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An SEM investigation of the pozzolanic activity of a waste catalyst from oil refinery

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The most active phase of the fluid catalytic cracking (FCC) catalyst, used in oil refinery, is zeolite-Y which is an aluminosilicate with a high internal and external surface area responsible for its high reactivity. Waste FCC catalyst is potentially able to be reused in cement-based materials - as an additive - undergoing a pozzolanic reaction with calcium hydroxide ($Ca(OH)_2$) formed during cement hydration [1-3]. This reaction produces additional strength-providing reaction products i.e., calcium silicate hydrate (C-S-H) and hydrous calcium aluminates (C-A-H) which exact chemical formula and structure are still unknown. Partial replacement of cement by waste FCC catalyst has two key advantages: (1) lowering of cement production with the associated pollution reduction as this industry represents one of the largest sources of man-made CO_2 emissions, and (2) improving the mechanical properties and durability of cement-based materials. Despite these advantages, there is a lack of fundamental knowledge on pozzolanic reaction mechanisms as well as spatial distribution of porosity and solid phases interactions at the microstructural level and consequently their relationship with macroscopical engineering properties of catalyst/cement blends.

Within this scope, backscattered electron (BSE) images acquired in a scanning electron microscope (SEM) equipped with Energy-Dispersive Spectroscopy (EDS) and by X-ray diffraction were used to investigate chemical composition of hydration products and to analyse spatial information of the microstructure of waste FCC catalyst blended cement mortars. For this purpose mortars with different levels of cement substitution by waste catalyst as well as with different hydration ages, were prepared. The waste FCC catalyst used is produced by the Portuguese refinery company Petrogal S.A.

The BSE images of the waste FCC catalyst blended cement mortars showed that (see Figure 1):

- FCC catalyst particles in the cement composite materials are homogeneously dispersed in the matrix;
- Significant amounts of Ca incorporated by FCC catalyst particles (observed in X-ray map of elements and EDS spectra) reveal their ability to react with CH and, therefore, their pozzolanic nature;
- Ca content in the FCC pozzolanic particles increases with hydration time;
- Pozzolanic reaction in FCC catalysts seems to occur almost uniformly all over the particle's volume: (1) in general, X-ray element maps do not show a reaction front associated to a gradual penetration of Ca, and (2) EDS examinations in different areas across the section, even for young cement mortars, show that Ca content is usually almost uniform. This fact may be attributed to the three-dimensional internal structure of zeolite consisting of a network of interconnected tunnels and cages where diffusion can easily occur;
- Pozzolanic reaction products appear to be confined within the catalyst particles original boundaries and should not induce expansion.

Major findings in this investigation reveal that BSE-SEM images analysis coupled with EDS and X-ray diffraction data provide a fundamental insight into the reaction mechanisms underlying the FCC catalysts pozzolanic activity. This knowledge is important to make possible the use of waste FCC catalyst as a steady supply for cement industry. This will make a significant contribution to produce environmental-friendly blended cements.

References

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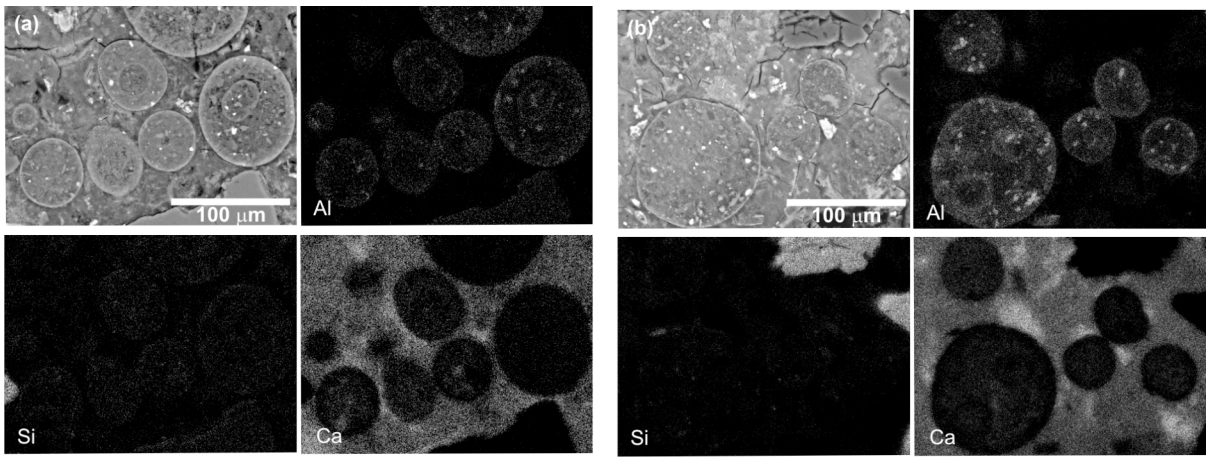

Figure 1. BSE-SEM images and corresponding X-ray maps of (a) 7 and (b) 28 days cured mortar with 15% of waste FCC catalyst incorporation.

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