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Effect of surfactants on the characteristics and activity of Mg-Al Hydrotalcite in Knoevenagel condensation reaction

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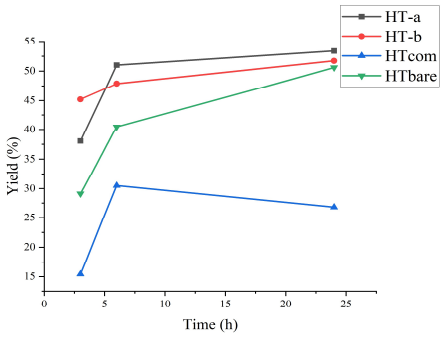
[Background and purpose] Hydrotalcite (or layered double hydroxides) is a solid inorganic layered material consisting of layers of divalent and trivalent metal hydroxides. This material acts as a heterogeneous base catalyst and is widely appreciated for various organic transformations [1]. The crystallinity and morphology of hydrotalcite can be influenced by varying synthesis conditions and methods by adjusting synthetic parameters, colloid mill rotation, dispersion-precipitation techniques [2,3]. In this study, surfactants were introduced during the synthesis of hydrotalcite under constant conditions to examine their effects on properties, morphology and reactivity in Knoevenagel condensation reaction. The study also aimed to identify surfactants that positively influence the activity of hydrotalcite, proposing a cost-effective and simpler method to enhance the catalytic activity of this material.

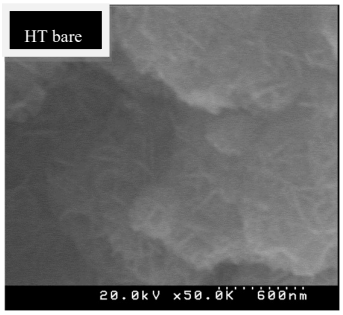
[Experimental results] Benzyldimethyltetradecyl ammonium chloride, a cationic surfactant, positively impacts the reactivity of Mg-Al hydrotalcite (HT) in Knoevenagel condensation reaction of furfural, as evidenced by high yields (above 50%) and increased reaction speeds compared to other samples. From XRD measurement method, the crystallite size of the Benzyldimethyltetradecyl ammonium chloride -modified hydrotalcite (as HT-a) exhibits a size distribution ranging from 7 to 10 nm in the (003) plane. SEM images also demonstrate that hydrotalcite synthesized in the presence of this surfactant exhibits a reduction in grain size, optimizing the exposure of active base sites, thereby enhancing accessibility and reactivity.

[Conclusions] The findings indicate that hydrotalcite not only can still form and crystallize in the presence of the surfactant but also its increasing reactivity by the cationic surfactant. The increase in reactivity of hydrotalcite is primarily attributed not to the crystal size, but to the morphology and particles size induced by the impact of the surfactant.

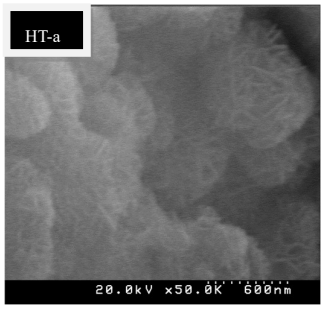


HT-a: HT/Benzyldimethyltetradecyl ammonium chloride
HT-b: HT/Hexadecyltrimethylammonium bromide
HTbare: Non surfactant modified hydrotalcite
HTcom: Hydrotalcite commercial

Figure1 Time-based reaction progression



a) SEM images of non-surfactant modified hydrotalcite (HT bare)



b) SEM images of HT/Benzyldimethyltetradecyl ammonium chloride

Figure2 Morphology comparison

[References] [1]M. Lakshmi Kantam, B.M.Choudary, Ch.Venkat Reddy, K. Koteswara Rao and F. Figueras, *Chem. Commun.*, 1998, 1033-1034. [2]Y.Zhao, F.Li,R.Zhang ,D.G.Evans and X.Duan, *Chem. Mater.,* 2002,14, 4286. [3]S.Abello and J. Perez- Ramirez, *Adv. Mater.*, 2006, 18, 2436.

[Keywords] Surfactant, hydrotalcite, Knoevenagel condensation reaction