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Preparation and characterization of natural and pillared clays for catalytic wet peroxide oxidation of 4nitrophenol

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This work deals with the evaluation of two natural clays (NCs) extracted from Karatau (KNC) and Akzhar (ANC) deposits (located in the Zhambyl region of Kazakhstan) to prepare pillared clays (PILCs) for catalytic wet peroxide oxidation (CWPO) of 4-nitrophenol (4-NP), used as model pollutant. NCs were washed with HCl 1 M and then pillared using a solution containing Fe, Cu and Zr. NCs and PILCs were characterized by Electron Microprobe (EMP), X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Transmission Electron Microscopy (TEM), as detailed elsewhere [1,2]. EMP confirms the cation exchange of the Ca contained in the NCs (Ca > 15%) in the form of calcite (according to DRX and FTIR), by polycations of the pillaring solution (Ca < 2% for the PILCs). TEM reveals that the impregnation of polycations on the washed NCs also take place, coupling with its pillarization. CWPO runs were performed following the methodology and operational conditions described in previous works [1,2]. All materials show catalytic activity, since the H_2O_2 is consumed (Fig. 1A) to oxidize the 4-NP. Both KPILC and APILC, prepared from KNC and ANC, respectively, allow the complete removal of 4-NP after 4 h, whereas the conversion of 4-NP was less than 20% with the NCs (Fig. 1B). TOC conversions higher than 60 % were achieved with PILCs after 8 h. The subtraction of the theoretical TOC contribution of 4-NP from experimental TOC allowed to observe the formation of oxidazable intermediate compounds (maximum value of $TOC_{experimental}$ - $TOC_{4\text{-}NP}$ at 1 h of reaction), which are oxidized to form refractory products (Fig. 1C). Based on these contributions of the TOC, a kinetic model based on TOC lumping into three blocks ($TOC_A \rightarrow TOC_B \rightarrow TOC_C$, corresponding to the initial TOC of 4-NP, oxidazable intermediates and refractory products, respectively) was developed for the NCs and PILCs, predicting suitably the evolution of 4-NP, H_2O_2 and TOC in the CWPO of 4-NP (Fig. 1).

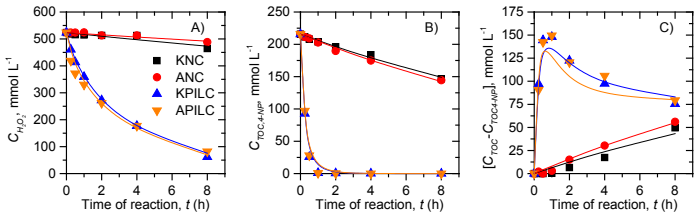

Fig.1. Evolution of (A) H_2O_2, (B) theoretical TOC contribution of 4-NP and (C) the sum of TOC_B + TOC_C (symbols as experimental data and curves as predicted values by the developed kinetic model)

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