Dynamic Speckle Laser Technique for the Characterization of Electrotechnical-porcelain

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Abstract— This work analyzes the quality of two types of commercial electro-technical-porcelain using textural and physical-chemical methods: BET surface area and pore size by gas adsorption, X-ray powder diffraction; Electronic Microscopy and Energy dispersive X-ray analysis, Infrared Spectroscopy and their water adsorption capacity by the Dynamic Speckle Laser technique.

Experimental results showed the evolution of the speckle patterns during hydro-adsorption process, permitting to discriminate different behavior for each material. It was determined that it is possible to correlate changes in the speckle patterns with the porosity and chemical composition of materials.

1. INTRODUCTION

Dynamic speckle is a random scattering phenomenon occurring when coherent laser light illuminates an active surface [1]. The coherent electromagnetic waves beat on the detector producing light intensity variations. As a result, the surface of the samples appears to be covered by random tiny, bright and dark dots that fluctuate in time.

The study of the temporary evolution of the speckle patterns may provide an interesting tool to characterize the parameters involved in the sample dynamic processes. It has been used for several applications in biology, medicine and industry [2].

In this paper, we use this technique to analyze the hygroscopic properties of different types of porcelain for electro-technical purposes.

The typical porcelain consists of a mixture of three aluminum-silicates in a ratio: 50% kaolin, 25% quartz and 25% feldspar. This porcelain has basic electrical, mechanical, thermal and porosity properties, which naturally vary according to the composition of the mixture.

These materials are commonly used due to their insulating quality (e.g., insulation resistance) which basically depends on its porosity and hygroscopicity.

This work characterizes two types of commercial electrotechnical-porcelain using their water adsorption capacity by the Dynamic Speckle Laser technique (DLS), based on the textural, chemical composition, structural and spectroscopic characteristics.

2. EXPERIMENTAL

2.1. Materials and Methods

Dynamic speckle patterns were obtained by illuminating the samples with a 10 mW He-Ne laser and the images were recording with a CCD camera connected to a PC with an image digitizer. See Figure 1. To show the temporal evolution of speckle was used the Oulamara et al. method [3]. For each state of the adsorption process, 320 successive images of dynamic speckle are recorded, capturing 25 frames per second and select a column of them. These columns are building a pseudo-image of 320×240 so-called Temporary History Speckle Patterns (THSP). The activity of the sample changes of intensity (gray levels) in the horizontal direction. So, when a phenomenon shows low activity, time variations of the speckle pattern are slow and the THSP shows elongated shape. When the phenomenon is very active, the THSP resembles an ordinary speckle pattern. See Figure 2.

In our case, speckle high activity corresponds to the initial hydro-adsorption process. When this process is complete, the speckle activity is minimal. To establish a quantitative estimate of THSP, we use the moment of inertia of the co-occurrence matrix method [4].

Two different samples (P1 and P2) of commercial porcelains were selected.

Characterization of original porcelains was performed by Electronic microscopy using a Philips SEM 505 combined with semiquantitative analysis by EDS (Energy dispersive X-ray analysis), with an analyzer EDAX 9100.

Diagrams of X-ray diffraction powders were obtained by a Philips PW 1714 with a CuKα radiation and Ni filter from $2\theta = 5°$ to $60°$.

Infrared FTIR Spectra were performed by a Bruker IFSS 66 FT-IR equipment, from 4000 to 400 cm^{-1} wave-numbers.

Surface areas and porosity of porcelains were determined by physical N_2 adsorption at 77 K (BET method) using a Micromeritics apparatus ASAP 2010.

For DLS technique, 30 mg of each sample were wet with 10 µl distilled water. The experiment was monitored every two minutes from the beginning of the process and then every five minutes at the end of the process. The evolution was followed for one hour. Experimental environment parameters were: Temperature: 19°C, Humidity: 60%.

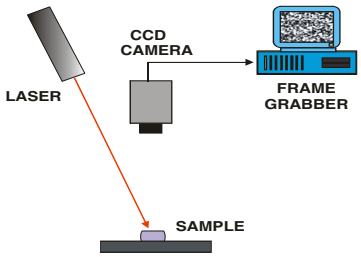

Figure 1: Dynamic speckle experimental set up.

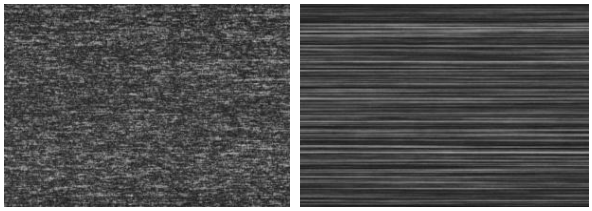

Figure 2: Temporal history of Speckle Pattern (THSP).

3. RESULTS AND DISCUSSION

3.1. Physical-chemical Analysis of Porcelain

Porcelain is the most important ceramic materials with multiple uses in electrical engineering. Typical composition comprises three aluminosilicate mixture in a ratio of 50% kaolin, 25% quartz and 25% feldspar. Its basic electrical, mechanical and thermal properties are according to the composition of the mixture. One of the characteristics that determine the quality of these materials is impermeability to water and gases depending on its porosity, as well as its Si/Al ratio. It has been considered that a high Si/Al surface is associated with increased Brønsted acidity, resulting in higher hygroscopicity.

Table 1 shows the textural characteristics by the BET method to P1 and P2. Specific surface area and pore size were slightly but P1 showed the less porosity level.

Table 1: Textural properties by the BET Method for P1 and P2.

Sample	S_{BET} (m^2/g)	Pore volume (cm^3/g)	Pore size (Å)
P1	0.5664	0.001314	92.78
P2	0.6594	0.001775	107.65

Table 2 shows EDS chemical data (% element is the relative weight percent of the elements, Na, Mg, K, Al, Si, Ti and Fe), and Si/Al ratio for porcelain samples.

The EDS analysis revealed different composition and Si/Al ratio while SEM Micrographs (Figures 3 and 4) allowed observing the presence of porous in the porcelain surfaces.

Figure 5 shows comparative XRD patterns for P1 and P2. It is possible to observe that the main component is quartz (SiO_2) in both samples, according to the main peaks located at $2\theta = 20.5°$ and 26.3° and Alumina (α-Al_2O_3) at $2\theta = 25.5°$ and 35.1°. In XRD diagram of P2 is possible to identify an additional peak of feldspar located at $2\theta = 22°$.

Table 2: SEM-EDS chemical data for P1 and P2 porcelain samples.

Element Wt%	P1	P2
Na	0.81	0.80
Mg	0.41	-
Al	23.40	17.35
Si	63.26	75.41
K	5.87	4.78
Ti	1.97	-
Fe	4.28	1.66
Si/Al	2.70	4.34

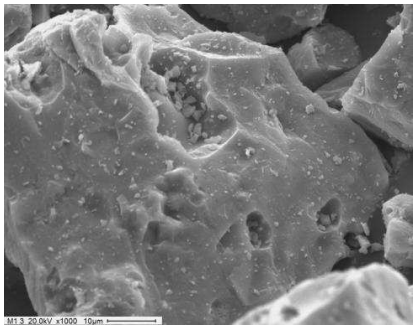

Figure 3: SEM Micrograph of P1 Magnification ×1000, scale bar 10 µm.

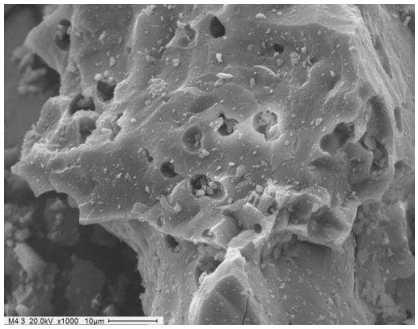

Figure 4: Micrograph of P2 Magnification ×1000, scale bar 10 µm.

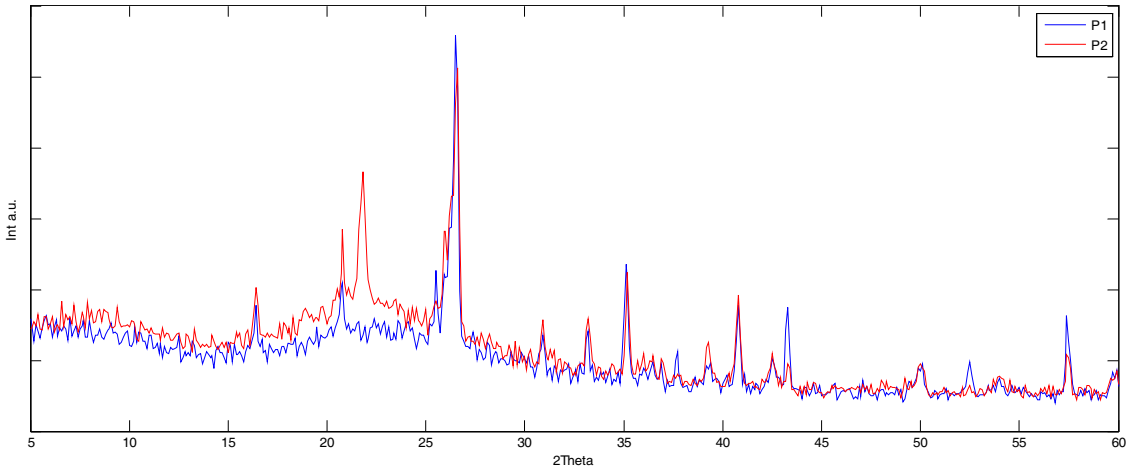

Figure 5: XRD diagrams for P1 and P2 samples (range $2\theta = 5°$ to $60°$).

Figure 6 shows comparative FTIR spectra for porcelains P1 and P2. Using this technique is very difficult to differentiate between the two samples studied. Since the vibrational spectroscopy allows analyze functional groups or molecular units, which in this study are similar as Si-O-/AL-O- or Si/Al-OH, -OH etc. In these spectra are observed no bands corresponding to OH stretching, in the high frequency range. In the next range around 1000 cm^{-1} intense antisymmetric stretching bands of the tetrahedral groups TO_4 (T = Si or Al) appears.

3.2. DLS Technique: Hydro-adsorption Analysis of Porcelain

Analyzes were performed by Dynamic Speckle Laser during adsorption of water from both samples. DLS curves provided a quantitative measurement of this process using the Inertia Moment (IM) method.

Figure 7 shows the DLS hygroscopic behavior of porcelain. After initial fall of speckle activity, stabilization of it was reached. It can be observed that the porcelain P2 (sample b) showed the

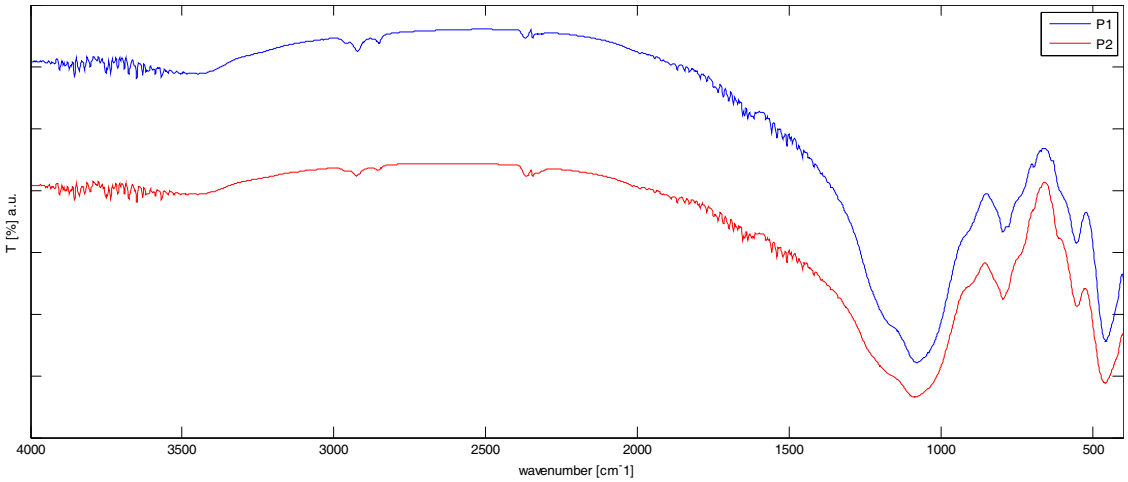

Figure 6: FTIR spectra for P1 and P2 samples (range 4000–400 cm^{-1}).

shortest stabilization time (about 200 sec), indicating a rapid hydro-adsorption process, P1 (sample a) presented the highest IM and stabilized time after 600 sec. Compared with Tables (1 and 2) it is possible to observe that the lower Si/Al, and porosity corresponds a greater IM in the H_2O adsorption process.

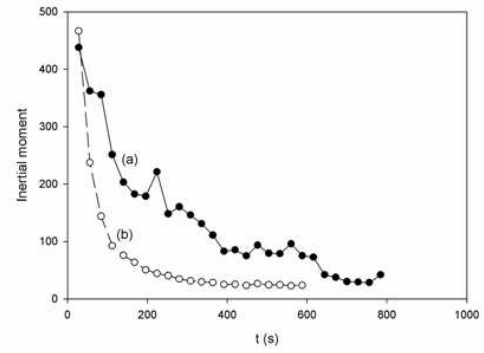

Figure 7: Dynamic speckle measurements, (a) P1, (b) P2.

4. CONCLUSIONS

The DSL technology provides a simple, more efficient and nondestructive tool with respect to other physicochemical techniques to compare different quality ceramic materials, which is important to estimate hygroscopicity.

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