The Malaysian Journal of Analytical Sciences, Vol 11, No 1 (2007):48-56

SYNTHESIS OF ZINC-ALUMINIUM-PAMOATE NANOCOMPOSITE USING DIRECT CO-PRECIPITATION AND ION-EXCHANGE METHODS

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Key words: Organic-inorganic hybrid; pamoic acid; layered double hydroxide, nanocomposite

Abstract

The intercalation of pamoate anions into the interlamellae of zinc-aluminium layered double hydroxide (LDH) was carried out by two different methods; direct co-precipitation and ion-exchange methods. The Zn/Al molar ratio, R of the LDH was set at 2 - 5 (x = 0.33 – 0.16, where $x = [Al^{3+}]/[(Zn^{2+} + Al^{3+})]$ and the concentration of pamoate ion used was fixed at 0.02 M. A sharp, intense and symmetrical peak could be observed from the XRD patterns for all the samples synthesized by direct co-precipitation method, indicating a well-ordered nanolayered structure in the nanocomposite. However, the XRD patterns of the nanocomposite prepared by ion-exchange method show that the second peak for (006) reflection are broad and not symmetrical due to the overlapping of two peaks at $2\theta = 10°$, which indicated the presence of LDH with nitrates as the counter anion. The percentage of PA intercalated into the interlamellae of the nanocomposite prepared by ion-exchange method is in the range of (29 – 45)%, which is lower than the one prepared by direct co-precipitation method, (43 – 51)%. This shows that direct co-precipitation method yielded nanocomposites with higher loading of the organic moiety and better crystallinity compared to the ion-exchange method.

Abstrak

Penyisipan anion pamoat ke dalam lapisan hidrosida berlapis ganda (HBG) zink-aluminium telah dilakukan menggunakan dua kaedah berbeza iaitu kaedah pemendakan bersama dan penukar ion. Nisbah molar Zn/Al, R bagi HBG ditetapkan pada 2 –5 (x = 0.33 –0.16), di mana $x = [Al^{3+}]/[(Zn^{2+} + Al^{3+})]$ dengan kepekatan ion pamoat 0.02 M. Puncak yang tajam, berkeamatan tinggi dan simetri dapat diperhatikan daripada analisis pembelauan sinar-X (XRD) bagi kesemua sampel yang disediakan menggunakan kaedah pemendakan bersama, ini menunjukkan bahan nanokomposit berada dalam susunan yang teratur. Manakala, corak XRD bagi nanokomposit yang disediakan dengan kaedah penukar ion menunjukkan puncak kedua (006) yang lebar dan tidak simetri, ia mungkin disebabkan oleh pertindihan dua puncak pada $2\theta = 10°$, yang menunjukkan kehadiran HBG dengan nitrat sebagai pengimbang cas. Peratus PA yang tersisip di antara lapisan nanokomposit yang disediakan dengan kaedah penukar ion adalah antara (29 – 45)%, yang mana ia lebih rendah berbanding dengan yang disediakan dengan kaedah pemendakan bersama, (43-51)%. Kajian ini menunjukkan kaedah pemendakan bersama menghasilkan nanokomposit dengan penyisipan bahan organik yang lebih tinggi dan kehabluran bahan yang lebih baik berbanding menggunakan kaedah penukar ion.

Introduction

Recently, there has been rapid expansion on the development of organic-inorganic hybrid type of materials for various purposes and applications such as catalyst precursors or supports, adsorbents, anion exchanger and in the pharmaceutical industry [1-4]. The use of layered double hydroxide (LDH) as host material for the synthesis of organic-inorganic nanolayered has received great interest. LDH is available as both naturally occurring minerals and synthetic materials. The general composition of these intercalation materials can be represented as $[M^{2+}_{1-x} M^{3+}_{x} (OH)_2][(A^{n-})_{x/n} \cdot yH_2O]$, where M^{2+} and M^{3+} represent metallic cations such as Mg^{2+}, Ca^{2+}, Cu^{2+}. Mn^{2+}, Ni^{2+}, Zn^{2+} and Al^{3+}, Cr^{3+}, Fe^{3+}, Mn^{3+}, Co^{3+}, respectively. A^{n-} is an anion of charge n such as Cl^-, CO_3^{2-}, NO_3^-, SO_4^{2-}, or organic anions. x value is equal to the molar ratio of $M^{3+}/(M^{2+} + M^{3+})$. The crystal structure consists of positively charged brucite-like octahedral hydroxide basal layers, which are electrically neutralized by the interlayer anions and water molecules occupying interlayer spaces [5].

The intercalation of organic compounds into LDH has been investigated from a wide range of scientific and practical interests, especially because of the nanostructures of the resulting intercalation compounds [5]. From the comparison of various methods made by Carlino, (1997), it was found that the co-precipitation and rehydration methods were the most effective to intercalate both mono- and dicarboxylic acids into layered double hydroxide or their oxides. These two methods gave single phase reaction products as indicated by PXRD. The direct ion-exchange method was found to be the least effective owing to the carbonate anion, which is present in the LDH.

Methods of comman basic ion-exchange, reconstruction in aqueous solution and co-precipitation have been studied by Dupin *et al*. (2004) to determine the best-suited process of insertion of dichlophenac into Mg-Al-LDH. The reconstruction method led to the greatest increase with an enlargement of the interlayer space of about 3.9 Å. The co-precipitation method is not well adapted to intercalate dichlophenac into Mg-Al-LDH because it gave unstructured and a poorer phase of materials. The PXRD pattern of material prepared with the use of glycerin during the ion-exchange appears quite similar to the parent-LDH. No modification of the interlayer distances was observed, thus indicating the insertion process did not occur in the LDH.

In this paper, we would like to report on the physicochemical properties of the nanocomposite obtained from the intercalation of pamoate ion i.e. the anion of pamoic acid, 1,1'-methylene-bis-[2-hydroxy-3-naphthoic acid] into the interlamellae of Zn-Al LDH using two different methods namely direct co-precipitation and ion-exchange methods. These two methods were used to determine the best-suited process for the intercalation of pamaote anion to form nanocomposite with well-ordered layered structure. The formation of the new nanocomposite by intercalating pamaote anion into Zn-Al LDH is able to improve its properties and has the potential to be used as a controlled release formulation for slow drug release in pharmaceutical industry.

Experimental

The synthesis of the nanocomposites, ZAPRs was done by direct co-precipitation method. A mother liquor containing Zn^{2+} and Al^{3+} cations with Zn/Al initial ratio (R_i = 2, 3, 4, 5) and PA was prepared and the pH was adjusted to about pH 7. The concentration of PA was 0.02 M and the reaction was carried out with stirring under nitrogen atmosphere. The solution was aged for 18 h in an oil bath shaker at 70 °C. The resulting precipitate was centrifuged, thoroughly washed and dried in an oven at 70 °C for 3 days and kept in a sample bottle for further use and characterizations.

A similar method was adopted for the preparation of Zn-Al-LDH with nitrate as the intergallery anion (ZAL) by omitting the addition of PA solution in the mother liquor. In the synthesis process of ZAPXs, about 0.5 g of Zn-Al-LDH of Zn/Al molar ratio, R_i = 2 to 5 was added into 125 mL decarbonated water and aged in an oil bath shaker at 70 °C for 18 hours. Then, 0.02 M of pamaote solution was added into the Zn-Al-LDH solution and stirred for an hour. The mixture of solutions was then aged in an oil bath shaker at 70 °C for 18 hours. The precipitate formed was cooled, centrifuged, washed several times and dried and kept in a sample bottle for further characterization.

Powder x-ray diffraction (PXRD) patterns of the samples were obtained by a Shimadzu Diffractometer XRD-6000, using filtered CuK_α radiation. FTIR spectra were recorded by a Perkin-Elmer 1750 Spectrophotometer. KBr pellet containing 1 % sample was used to obtain the FTIR spectra. The surface morphology of the samples was observed by a scanning electron microscope (SEM), using JOEL JSM-6400. CHNS analyser, model EA 1108 of Finons Instruments was used for CHNS analyses. UV-visible technique was used to determine the percentage composition of PA in ZAPRs and ZAPXs using a Perkin-Elmer UV-visible Spectrophotometer model Lambda 20. Surface characterization of the materials was carried out by nitrogen gas adsorption-desorption at 77 K using a Micromeritics ASAP 2000. Samples were degassed in an evacuated-heated chamber at 120 °C, overnight.

All chemicals used in this synthesis were obtained from various chemical suppliers and used without any further purification. All solutions were prepared using deionized water. In the discussion, ZAPR2 is used to name the nanocomposite with initial Zn/Al molar ratio 2, prepared by direct co-precipitation method. ZAPX2 is the nanocomposite with initial Zn/Al molar ratio 2, prepared by ion-exchange method. ZAL is Zn-Al-NO_3-LDH and PA is pamoate anion.

Results and Discussion

Powder X-Ray Diffraction

Figures 1(a) and (b) show the PXRD patterns of the Zn-Al-PA nanocomposites (ZAPRs and ZAPXs), prepared using various initial molar ratio of Zn/Al, R_i = 2, 3, 4, 5. ZAPR2 is the nanocomposite prepared by direct co-precipitation method using initial Zn/Al molar ratio 2 whilst ZAPX2 is the nanocomposite prepared by ion-exchange method with initial Zn/Al molar ratio 2 and so on. The optimum pH for the synthesis of nanocomposite was pH 7. A sharp, intense and symmetry peak could be observed from the XRD patterns of the nanocomposite prepared by direct co-precipitation method as shown in Figure 1(a), indicated a well-ordered layered structure was formed. A good crystallinity of well ordered layered structure of nanocomposite was determined by the presence of intense, sharp, and symmetry peaks of 003 and 006 reflections. The peaks of the PXRD patterns obtained in Figure 1(b) were not as intense as the PXRD patterns of the nanocomposite synthesized by direct co-precipitation method (Figure 1a).

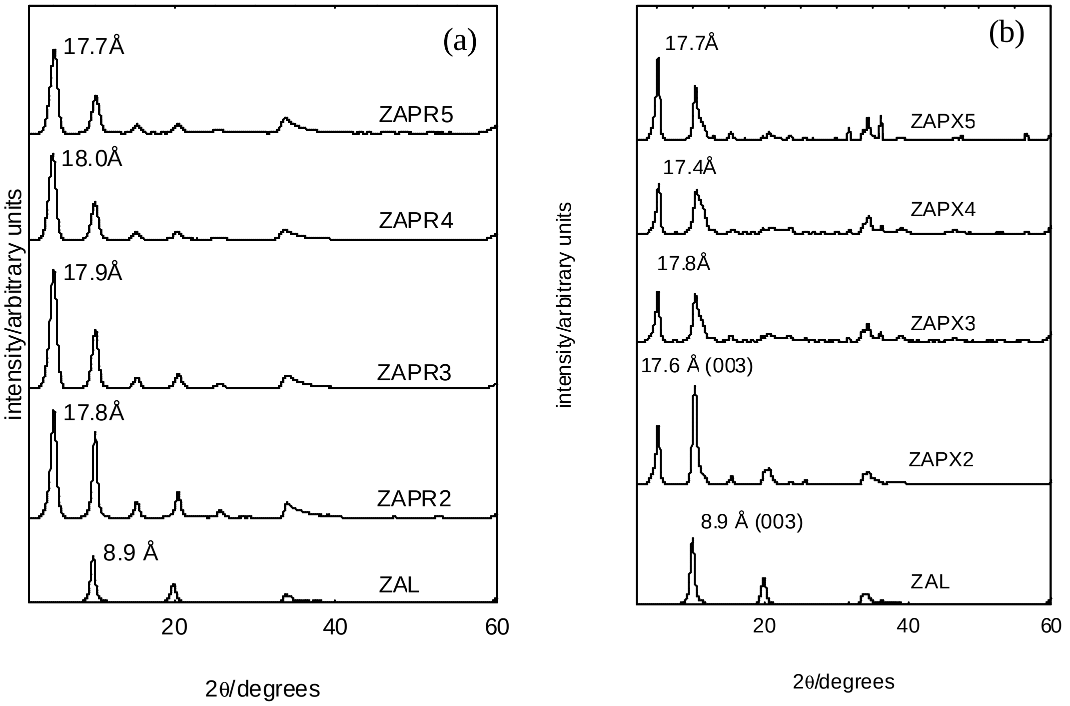

Figure1: PXRD patterns of the Zn-Al-PA nanocomposite (ZAPRs) synthesized by direct co-precipitation method (a) and Zn-Al-PA nanocomposite (ZAPXs) obtained by ion-exchange of NO_3^- in LDH (b) prepared at various initial Zn/Al molar ratios.

As shown in Figure 1(b), the second peak for (006) reflection in almost all the samples are broad and not symmetry. It likely that there are two peaks overlapping at $2\theta = 10$ °. The most possible explanation for this, is that the nitrate ion was not completely exchanged by pamoate anion in the layered structure of Zn-Al-LDH, resulting in two different phases co-existed in the resulting materials, namely LDH phase and nanocomposite phase. Typically, the PXRD patterns of the PA-intercalated LDH shows three major reflections: (i) the large basal spacing (d_{003}); (ii) the half-height harmonic (d_{006})(where $d_{006} \approx ½\, d_{003}$) and (iii) the d_{009} reflection (where $d_{009} \approx$? d_{003}). The remaining reflections can be indexed as either reflections of the intercalated or unintercalated LDH phase [6].

Figure 1(a) shows the intensity of the d_{003} reflection is higher than the d_{006} reflection and the d_{006} reflection is higher than d_{009} reflection, which indicates a typical nanocomposite phase. Figures 1(b) shows the intensity of the d_{006} reflection in all the samples is higher or similar to the d_{003} reflection. Again these indicated that the PXRD pattern is not a typical PXRD pattern of an organic-intercalated LDH. It is a mixture of LDH and nanocomposite phases. Noteworthy also in Figure 1(b), a number of peaks, which are characteristic of PA phase were detected. This might be due to the unintercalated PA, which was adsorbed onto the surface of ZAPXs during the ion-exchange process.

PXRD patterns of ZAPXs show that the expansion of basal spacing between 17.4 –17.8 Å is due to the intercalation of PA into the interlayer structure of Zn-Al-LDH through ion-exchange mechanism. The basal spacing obtained in the nanocomposite prepared by ion-exchange method is slightly lower than the basal spacing of the nanocomposite prepared by direct co-precipitation method, which is in the range of 17.7 – 18.0 Å. This is presumably due to the slightly different spatial orientation of PA in the interlamellae of the nanocomposite.

Comparing the PXRD patterns of nanocomposite prepared by direct co-precipitation and ion-exchange method, it is clear that direct co-precipitation method give pure-phase nanocomposites with good crystallinity, higher basal spacing and well-ordered layered structure of the resulting materials.

Fourier Transform Infrared (FTIR) spectroscopy

Figures 2(a) and (b) show the FTIR spectra for the nanocomposites prepared by co-precipitation and ion-exchange methods. As shown in the figure, all the nanocomposites show the same absorption bands since the same functional groups are present in the materials as the results of the intercalation of PA into the interlayer spacing of Zn-Al layered double hydroxide. A broad absorption band centred at around 3440 cm^{-1} is due to the presence of OH stretching of the hydroxyl group of LDH and/or physically adsorbed water molecule. The band at 1638 cm^{-1} is due to $\nu_{H\text{-}O\text{-}H}$ bending vibrations. A sharp intense band located at 1459 cm^{-1} is attributed to CH_2 scissoring [8].

Another sharp and very intense band at 1384 cm^{-1} is attributed to the ν_3 (NO_3^-) vibration. This is in agreement with the data shown in Table 1 which shows the presence of more than 1% of nitrogen in the nanocomposite which indicated that nitrate ion was not totally replaced by pamoate ion during the ion-exchange process. Strong bands near 741-816 can be attributed to the presence of phenyl ring substitution [9]. Another two bands at 616 and 428 cm^{-1} are attributed to the Al-OH and Zn-Al-OH bending vibrations, respectively [10].

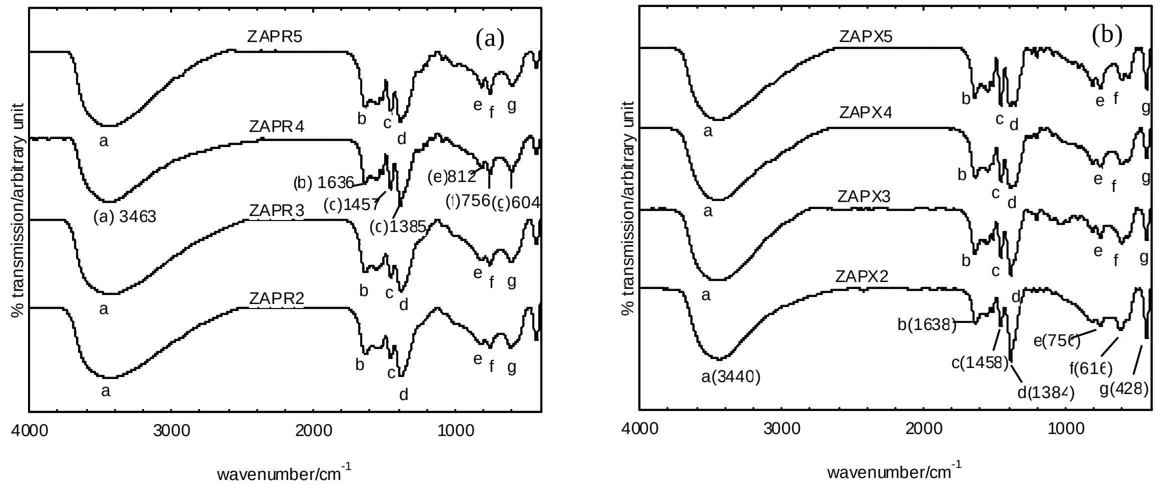

Figure 2: FTIR spectra of Zn-Al-PA nanocomposites (ZAPRs) synthesized by co-precipitation method (a) and Zn-Al-PA nanocomposites (ZAPXs) obtained by ion-exchange of NO_3^- in LDH (b) prepared at various Zn/Al molar ratios, R_i = 2-5.

Organic-Inorganic Composition

The weight percentage of carbon in the nanocomposites prepared by co-precipitation and ion exchange method obtained by CHNS analysis is shown in Table 1. The weight percentage of carbon increases with the increase of Zn/Al molar ratio from 2 – 5. The weight percentage of carbon in the nanocomposite prepared by direct co-precipitation method is in the range of (22 - 26)%, which is slightly higher than the nanocomposite prepared by ion-exchange method, (15 – 22)%.

The percentage weight of PA in the nanocomposites was determined by UV-visible analysis shows that the percentage of PA intercalated in the interlamellae of ZAPRs is in the range of (45 – 51)%, which is higher than in the nanocomposite prepared by ion-exchange method, in the range of (29 – 44)%. This shows that direct co-precipitation method yielded nanocomposites with higher loading and better crystallinity compared to the ion-exchange method. Table 1 also shows that the weight percentage of nitrogen in the nanocomposite prepared by ion-exchange is more than 1%, which is quite high compared to the nanocomposite prepared by co-precipitation method. This shows that in ion-exchange method, the nitrate ion was not totally replaced by pamoate ion or absorbed on the surface of the resulting nanocomposite.

Table 1: Some physico-chemical properties of Zn-Al-PA nanocomposites prepared at various initial Zn/Al molar ratios by direct co-precipitation (ZAPRs) and ion-exchange methods (ZAPXs).

Samples	x_i	C wt.%	PA wt.% (from UV)	N wt.%	BET surface area (m^2g^{-1})	BJH desorption pore volume ($cm^{-3}g^{-1}$)	BJH average pore diameter (Å)
ZAPR2	0.33	22.9	48.8	1.1	0.5	0.001	100.3
ZAPR3	0.25	24.5	50.8	0.6	28.1	0.089	126.3
ZAPR4	0.20	24.1	45.6	0.8	90.1	0.342	114.1
ZAPR5	0.16	26.4	51.0	0.7	112.0	0.435	151.3
ZAPX2	0.33	15.6	29.2	2.08	7.1	0.047	189.9
ZAPX3	0.25	19.4	37.4	1.41	20.6	0.106	160.5
ZAPX4	0.20	19.1	28.2	1.44	21.3	0.108	160.4
ZAPX5	0.16	22.9	44.6	1.01	25.9	0.139	171.6

Isotherm, Surface Area and Pore Size Distribution

The surface properties of ZAPRs and ZAPXs are summarized in Table 1. Table 1 shows that the BET surface area of the nanocomposites of ZAPRs increased as the mole fraction, x decreased from 0.33 to 0.16. The smaller surface area for lower Zn/Al molar ratio was due to the higher charge density of the LDH layers that caused a stronger bonding between the layers and the pamaote anion. Similar results were also obtained for the nanocomposites prepared by ion-exchange method. Comparing the BET surface area of nanocomposites prepared by ion-exchange method and those prepared by direct co-precipitation method, the latter produced nanocomposites with higher BET surface area, as shown in Table 1. However, the BJH average pore diameter of the nanocomposites prepared by ion-exchange method is in the range of (160-189) Å, which is larger than the pore diameter of the nanocomposite prepared by direct co-precipitation method, (100-151) Å, this might be due to the unintercalated PA, which was absorbed onto the surface of ZAPXs during the ion-exchange process as shown in the PXRD pattern in Figure 1(b).

Figures 3 (a) and (b) show that the adsorption-desorption isotherms for ZAPRs and ZAPXs are of Type IV, again indicating mesopores type material (20-500 Å), with adsorption increasing slowly at low relative pressure in the range of 0.0 – 0.06. Further increase of the relative pressure to greater then 0.6 resulted in the rapid adsorption of the adsorbent, reaching a maximum at about 60, 220 and 280 cm^3/g with increasing initial Zn/Al molar ratio to 3 (ZAPR3), 4 (ZAPR4) and 5 (ZAPR5), respectively. For maximum adsorption of the adsorbent of the nanocomposite prepared by ion-exchange method, it was found that it is increased with the increase of the Zn/Al molar ratio, 36, 74 and 92 cm^3/g for R = 2, 3, 4 and 5, respectively. The maximum adsorption for ZAPX5 (92 cm^3/g) is very much lower than the maximum adsorption of the nanocomposite prepared by direct co-precipitation method, ZAPR5 which is 280 cm^3/g as shown in Figure 3(a).

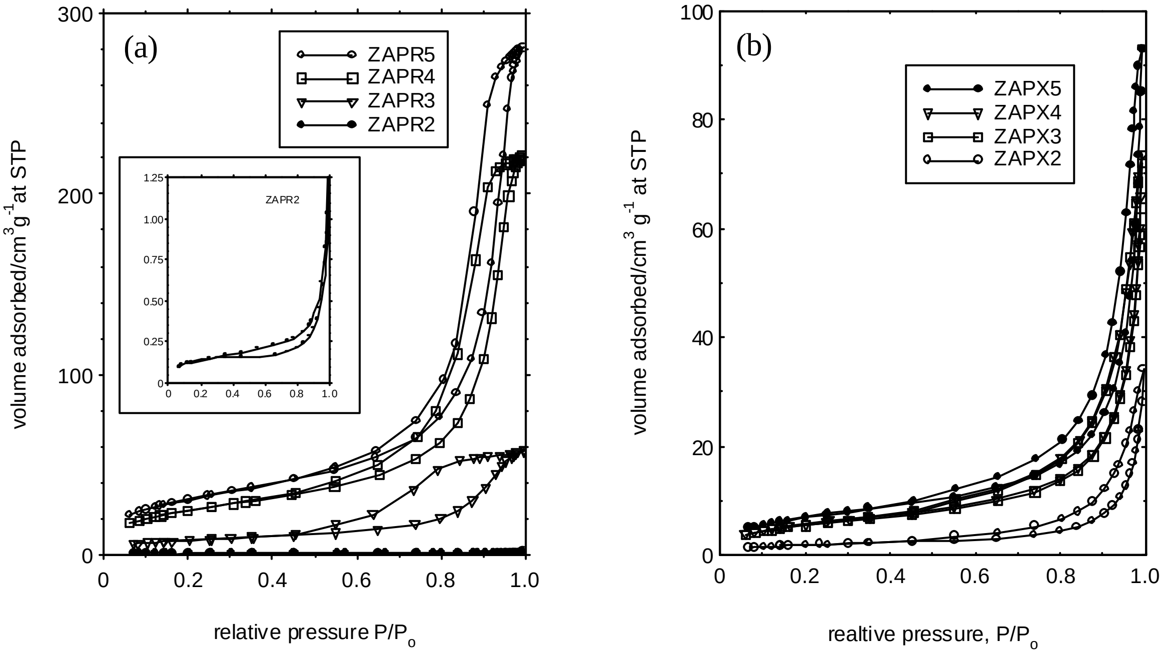

Figure 3: N_2 adsorption-desorption isotherms of Zn-Al-PA nanocomposites (ZAPRs) synthesized by co-precipitation method (a) and Zn-Al-PA nanocomposites (ZAPXs) obtained by ion-exchange of NO_3^- in LDH (b) prepared at various Zn/Al molar ratios.

The BJH pore size distribution for ZAPRs and ZAPXs are presented in Figure 4(a) and (b). These materials show mesopores property, in agreement with Type IV adsorption isotherm. BJH pore size distribution for ZAPRs in Figure 4(a) shows a very intense peak compared to ZAPXs in Figure 4(b) shows a broad peak, indicating different pore texture as a result of incompletely exchanged of nitrate ion by pamoate ion during the synthesis process of ZAPXs. The intensity of the peaks of BJH distribution for ZAPXs increases with the increasing Zn/Al molar ratio used. However, the intensity of the peak of BJH distribution in ZAPX5 is very much lower compared to the intensity of distribution in ZAPR5. This shows that different methods of synthesis resulting in different surface properties of the resulting nanocomposites.

Detail observation shows that the intensity of pore size distribution generally increased with the increased of R_i or with the decreased of mole fraction of Al^{3+} (x_i). As the mole fraction of Al^{3+} decreases, the charge density in the inorganic layers is also decreases. As a result, the pore diameter of the resulting materials becomes larger due to the less charge density, which is responsible to the less tight binding between the organic and the inorganic phases in the nanocomposites. This presumably explained why the intensity of the pore size distribution generally increased as the x_i value decreased.

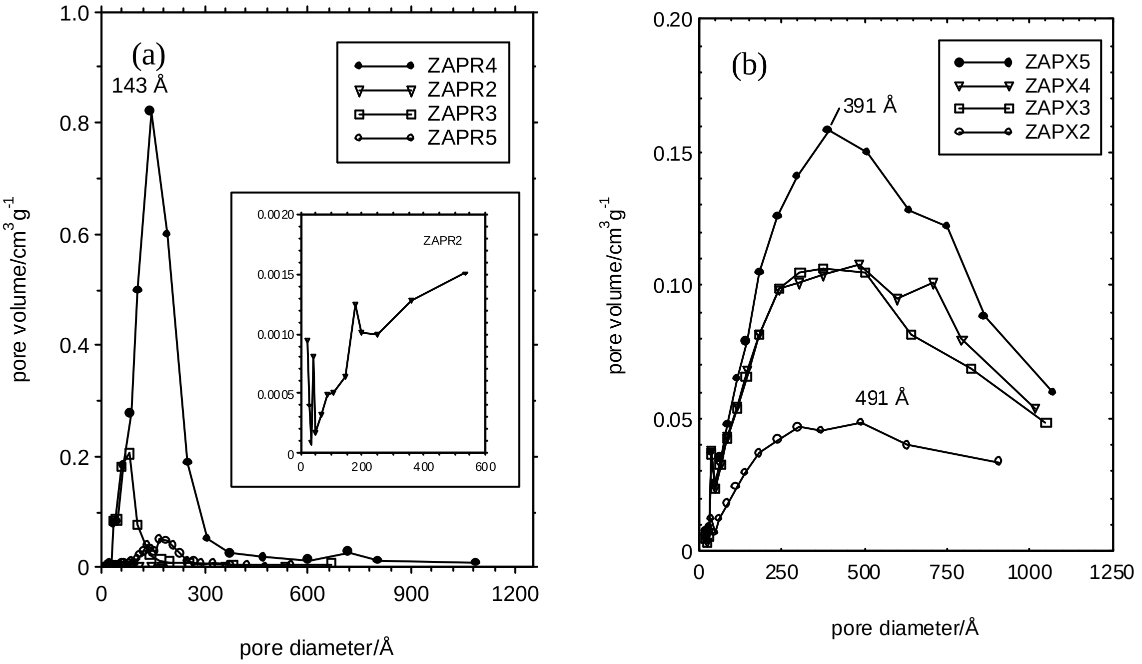

Figure 4: BJH pore size distribution for Zn-Al-PA nanocomposite (ZAPRs) synthesized by co-precipitation method (a) and Zn-Al-PA nanocomposite (ZAPXs) obtained by ion-exchange of NO_3^- in LDH (b) prepared at various initial Zn/Al molar ratio.

Surface morphology

Figure 5 shows the morphology of ZAPRs and ZAPXs obtained by a scanning electron microscope. As shown in the figure, ZAPXs show morphology of flake-like structure, which is slightly different from the morphology of ZAPRs, in which the latter show agglomerates of compact and non-porous granule structure. The different morphology obtained in ZAPXs presumbly due to the presence of two different phases namely LDH and nanocomposite phase, as the result of the incompletely ion-exchanged of nitrate by the pamoate ion during the intercalation and deintercalation processes.

Conclusion

Zn-Al-pamoate nanocomposite was prepared by ion exchange of NO_3^- in LDH at various Zn/Al molar ratios and concentrations. The PXRD patterns show that the second peak for (006) reflection are broad and not symmetry due to the overlapping of two peaks at $2\theta = 10°$, which indicated that the nitrate ion was not completely exchanged by pamoate anion in the layered structure of Zn-Al LDH, which means that there are two different phases existed, namely the LDH phase and the nanocomposite phase. The percentage of PA intercalated into the interlamellae is in the range of (22-45)%, which is lower than the loading percentage of PA in the nanocomposite prepared by co-precipitation method, which is in the range of (43-51)%. This shows that under our experimental conditions, the co-precipitation method yielded nanocomposite with high PA loading and better crystallinity. The BET surface area of the nanocomposites is higher for the one prepared by co-precipitation method. Surface morphology of the nanocomposites synthesized by ion-exchange method shows a flake-like structure, which is slightly different from the morphology of nanocomposites prepared by co-precipitation method in which the latter show agglomerates of compact and non-porous structure. This presumably due to the presence of two different phases existed in the nanocomposite as the result of the incompletely ion-exchanged of nitrate by the pamoate ion during the intercalation and deintercalation processes.

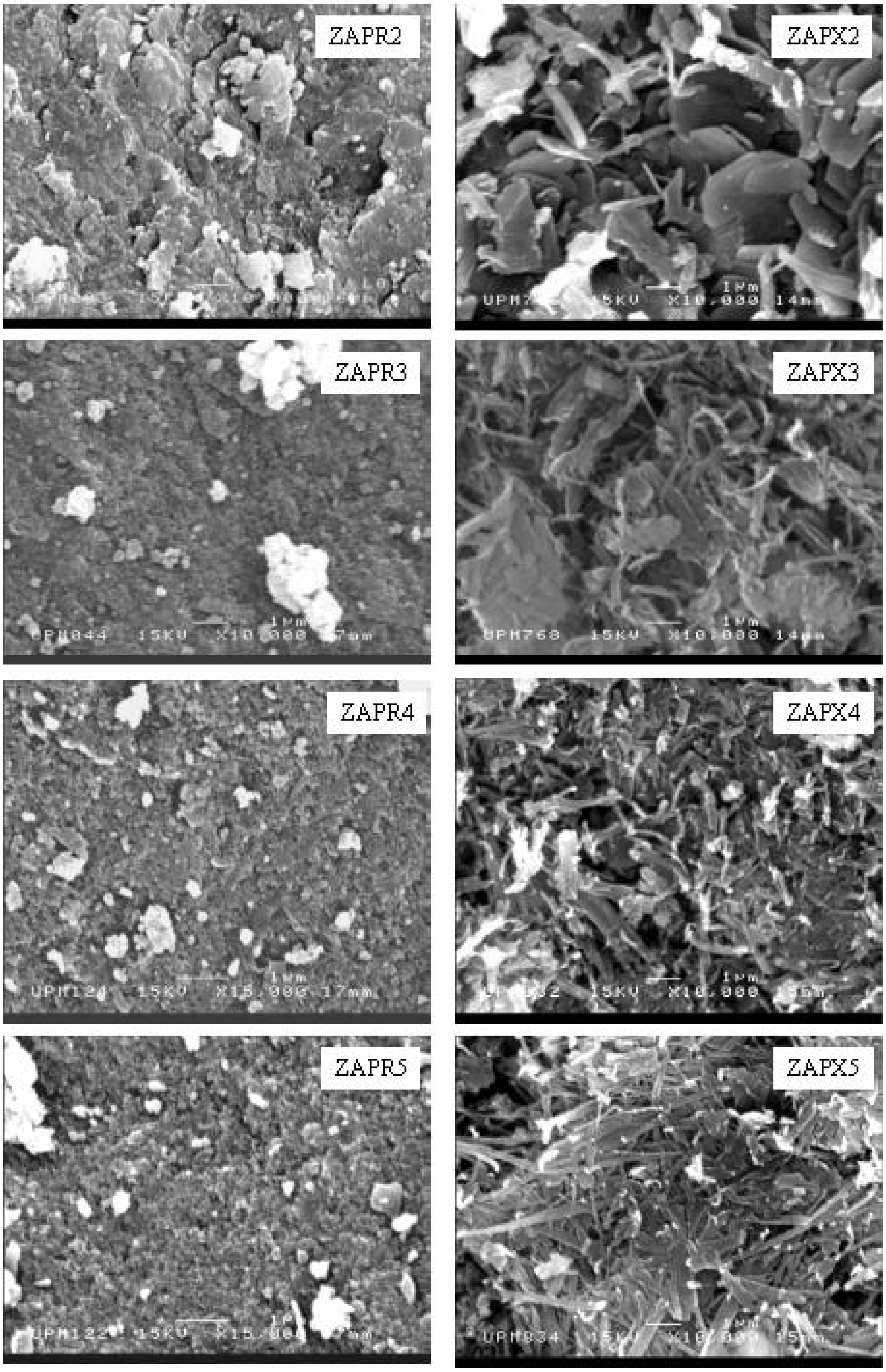

Figure 5: Surface morphology of the nanocomposites synthesized by direct co-precipitation (ZAPRs) and by ion-exchange method (ZAPXs) at 10,000x magnification.

Acknowledgements

We are grateful to the Malaysian Government for providing the grant under IRPA No: 09-02-0897-EA001. One of us (Z.J.) would like to thank Universiti Tenaga Nasional (UNITEN) for the scholarship.

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