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Intercalation of alkaloids of *Cassia auriculata* linn. into h^+- and al^{3+}-exchanged montmorillonite clays

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Montmorillonite [$(Na,Ca)_{0.33}(Al,Mg)_2Si_4O_{10}(OH)_2.nH_2O$] is a layered aluminosilicate mineral having an expandable interlayer space of 1-2 nm. The inherent net negative charge on outer surface of the layers is counteracted by the presence of exchangeable cations in the interlayer nanospace. Cation-exchanged H^+- and Al^{3+}-montmorillonite (MMT) clays show Lewis and Bronsted acidities and have the potential to trap alkaloids. In this study, the cation-exchanged clays were used to trap alkaloids present in the *Cassia auriculata* Linn. leaf extract; *C. auriculata* is a medicinal plant having anti-diabetic, antioxidant, antimicrobial, anticancer, anthelmintic, anti-inflammatory and analgesic activities. However, it contains hepatotoxic pyrrolizidine alkaloids and their removal may enhance the medicinal value of the plant. Leaf extracts of *C. auriculata* were obtained by successive extraction of dried leaf powder into hexane and methanol using a Soxhlet apparatus. Alkaloids present in the leaf extracts were analyzed qualitatively by TLC and quantitatively by ion-pair formation (IPF) method coupled with UV-Vis spectroscopy. H^+- and Al^{3+}-MMT clays were prepared by stirring purified Na^+-MMT in aqueous solutions of HCl and $AlCl_3$, respectively. Alkaloid intercalated H^+- and Al^{3+}-MMT clay composites were prepared by stirring suspensions of H^+- and Al^{3+}-MMT clays in solutions of *C. auriculata* leaf methanol extract in 50% MeOH/CH_2Cl_2. The amounts of alkaloids present in the supernatant before and after the intercalation were determined using the coloured ion-pair complexes formed between the protonated alkaloids and methyl orange at an optimum pH of 4.4. UV-Vis measurements of the ion-pair complexes were carried out at 428 nm using brucine as the standard alkaloid. All the clays and their alkaloid composites were characterized using X-ray diffraction (XRD) and Fourier-Transform Infrared (FT-IR) spectroscopy. The intercalation of alkaloids in the MMT clay composites was further confirmed by releasing of the trapped alkaloids in NaOH solution, which was quantified by IPF/UV-Vis method. The results indicated intercalation of 97% and 98% of *C. auriculata* alkaloids of methanolic leaf extract into H^+- and Al^{3+}-MMT clays, respectively. When the alkaloid-intercalated clay composites were stirred in 0.5 M NaOH, slow releasing of the trapped alkaloids was observed, reaching a maximum of 59% release (after 3 h) from H^+-MMT and 50% release (after 4 h) from Al^{3+}-MMT clay composites. It can be concluded that the alkaloids present in the leaves of *Cassia auriculata* can be successfully intercalated into cation-exchanged MMT clays and the intercalated alkaloids can be released slowly upon alkali treatment.