



Microscopic characterisation of zeolite particles synthesised in a soil polluted by Cu or Cd and stabilised with a coal fly ash-treatment

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Direct zeolite synthesis in polluted soils can be promoted in order to reduce heavy metal (HM) mobility and availability by entrapping metal precipitates inside the forming minerals. This process can provide remediation technologies with a more effective alternative to the use of zeolites as cation exchangers since such a process leads to the formation of scarcely soluble metal hydroxide/oxide precipitates which can even become occluded inside the structure of the growing zeolite crystals. Coal fly ash, a by-product of coal combustion, was successfully used to remediate polluted soils by acting as a cheap source of Si and Al for direct zeolite synthesis.

In this research, the formation of different types of zeolites (X, P, and A) was observed by treating soil samples, artificially contaminated by high concentrations of Cu or Cd (15 mg/g), with fused coal fly ash at 30 and 60°C, over a period of one year. The formed zeolites were characterised for their amount, structure, chemical composition and size. To accomplish this survey, beside quantitative X-ray diffraction analyses (XRD), an automated single particle analysis method using Electron Probe X-ray Microanalysis (EPXMA) was employed for the first time for direct zeolite characterisation in soil samples. The obtained results provided new information for assessing the role of HM in zeolite crystallisation in coal fly ash-treated soils.

In the Cu-contaminated soil, the formation of zeolite X and P was observed, and their composition was found to be not significantly different from that of the same zeolites usually synthesised directly from coal fly ash. Zeolite X crystals synthesised in the

presence of Cu were generally larger (ca. 10 μm) than those formed in the presence of Cd (ca. 5 μm) and, in their turn, they were larger than those synthesised in the absence of metal ions contamination (ca. 2 μm). Both Cu and Cd contamination caused a delay in the beginning of zeolite synthesis at 60°C and, at this temperature, the presence of Cd also significantly hindered zeolite P crystallisation. Zeolite A was formed only at 60°C in the presence of Cd ions and, surprisingly, this zeolite was characterised by almost the same chemical composition of zeolite X. In general, Cu and Cd ions seemed to drive zeolite synthesis toward the formation of sodalite unit-based zeolite X and zeolite A, even at 60°C.

In comparison with the results obtained for the unpolluted soil, HM contamination, while not significantly changing the type and amount of zeolites attained by treating the soil at 30°C, may interfere in the zeolite synthesis process carried out at 60°C. In this situation, the presence of high concentrations of Cu or Cd not only may have caused a delay in the beginning of zeolite formation but also the preferential crystallization of zeolites with larger constitutive building units such as zeolite X and zeolite A. Finally, it is likely that the presence and nature of metal precipitate occlusions inside the forming zeolite minerals might have favoured the preferential synthesis of certain zeolitic structures over others.

All these information can contribute to the development of new remediation technologies exploiting the process of direct zeolite synthesis in polluted soils.