Ca-bearing secondary raw materials in traditional ceramics: XRD to tackle technological properties

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Since decades, traditional ceramics stimulated research studies on the possibility to optimize a formulation by re-balancing natural raw materials, such as clays, feldspar and quartz, or by introducing an alternative raw material, with the aim of improving the final product properties. Nowadays, research is pushed by sustainability, to reduce the natural raw materials consumption and the energy costs related to the firing process, or by geopolitical reasons, as in the case of feldspar, which was recently inserted in the EU critical raw materials list.
This study was focused on the effects of cattle bone ashes (hereafter CBA) in partial replacement of albite (i.e. Na-feldspar) fluxing agent at 5 and 2.5 wt.% into the Vitreous China sanitary-ware formulation [1]. CBA were selected in light of their strong fluxing role upon firing.
Both CBA-bearing formulations allowed us to reach target porosity, represented by the water absorption required by the UNI-EN 997 regulation, even firing at lower temperature compared to the reference formulation. However, the risk of overfiring the product was observed as well as a 10% reduction in flexural strength.
X-ray powder diffraction technique was applied to evaluate differences in qualitative and quantitative phase analyses between the reference formulation and the two CBA-bearing formulations. To better comprehend the phase evolution, three rectangular plates (6 x 4 x 1 cm) were prepared for each formulation and finally fired at three different peak temperatures (i.e. 1140, 1180 and 1220°C).
Qualitative phase analysis results disclosed the presence of mullite, residual quartz and albite were observed in all samples, with the latter crystalline phase detected only in samples fired at 1140°C peak temperature. Moreover, Ca-rich feldspar (possibly andesine-type [2]) was detected in both CBA-bearing formulations fired at a peak temperature equal/higher than 1180°C (see Figure 1 as an example).
Samples were then spiked with 10 wt.% of corundum as internal standard to perform quantitative phase analysis by the Rietveld-RIR method [3] using the GSAS-II program [4]. These results revealed that the glassy phase development became more rapid when the starting CBA content in the formulation increased, an effect that explains the reduction in the sintering window of the ceramic body. Again, the mullite crystalline phase content was progressively reduced when the starting CBA content in the formulation was increased, an effect that can explain the reduction in the mechanical performances.
These findings highlight the importance of the powder diffraction technique for designing novel and more sustainable traditional ceramic materials.

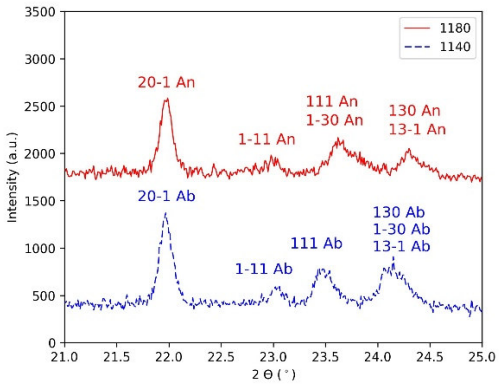


Figure 1. XRD pattern of formulation with 5 wt.% CBA fired at 1140°C (in blue) and 1180°C (in red) peak temperatures. "*Ab*" label refers to albite type of feldspar while "*An*" label refers to andesine type feldspar.

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