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**Preparation of Al/Fe-PILC clay catalyst from concentrated precursors: process intensification towards scaling-up**

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**RESUMEN**

Structural modification of bentonites by pillaring with Al/Fe mixed oxides provides them excellent catalytic response in catalytic wet peroxide oxidation (CWPO) of organic pollutants present in wastewaters but also potentially in surface waters for production of drinking water. However, in order to apply this process to real-scale, the scaling-up of the preparation of the catalyst from high-concentrated metal and clay precursors is mandatory. This work was then devoted to compare the preparation of highly-concentrated Al/Fe interlayering solution by three methodologies: (i) addition of elemental aluminum on the starting chlorides; (ii) microwave-assisted hydrolysis of the metals via urea decomposition; and (iii) basic hydrolysis by addition of sodium hydroxide under diluted conditions, used as reference method. Other series of solids (scale 50 g) were prepared for comparison of three methods of clay-interlayering: (a) addition of interlayering solution on ethanol clay-suspension (25 % w/v); (b) the same as (a) but using water instead of ethanol; and (c) clay powder directly added on interlayering solution (without previous clay-suspension). Al/Fe-PILC from methods (i) and (c) displayed the best physicochemical and catalytic properties based on XDR, XFR, textural properties from N<sub>2</sub> isotherms, H<sub>2</sub>-TPR, DRIFTS and catalytic performance measured in CWPO phenol degradation (26 mg/dm<sup>3</sup>) at 25°C.