

## SYNTHESIS, CHARACTERISTICS AND APPLICATION OF COMPOSITE MATERIALS

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### Introduction

Recent studies have been targeted towards the generation of functional materials especially the so-called nanocomposite materials, which have potential uses as catalysts, selective photochemical and photochromics system, control release material and quantum well structures. One candidate of the layered solids that can be molecularly engineered to form new nanocomposite material is hydrotalcite-like materials or the so-called layered double hydroxide (LDH) from anionic clay family. Synthetic LDHs have many uses in various industrial applications and can be used as a host for the formation of nanocomposites materials. The objectives of this study were to synthesis nanocomposite and the host (layered solids) materials, and characterise the properties of the resulting materials.

### Materials and Methods

The LDHs were synthesised according to the method of Reichle (1986). The LDH formed was cooled, centrifuged, thoroughly washed and dried in an oven at 120 °C, overnight and kept in a sample bottle for further use and characterisation. The synthesis of LDHs nanocomposites was done by spontaneous self-assembly method. A solution containing LDHs formation salts was prepared and the pH was adjusted to about pH=10. The concentration of the guest molecules was adjusted, aged for several days at 80 °C in an oil bath or by microwave-assisted technique, so that good crystallinity of the nanocomposite can be obtained. The resulting material was centrifuged, thoroughly washed and dried in an oven at 120 °C, overnight and kept in a sample bottle for further use. Characterisation was done by XRD, SEM, FTIR, surface analyser and uv-visible spectrophotometer.

### Results and Discussion

Powder x-ray diffraction (PXRD) patterns of the nanocomposites showed that they afford good crystallinity and expansion of the basal spacing of the host was observed, from 11 to 26Å depending on the size and orientation of the guest molecule. This shows the occurrence of the intercalation process of the guest in the LDH interlayer to form nanocomposite materials. However, a broader and asymmetric of the 003 reflections compared to LDH host was also observed. This indicates that disorder may also be present in the stacking of the layer, which lowered the different in the relative intensities. PXRD patterns for calcined nanocomposite in a temperature range of (100-1000) °C was obtained to gather more information about the stability of the nanocomposite on heat treatment. The layered structure of the nanocomposite was collapsed when the nanocomposite was heated at certain temperature, depending on the host and the guest molecules and resulting in formation of an "amorphous-like" or metal

oxide new phase. Qualitative and quantitative colour analysis of the nanocomposites was determined by using a Perkin-Elmer spectrophotometer Lambda 20 with CIE L\*a\*b\* colour space reading. The colour difference,  $E_{ab}^*$ , was calculated by  $\Delta E_{ab}^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ . In general, the colour different,  $\Delta E_{ab}^*$  of the heat-treated nanocomposite relative to the precursor increased with the calcination temperature. This shows that the tendency of the calcined nanocomposite to become lighter in colour increased as the calcination temperature increased. Expulsion or degradation of the colour bearing material is the only reason for colour reduction. A major change in the morphology of the calcined nanocomposite was observed at 1000°C. A mixture of amorphous and "porous-like" structures appeared (Mohd Zobir et al. 1998a). Nanocomposite of an ionic clay-dye of Mg-Al layered double hydroxide-Evan's blue system synthesised by microwave-assisted method showed that the physico-chemical properties of the resulting materials were similar to those prepared by conventional method. The advantage of shorter aging time was observed for the microwave-assisted method in preparing nanocomposite materials (Mohd Zobir et al. 1998b). Due to the "memory effect" property, the regeneration of Mg-Al LDH from the heat-treated Mg-Al LDH was obtained when the heat-treated Mg-Al LDH was dye-adsorbed, and the basal spacing of around 7.9Å was maintained. The surface area increased by about 75%, from 88.7 in Mg-Al LDH to 154.7m<sup>2</sup>/g in heat-treated Mg-Al LDH, but the micropore surface area decreased by about 33%, from 10.6 in the former to 7.1 m<sup>2</sup>/g in the later. Batch kinetic study showed that these adsorbents can efficiently adsorbed the dye with maximum adsorption capacities ranged between 16 to 32 mg of dyes per g of adsorbent, with heat-treated Mg-Al LDH showed a better adsorbent than the precursor. The adsorption can be better fitted to Langmuir than Freundlich adsorption isotherms, although the difference was not significant.

### Conclusions

This study showed that nanocomposites of layered double hydroxide-dyes could be synthesised by spontaneous self-assembly of the inorganic and organic phases from homogeneous aqueous solutions either by conventional or microwave-assisted techniques. Optical spectra of the nanocomposites showed that the absorption peaks is blue-shifted with respect to the dyes, due to nanostructured behaviour of the dye in the interlayer. The nanocomposites afford well-crystallised layered structure with expanded basal spacing, and thermally more stable than the host. The heat-treated or the as synthesised LDHs were found to be suitable for colour adsorption for water treatment.

### References

- Mohd Zobir bin Hussein, Zulkarnain Zainal and Pua Eu Kok, 1998a. Microwave-assisted synthesis of anionic clay-dye nanocomposite of mg-al-layered double hydroxide-Evan's blue system, paper presented at the *Malaysian chemical congress '98 (mcc'98)* at Kota Kinabalu Sabah, 24-25 November, 1998.
- Mohd Zobir bin Hussein, Zulkarnain Zainal and Sivajothi Nadarajah. 1998b. Properties of heat-treated zn-al layered double hydroxide-congo red nanocomposite, *Science International (Lahore)*. 10(3).
- Reichle, W.T. 1986. Synthesis of anionic clay minerals (mixed metal hydroxides, hydrotalcite). *Solid state ionic*. 22: 135-141.