

# **Synthesis and characterization of a novel hybrid material titanium amino tris(methylenephosphonic acid) and its application as a cation exchanger**

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## **Abstract**

In the present work, a novel hybrid ion-exchange material was synthesized by a sol-gel route, by treating titanium tetrachloride with claw-type amino tris(methylenephosphonic acid) (ATMP) to give titanium amino tris(methylenephosphonic acid) (Ti-ATMP). Ti-ATMP was characterized by elemental analysis (ICP-AES and C, H, N analysis), spectral analysis (FTIR), thermal analysis (TGA), XRD, SEM, and EDX spectroscopy, including physicochemical and ion-exchange characteristics. The equilibrium exchange of transition-metal ions (viz., Cu<sup>2+</sup>, Ni<sup>2+</sup>, Co<sup>2+</sup>, Zn<sup>2+</sup>) and heavy-metal ions (viz., Pb<sup>2+</sup>, Cd<sup>2+</sup>, Hg<sup>2+</sup>) with the H<sup>+</sup> ions contained in ATMP was studied at constant ionic strength and varying temperatures, and various thermodynamic parameters such as the equilibrium constant (K), standard Gibbs free energy ( $\Delta G^\circ$ ), enthalpy ( $\Delta H^\circ$ ), and entropy ( $\Delta S^\circ$ ) were evaluated. The Nernst-Planck equation was used to study the ion-exchange kinetics, and various kinetic parameters, namely, the self-diffusion coefficient (D<sub>0</sub>), energy of activation (E<sub>a</sub>), and entropy of activation ( $\Delta S^*$ ), were evaluated under conditions favoring a particle-diffusion-controlled mechanism. Metal-ion adsorptions with variations in concentration and temperature were studied using the Langmuir and Freundlich adsorption isotherms. The distribution coefficient (K<sub>d</sub>) and breakthrough capacity (BTC) values for transition- and heavy-metal ions were determined. Based on K<sub>d</sub> (mL·g<sup>-1</sup>), the selectivity order for metal ions toward Ti-ATMP was found to be Cu<sup>2+</sup> (19820) > Zn<sup>2+</sup> (3280) > Co<sup>2+</sup> (2630) > Ni<sup>2+</sup> (2390) among transition-metal ions and Pb<sup>2+</sup> (3590) > Cd<sup>2+</sup> (2340) > Hg<sup>2+</sup> (610) among the heavy-metal ions. The elution behavior of these metal ions was studied using different acids and electrolytes. A study on regeneration and reuse of the ion exchanger Ti-ATMP shows that it is effective up to five cycles without much decline in performance, which indicates that Ti-ATMP has good potential for use as a cation exchanger.

**Keywords:** methylenephosphonic; Freundlich