Heterometallic calcium–alkali metal aryloxides as catalysts for the solvothermal alcoholysis of nylon-6 waste

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Streszczenie

In this study, we report the synthesis of homometallic and heterometallic calcium aryloxides: $[Ca(sal\text{-}Me)_2(MeOH)]_n$ (**1**), $[Ca_3(sal\text{-}Me)_6(MeOH)_2]$ (**2**), $[Ca_2Li_2(sal\text{-}Me)_6(THF)_2]$ (**3**), $[Ca_2Na_2(sal\text{-}Me)_6(MeOH)_4]$ (**4**), and $[Ca_2K_2(sal\text{-}Me)_6(MeOH)_4]$ (**5**). Compounds **1** and **2** were obtained *via* direct reaction of metallic calcium with methyl salicylate (Hsal-Me) in methanol (MeOH). A similar synthetic route, incorporating one equivalent of an alkali metal (M′ = Li, Na, K) and tetrahydrofuran (THF), was used to obtain the heterometallic derivatives **3–5**. A central focus of this work is the synthesis of heterometallic Ca^{2+}–M'^{+} complexes, of which only 13 examples have been previously documented. A detailed investigation of the reaction pathways led to the identification of intermediate compounds: $[Ca_3(sal\text{-}Me)_6(THF)_4]$ (**2a**), $[CaLi_6(sal\text{-}Me)_8]$ (**6**), and $[Ca_3Na_4(sal\text{-}Et)_{10}(Hsal\text{-}Et)_2]$ (**7**) (Hsal-Et = ethyl salicylate). Additionally, reactions conducted under atmospheric moisture conditions yielded the uncommon pentanuclear complexes $[Ca_4Na(\mu_5\text{-}OH)(sal\text{-}Et)_8(MeOH)]$ (**8**) and $[Ca_4K(\mu_5\text{-}OH)(sal\text{-}Et)_8(EtOH)]$ (**9**). Compounds **1**–**9**, along with previously reported $[M'_6(sal\text{-}Me)_6]$ (M'^{+} = Li^+(**10**), Na^+(**11**), K^+(**12**)), $[Mg_2(sal\text{-}Et)_4(EtOH)_2]$ (**13**), $[Zn_4(sal\text{-}Me)_8]$ (**14**), $[M_2M'_2(sal\text{-}Me)_6(THF)_x]$ (M^{2+} = Mg^{2+}; M'^{+} = Li^+(**15**), Na^+(**16**), K^+(**17**); M^{2+} = Zn^{2+}; M'^{+} = Li^+(**18**), Na^+(**19**), K^+(**20**) ; x = 0, 2, 4), and $[Mg_4Na_2(sal\text{-}Me)_6(sal)_2(THF)_4]$ (**21**) were evaluated as catalysts for the chemical recycling of polyamide waste. Among them, the heterometallic catalysts **3**, **6**, and **18–20** exhibited the highest catalytic activity in the methanolysis of nylon-6 at 220 °C. Particular emphasis is placed on the reaction conditions and the high efficiency of the catalysts.

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