## METHODOLOGY FOR FORMULATING RED CERAMIC FLOOR TILE BODIES BY STUDIES OF DILATOMETRIC BEHAVIOUR

## S. Cava, C. A. Paskocimas, H. R. Pereira, E. Longo

Universidade Federal de São Carlos Departamento de Química Laboratório Interdisciplinar de Eletroquímica e Cerâmica Rod. Washington Luis, km 235 - C.P. 676 - CEP 13565-905 São Carlos - SP Brazil

A methodology is proposed for formulating ceramic bodies by means of dilatometric behaviour studies, in which the starting clay raw materials (referenced C, L and V) contain high calcite concentrations, as shown by the mineralogical analysis detailed in Table 1, determined by chemical analysis and X-ray diffraction.

	Quartz	Kaolinite	Illite	Vermiculite	Calcite
С	33.90	24.11	11.03	14.12	16.84
L	39.84	26.16	11.78	14.48	7.74
V	37.91	31.20	13.66	13.53	3.70

Table 1 – Mineralogical composition of the clay raw materials with high calcite concentrations.

Formulations of ceramic floor tile bodies produced by dry milling were optimised, to illustrate the effect of the calcite present in the clays in different concentrations, keeping the other components more or less constant. Thermal dilatometric tests determined the thermal behaviour of the different clays, as shown in Figure 1.



Figure 1 – Plots of thermal dilatometric analysis corresponding to clays C, V and L.

A direct comparison of the three resulting curves shows how calcite delays the densification process, as evidenced by a retention between 900°C and 1150°C in the dilatometric curve of C. The results follow the expected densification tendency perfectly as a function of the quantity of CaO contained in the three studied clays. The most pronounced sintering was observed in V, which contained the smallest quantity of calcite, and the least pronounced sintering for C, which had the largest calcite concentration. L exhibited intermediate behaviour.

The BET method was used to determine clay specific surface area. The results were as follows: V:  $34.69 \text{ m}^2/\text{g}$ , C:  $18.59 \text{ m}^2/\text{g}$ , L:  $16.58 \text{ m}^2/\text{g}$ . The specific surface area of V is a favourable parameter, and is related to the high reactivity of this raw material. However, a product consisting exclusively of this clay would present problems with regard to dimensional control, since at temperatures in the range 1100 to  $1120^{\circ}$ C, V undergoes linear shrinkage of about 7.5%. Another aspect to be considered is the need for rational use of the three raw materials, as they are found in very close lying areas. Once the individual effect of each was determined, two series of compositions were prepared combining 1) V and L, and 2) V and C. Using V as a reference, the combined effect of the clays was established in pairs. Table 2 lists the group of studied compositions.

	VL-1	VL-2	VL-3	VL-4	VC-1	VC-2	VC-3	VC-4
V	80	60	40	20	80	60	40	20
L	20	40	60	80				
С					20	40	60	80

Table 2 –	Series	of	compositions	using	V	as a reference.

Using dilatometry, a study was carried out to identify the effect of combining raw materials L and C on the shrinkage behaviour of V. Figure 2a presents a group of dilatometric curves found for the series V/L, where the addition of 20% L reduces the shrinkage of V from 5.8% to 3.7%, and the addition of 20% V in L raises shrinkage from

1.5% to 2.2%, both results being established at a temperature of 1100°C. Thus, in the compositions VL-1 to VL-4, a transition in behaviour is observed, mainly due to V for behaviour marked by the character of L.



Figure 2 – Dilatometry plots corresponding to the series a) V/L, b) V/C.

The dilatometric curves corresponding to the series V/C are shown in Figure 2b. The results show that the addition of 20% C in V reduces shrinkage from 5.8% to 3.8%, causing practically the same effect that L had on V. As the concentration of C in the composition rises, a clear tendency to delay the densificación process in the temperature range from 900°C to 1150°C is observed, as in the case of the system V/L. These results confirm the effect of CaO on slowing the sintering process in ceramic tile body compositions.

The adopted methodology was found to be appropriate for developing ceramic compositions, allowing quick, accurate identification of the best combination of raw materials from a small number and volume of samples The use of two firing curves with retention and practically identical heating rate temperature changes, differing by only 40°C at peak temperature, was designed to verify the susceptibility of physical and mechanical properties at peak temperature. Thus, with this procedure, the ideal processing conditions can be established for the clay systems involved.