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Inactivation of exhausted zeolite-based Pb-exchangers

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The high selectivity of clinoptilolite toward lead cation is well known. Lead uptake from clinoptilolite has been widely investigated under different experimental conditions. However, problems related to the management of exhausted Pb-exchangers, that can become a pollution source, have not received the same attention. Thermal treatments could represent a way to achieve the inactivation of exhausted Pb-exchangers; indeed, heating affects the structure and, consequently, the properties of zeolites. The present research has been started to evaluate the effects of heating on a Pb-clinoptilolite, observed in terms of Rehydration Capacity (RC) and lead release variations, and phase modifications.

Starting from a Sardinian rock containing 66% of clinoptilolite (XRPD analysis, Topas software), a material with 88% of zeolite has been obtained through a beneficiation process involving autogenous comminution, and dry and wet separations. The zeolite has been previously conducted in sodium then in lead form by ionic exchange, obtaining a Pb-clinoptilolite end-member simulating an exhausted exchanger in its condition of highest dangerousness (ICP analyses). Eight distinct aliquots of this material have been subjected to two hour thermal treatments from 200 to 900°C. Lead release tests have been carried out at 25°C by exchange in 0.5 M K-solution (pH 8.2) on unheated and heated materials. Exchanged lead has been determined through ICP analyses of the powders, whereas the metal remained in solution has been measured by AAS analyses of eluates.

XRD analyses have shown that the structure of Pb-clinoptilolite is detectable until 600°C. Up to this temperature, RC trend has evidenced a linear decrease from 100% to 69%.

The untreated material has exchanged about 64% of its initial Pb2+ content (2.28 meq/g), but only 78% of the exchanged metal has remained in solution, probably due to the formation of lead monodentate and bidentate complexes at the tested conditions. The exchanged metal has shown a weak decline until 400°C (54% with respect to the initial lead content), then a strong reduction up to 700°C, where release has been close to zero as the zeolite structure has been destroyed. Exchanged lead and RC have shown similar trends with respect to heating temperature. A complete inactivation of the exchanger has been obtained after a heating at $T \geq 800$°C, with Pb-feldspar and silica polymorphs nucleation.

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