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Preparation and Study of Maghemite-Zeolite Composites

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Zeolites are hydrated aluminosilicates of the alkaline and alkaline-earth metals. Natural zeolites forming the corresponding group of tectosilicate mineral subclass, due to their specific crystal chemical characteristics providing the unique ion-exchange and molecular sieve properties, are known as effective adsorbents and catalysts. The zeolites are highly rigid under dehydration as well as under various aggressive surroundings actions [1]. Ion-exchange of transition elements is one of the most common applications of zeolites [2]. Adsorption of Co^{2+} , Zn^{2+} , Mn^{2+} Pb²⁺, Cu^{2+} , Cr^{3+} and Fe³⁺ from waste waters has been intensively studied on natural [3,4] and synthetic [5] zeolites. Modification of zeolites by iron oxides and hydroxides provides important innovation for better applications [6,7]. Covering of zeolite surface by magnetic nanoparticles provides, together with adsorption of ions, also strong magnetic properties. Such modified zeolites possess better adsorption capacity for cations of heavy metals as well as anions [8]. Adsorption of anions on natural zeolite is difficult due to the negative charge of the zeolite lattice, which was tested with Al modified synthetic zeolites [9].

Natural zeolite (clinoptilolite) has been magnetically modified through maghemite (γ -Fe₂O₃) nanoparticles by precipitation route at various reaction conditions. An effect of the precipitation temperature, the weight ratio of Fe/zeolite and the interaction time on the magnetic and surface properties of maghemite-zeolite composites was monitored by room-temperature ⁵⁷Fe Mössbauer spectroscopy, TEM and BET surface area measurements. A decrease in reaction time and the Fe/zeolite ratio leads to the formation of smaller particles of γ -Fe₂O₃ while lowering the precipitation temperature results in the larger crystallites of maghemite. The reflection of the precipitation temperature as the key variable in the sorption properties of composites was tested with selected heavy metal ions. The sample prepared at the highest temperature reveals maximum sorption capacity for Pb^{2+} 97.2 mg/g at concentration of Pb^{2+} 400 mg/L, which is much higher than that commonly observed for natural zeolites. Good ability for sorption of anions was demonstrated with AsO_4^{3-} , which offers new applications of such modified zeolites. The composite sample with the best sorption properties was characterized by TEM, SEM (Fig. 1), XRD, in-field ⁵⁷Fe Mössbauer spectroscopy and magnetic measurements. They reveal that maghemite nanoparticles form aggregates, which are sorbed on zeolite inhomogenously. The particles are about 15 nm in diameter with spinel structure as documented through the ratio of tetrahedral and octahedral positions occupied by Fe^{3+} cations being 1/3. FC/ZFC curves confirm strongly interacting superparamagnetic particles with a blocking temperature of 230 K.



Fig 1 SEM images of modified zeolite at 85 °C (normal and composition mode).

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