

DETERMINATION OF CARBONATE CONTENT IN CLAYS AND CERAMIC COMPOSITIONS BY A PRESSURE SENSOR

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1. INTRODUCTION

The determination of the carbonate content in clays and ceramic compositions is a matter of great importance in ceramic tile manufacture. Owing to different problems relating to current equipment^[1], a system was developed for determining the carbonate content of powdered samples in which the pressure is measured of the CO₂ that is released when the carbonate reacts with hydrochloric acid, by means of a pressure sensor. The measuring system is based on previously developed assemblies^{[2], [3]} and has been registered as utility model no. U9.702.323.

2. EXPERIMENTAL

2.1. MATERIALS

A practically pure (99%) calcium carbonate (calcite) was used, together with three clays employed in Spanish tile manufacture: Villar, Chulilla and Mas Vell clay. The carbonate content of these samples, determined coulometrically (an analytical method of great accuracy and precision)^{[4], [5]}, was 3.0, 11.5 and 25.6% respectively.

[1]. SÁNCHEZ, E.; GINÉS, F.; AGRAMUNT, J.V. FELIU, C. *Determinación del contenido en carbonatos de materias primas y composiciones empleadas en la fabricación de baldosas cerámicas (II)*. (In press).

[2]. WILLIAMS, D.E. Soil Sci. Soc. Am. Proc., 127-129, 1948.

[3]. Consejo Superior de Investigaciones Científicas. *Calcímetro manométrico*. Int. Cl.: G01N 7/00. Invention patent ES 8405150.

[4]. ASTM D513-92.

[5]. SÁNCHEZ, E.; GINÉS, F.; AGRAMUNT, J.V. FELIU, C. *Determinación del contenido en carbonatos de materias primas y composiciones empleadas en la fabricación de baldosas cerámicas (II)*. (In press).

2.2. EQUIPMENT

Figure 1 schematically illustrates the assembly developed.

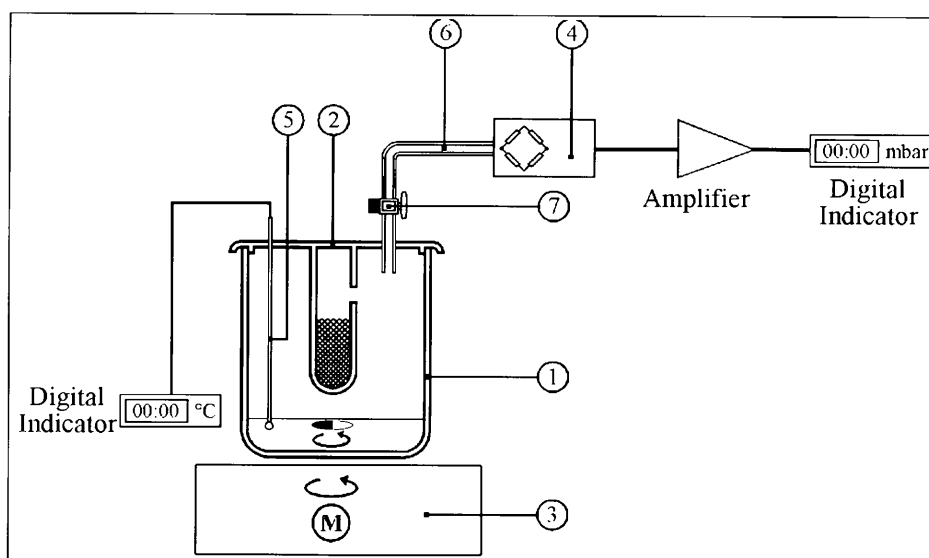


Figure 1. Schematic of the test set-up: (1) container, (2) airtight seal with HCl holder (3) magnetic stirrer, (4) pressure sensor, (5) temperature sensor, (6) flexible rubber tube, (7) valve.

Assuming that all the carbonate is calcic (PM=100), the perfect gas equation yields:

$$C(\%) = A \cdot \frac{P}{m \cdot T} \quad [1]$$

where C is the sample's carbonate content (wt%), m total mass of tested sample (g), T temperature (K), P pressure of released gas (at), while A is a constant depending on the volume of the recipient containing the sample, whose value is given by:

$$A = \frac{10^4 \cdot V}{R} \quad [2]$$

where V (ml) is container volume and R (82g·ml / mol·K) perfect gas constant.

2.3. EXPERIMENTAL PROCEDURE

The procedure adopted for the experimentation and testing conditions (sample quantity, water and hydrochloric acid) were the same as those employed with the Bernard calcimeter^{[1], [5]}.

3. RESULTS AND DISCUSSION

Initially, to calibrate the set-up, different amounts of calcite were used (C(≅100%).

- [1]. SÁNCHEZ, E.; GINÉS, F.; AGRAMUNT, J.V. FELIU, C. *Determinación del contenido en carbonatos de materias primas y composiciones empleadas en la fabricación de baldosas cerámicas (II)*. (In press).
- [5]. SÁNCHEZ, E.; GINÉS, F.; AGRAMUNT, J.V. FELIU, C. *Determinación del contenido en carbonatos de materias primas y composiciones empleadas en la fabricación de baldosas cerámicas (II)*. (In press).

The data satisfactorily fitted a straight line going through the origin of the coordinates. The value of V could then be calculated from the slope of the straight line, Eqs. (1) and (2) and test temperature (25°C):

$$m = \frac{A}{100} \cdot \frac{P}{T} \rightarrow A = \frac{100 \cdot T}{\text{slope}}; V = \frac{A \cdot R}{10000} \rightarrow V = 284 \text{ ml}$$

This value practically coincided with the actual volume of the container and the connections (≈ 280 ml), which was held steady throughout all the experiments.

On the other hand, the results of the determinations carried out on the three clay samples by the proposed technique were 3.1, 11.4 and 25.4%. On comparing these with the outcomes obtained with the coulometric method (Section 2.1), it can be observed that both sets of data match well, thus confirming the validity of the technique developed.