$

With a value of R2 (gl.) Equal to 98,21%, an average error of the estimated equal 0,000067 and a value of P <0.01 indicating that there is a significant correlation between both variables. In this case the assumption of a second order reaction kinetics was appropriate, obtaining in this case a reaction rate constant equal to 0,000025 (pix.min) -1.

The behaviours of the results predicted by the adjusted models and the experimental results observed are shown in Figures 5 and 6.

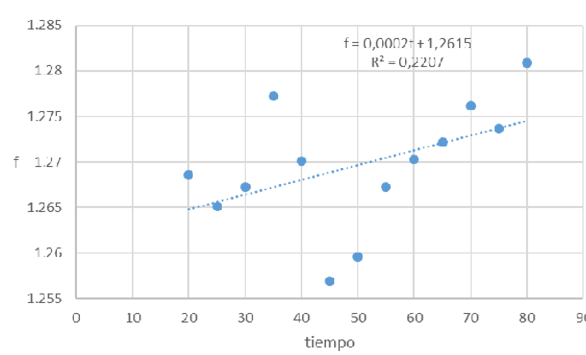

Figure 5. Comparison between results observed and predicted by the model adjusted for the first stage of the setting process

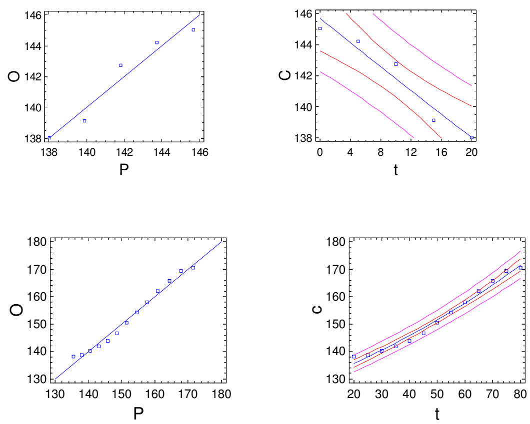

Figure 6. Comparison between experimental results and predicted by the model adjusted for the second stage of the setting process

From the results obtained, it is found that the model that describes the temporal behaviour of the intensity of the pixels is given by the differential equations:

$$\frac{dC}{dt} = -k_1 C \rightarrow t < \tau$$

$$\frac{dC}{dt} = k_2 C^2 \rightarrow t > \tau$$

Where $\tau$is the time required for the amount of free water on the solid surface to be practically equal to zero.

IV. CONCLUSIONS

Microscopic observation of the surface of a mixture of cement and sand resulted in a change in color, quantified through the average intensity of the pixels of the corresponding 8 bit which shows a decrease in a first stage and an increase in the second. This behavior is attributed in the first stage to the presence of free water in the system, which by reflecting the light increases this variable while in the second stage it is attributed to the crystallization process that takes place during the forge. From the statistical analysis of the observed experimental results, it was obtained that the decrease of free water in the system responds to a first order kinetics, while in the second stage it can be described through a second order kinetics.

ACKNOWLEDGMENT

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