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Influencing factors on kaolinite-potassium acetate intercalation complexes

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Abstract:

This paper presents an immersion method for preparing the kaolinite-potassium acetate intercalation complexes. The effectiveness of intercalation and influencing factors were analysed and evaluated. The results show that the intercalation of kaolinite by potassium acetate is strongly related to crystallinity of kaolinite, concentration of intercalating agent solution, aging time and pH. The well-crystallized kaolinite is conducive to intercalation by potassium acetate. A higher concentration of intercalating agent (≥30%) can complete the intercalation in a short time (<12h), but at lower concentrations intercalation took significantly longer (≥144h). The weak alkaline condition of pH=10 proved to be the most suitable environment for the formation of intercalation complex. A good intercalated complex can be obtained at room temperature.

Keywords: Kaolinite; Intercalation complex; Influencing factors, X-ray diffraction

1. Introduction

Kaolinite, with the chemical composition $Al_2Si_2O_5(OH)_4$, is the most abundant mineral of the kaolin group including dickite, nacrite and halloysite, and is a dioctahedral 1:1 phyllosilicate formed by superposition of silicon tetrahedral sheets and aluminum octahedral sheets. As a consequence of its tightly-packed structure, kaolinite particles are not easily delaminated and the kaolinite layers are not easily separated (Miranda-Trevino and Coles, 2003).

Advances in the preparation of hybrid organic–inorganic materials by intercalation of organic molecules into kaolinite represented the possibility for developing new and interesting materials (Gardolinski et al., 1999). Lately, kaolinite has become a widely studied clay mineral noted for its unique physiochemical characteristics and versatile industrial applications (Ming, 2004). Depending on the application, kaolinite is often modified from its natural state by physical or chemical treatment to enhance the properties of the material. One of the most studied systems to date is the intercalation of synthetic polymers into layered aluminosilicates (Liu and Zhang, 2007; Letaief et al., 2008; Liu et al., 2008; Zhang et al., 2009; Cheng et al., 2010a). In this way, it is possible to produce nanocomposites that usually present unique properties with the isolated starting materials. The complexes of kaolinite intercalated by organic molecules have gained much attention over the recent decades, essentially making the clay into a single layered mineral (Frost et al., 2003; Franco et al., 2004; Gardolinski and Lagaly, 2005; Cheng et al., 2010b).

Some methods for intercalation of kaolinite have been reported in which the layered kaolinite were intercalated with small molecules such as urea, potassium acetate, ammonium acetate, acrylamide, formamide, dimethylsulphoxide, etc. (Wada, 1961; Churchman et al., 1984; Frost et al., 1998b; Frost et al., 1998c; Franco and Ruiz Cruz, 2004). Meanwhile, the intercalation of polymeric composites has been reported (Gardolinski et al., 2005). The process builds on the previous intercalation of the precursor with subsequent thermally induced polymerization (Gardolinski et al., 1999). Potassium acetate (KAc) is an important precursor for the preparation of intercalated complexes through substitution reaction with some indirect insertion of organic molecules because KAc can directly insert into interlayer of kaolinite. Intercalation/deintercalation can enlarge the volume of the kaolinite/organic complex and improve the traditional application. At present, the methods for the preparation of kaolinite/organics complex include pressurization, pyrogenation, agitation (Frost et al., 2000a; Franco and Ruiz Cruz, 2004; Gardolinski and Lagaly, 2005; Wang and Zhao, 2006; Elbokl and Detellier, 2008). These methods require high energy consumption and long production periods. In order to achieve 90 % intercalation, they need at least four days time using the traditional method at room temperature or at least several hours heating in a water bath (Frost et al., 2002; Kelleher and O'Dwyer, 2002; Franco et al., 2004). It is critical to find a method with less time and energy consumption.

Although kaolinite-KAc intercalation complexes have been prepared in the laboratory, more detailed investigations are necessary to determine the factors which influence their preparation. The application of X-ray diffraction (XRD) to the study of intercalated kaolinite and degree of intercalation has proven to be of great value to application and scientific research. In the current study, the factors which influence the intercalation processes are investigated by XRD.

2. Experimental methods

2.1. Materials

Three kaolinite samples (Table1) used in this study were the natural kaolin from three different areas in China, with a mean particle size of 45 μm after sieving and grading crushed ore. Their chemical compositions are shown in table 2. The potassium acetate (KAc), ammonium acetate (NH_4Ac), acrylamide (AM), potassium chloride (KCl), sodium hydroxide (NaOH) and hydrochloride (HCl) were purchased from Beijing Chemical Reagents Company (China) in purities of at least 98 % and used without further treatment.

2.2. Preparation of the intercalation complexes

The kaolinite-KAc intercalates were prepared by immersing 10 g of kaolinite in 20 mL of KAc solution at a mass percentage concentration of 30 %. The samples were stirred for 10 minutes at room temperature. The kaolinite-NH_4Ac intercalate was prepared by mixing 10 g of kaolinite with

20 mL of NH_4Ac solution at a mass percentage concentration of 30 % in a container for a sufficient time (4 days) with occasional stirring to achieve maximum intercalation, at room temperature. The kaolinite-AM intercalate was prepared by stirring 10 g of kaolinite in 20 mL of AM solution at a mass percentage concentration of 30 % for 4 days, at room temperature. The kaolinite-KCl intercalation complex was also obtained by immersing 10 g of kaolinite in 20 mL of KCl solution at a mass percentage concentration of 30 %. After appropriate time, the complexes were allowed to dry at room temperature before the XRD analysis.

2.3. Characterization

All the samples were recovered by filtration and dried, then prepared for X-ray diffraction studies as random pressed powder. The powder X-ray diffraction (XRD) analysis was performed using a Rigaku D/max-rA X-ray diffractometer (40 kV, 100 mA) with Cu (λ=1.54178 Å) irradiation at the scanning rate of 2 °/min in the 2θ range of 2.6-50 °.

2.4. Calculation the degree of intercalation

According to the research by Wiewiora and Brindley (1969), the degree or extent of intercalation was determined by intercalation ratio using integrated areas of the reflections:

$$\text{Intercalation Ratio}= [I_{(001)\text{complex}}/(I_{(001)\text{complex}} +I_{(001)\text{kaolinite}})]\times 100\%$$

where $I_{(001)\text{complex}}$ and $I_{(001)\text{kaolinite}}$ represent the basal peak intensity of the complex (d ~ 1.42 nm) and of the unexpanded kaolinite component (d ~ 0.715 nm), respectively.

3. Results and discussion

3.1. Influence of the type of intercalated molecules

Fig. 1 shows the XRD patterns of the intercalation complexes by the (A) KAc, (B) NH_4Ac, (C) AM and (D) KCl, respectively. The XRD pattern of kaolinite-KAc intercalation complex reveals large expansions in the kaolinite structure caused by KAc intercalation. Intercalation causes a rapid decrease in the (001) reflection of the original non-intercalated kaolinite and after 24 h intercalation nearly no intensity remains in this peak.

Comparing the XRD patterns of samples with different intercalated molecules (Fig. 1), it is established that the different molecules result in different expansions after intercalation. The NH_4Ac and KCl cannot be intercalated into the layers of kaolinite easily at room temperature. The intercalation ratio of kaolinite by KAc is higher than that by the others. The 0.714 nm $d_{(001)}$ spacing of kaolinite is expanded to 1.428 nm by KAc (Fig. 1a) which is larger than for intercalations by AM. Among these intercalating complexes, KAc appeared to be the best; it caused the largest basal spacings for kaolinite by an easier way.

3.2. Influence of the solution concentration

Fig. 2a shows the XRD patterns of Kz and its intercalated complex with KAc. The XRD pattern shows that the basal $d_{(001)}$ of kaolinite expands from 0.72 to 1.42 nm; with an increase of 0.70 nm. This value is indicative of the intercalation of KAc in the interlamellar space, and this is consistent with the results published previously (Frost and Johansson, 1998; Deng et al., 2002; Franco and Ruiz Cruz, 2004). The intensity loss of peaks in the (022), $(1\bar{3}0)$, $(\bar{1}31)$, (003), $(1\bar{3}1)$ and $(\bar{1}13)$ reflections suggests that the well crystallized kaolinite suffers structural degradation after intercalation (Frost et al., 1998a; Frost et al., 1998c).

Fig. 2a shows the variation in the peak intensity of the intercalation complexes, which are intercalated with different solution concentrations of 5 %, 10 %, 20 %, 30 %, 50 % and 75 %, respectively. It can be concluded that a higher solution concentration results an easier intercalation for kaolinite. This trend can be observed in the study for sample Kz intercalated by KAc. However, the intercalation ratio did not increase too much after 30 %, even with a KAc concentration of 75 %. From the view of green chemistry, minimizing the use of reagent, a concentration of 30 % is good enough to gain a satisfactory intercalation result.

3.3. Influence of the reaction time

Fig. 3 shows the intercalation ratio as a function of reaction time. It can be observed that a reaction in solutions of concentrations 5% and 10% starts off slowly and remains at a low reaction level even up to 144 hours. However, only 12 hours are required to achieve 90 % intercalation ratio in the solutions concentrations of 30 % and 50 %. The intercalation ratios of 92 % remained the same even for longer reaction time. It is noticed that the greatest increase for the intercalation ratio occurs between 8 and 12 h. It is interesting to remark that the solution concentration at 30 % results a better intercalation than the other solution concentrations and 12 hours is better for the solution concentration at 30 %.

According to Weiss et al. (1969) the kinetics of the intercalation reaction cannot be described as a simple diffusion process. The relationship between the fraction of reacted kaolinite and the time can be best expressed by the logarithmic equation

$$\sqrt{-\ln(1-\alpha)} = kt$$

It is shown that intercalation ratio increases notably during the first hours of reaction when the mass percentage concentration of KAc solution is high, whereas the rate of intercalation remains almost unchanged even for 5 days of reaction time when the mass percentage concentration of KAc solution is low.

3.4. Influence of the structural order

Kaolinite is described as two kinds: low defect (ordered) kaolinite with a regular stacking sequence and high defect (disordered) kaolinite with disordered stacking. Because of the importance of estimating, even in a relative sense, the degree of disorder of a given kaolinite, empirical relations have been used. The most widely used relation for the kaolinite minerals is that proposed by Hinckley (1963).

Structural order in these samples was estimated using the Hinckley index (HI) (Hinckley, 1963). The samples from three different origins clearly reveal an increase in the ordering in the sense Kx<Kz<Kd. Fig. 4a shows the XRD patterns for different kaolinite types. In fact, a positive trend between the structural order and intercalation ratio is reported in Table 3. The Kz kaolinite with HI of 1.35 shows 93 % intercalation ratio. Thus, it is concluded that the well-crystallized kaolinite is propitious to intercalation by small molecules.

According to the above model from Weiss et al.(1969) , the length of elastically deformed zone in the interior of the crystal is taken as the “cooperative action length” and depends on the net increase in basal spacing as well as on the elastic properties of the layers, which are themselves related to the crystallinity of the kaolinite used: the better the crystallinity, the longer the "cooperation action length", the faster the reaction and the higher the intercalation ratio, since the weaker cohesion on the zone of the crystal favors further penetration (Gardolinski, 2005).

3.5. Influence of the temperature

The XRD patterns obtained at increasing temperatures are shown in Fig. 5 which reveals a slight increase in the intensity of complex reflections between room temperature and 60 °C, due to the increase of adsorbed molecules. This probably leads to a better intercalation ratio in the packing of the kaolinite. In the range above 60 °C, the extent of intercalation decreases. Furthermore, the partial removal of KAc causes a gradual decrease in intensity of the reflections of the complex, especially for the $d_{(001)}$ reflections (Fig. 5a). The data indicates that the best temperature is 60 °C for preparing the kaolinite-KAc intercalation complex.

This result is probably because, with the rise of temperature, the KAc molecular aggregates broke down and formed free-er molecules, which can move faster and diffuse into the interlayer of kaolinite. This is the main reason for the increase in rate of the intercalation. It is well known that the kaolinite-KAc intercalation complex is formed from expansion of kaolinite with both KAc and water molecules (Wada, 1961; Frost et al., 2000b; Cheng et al., 2010c). When the temperature of the system is at about 100 °C and water is being heated and vapour rising, the intercalation complex can not been formed. The intercalation complex will deintercalate if the reaction temperature is high. Therefore, the intercalation temperature should be below the temperature for water evaporation and deintercalation of the intercalation complex.

3.6. Influence of pH

The effect of pH on the intercalation of kaolinite is important; however it is not well documented yet. In the case of low pH =2.0, the reflection at $2\theta=12.34$ ° for the intercalated kaolinite complex can be found. However, the peak is of a much lower intensity, which indicates the very low intercalation ratio in the pH=2.0 condition. Kaolinite samples derived from a higher pH value (pH=10) exhibit a distinct intercalation with an intense reflection at $2\theta=12.34°$ and a small refection at 2θ=6.21° for non-intercalated kaolinite, as shown in Fig.6b. At the intermediate pH value, the intercalation ratio is upward trend, whereas this trend shows a reverse when pH>10.

This reveals that pH= 10.0 is the best pH value of those tested for intercalating KAc into kaolinite. This is may be due to two mechanisms:

1) Under acidic conditions, the alumina octahedral sheet in the structure of kaolinite protonated to form the surface complex $AlOH_2^+$ with positive charge; at the same time, the silica tetrahedral sheet in the structure of kaolinite deprotonated to form the complex SiO^- with a negative charge. Therefore, two adjacent layers in kaolinite are held together by electrostatic forces, which is not conducive to intercalation. However, the two adjacent layers in kaolinite deprotonated to form the complex AlO^- and SiO^- with negative charge under alkaline conditions. This is beneficial to intercalation.

2) Under acidic conditions, H^+ and acetate (CH_3COO^-) form acetic acid, which is a weak acid solution. It is difficult to dissociate acetic acid under this condition, and there is not enough effective KAc molecules in the solution.

4. Conclusions

The immersion method was used to intercalate KAc into kaolinite, which can shorten the reaction time from several days to 12 hours and even to a few hours and the complex product with a higher intercalation ratio is acquired. Therefore it improves shortcomings of the traditional method for the superfine kaolinite by intercalation and deintercalation, which can improve the surface area of kaolinite and can be used for absorption and delamination. XRD was used to study the intercalation complexes formed from different kaolinite samples and intercalated by KAc, NH_4Ac and KCl. It was found that intercalation ratio depends on several factors, some of which are related to the kaolinite properties especially the degree of ordering, whereas some other factors include the type of the regent, temperature, reaction time and pH.

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List of Tables

Table1 Kaolin samples

Kaolinite Sample	Location	Content of Kaolinite	Mean particle Size	Impurities
Kaolinite(Kz)	Zhangjiakou, Hebei province of China	95%	-45μm	Quartz
Kaolinite(Kd)	Datong, Shanxi province of China	97%	-45μm	Quartz
Kaolinite(Kx)	Xiaoxian, Anhui province of China	93%	-45μm	Quartz

Table 2 The chemical composition of three kaolinites

samples	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO	LOT
Kz	44.64	38.05	0.22	0.06	0.11	0.27	0.08	1.13	0.13	0.002	15.06
Kd	53.54	30.13	1.52	1.33	0.39	0.72	0.60	0.12	<0.1	0.065	11.61
Kx	62.36	27.54	0.88	0.63	0.05	0.11	0.64	0.04	<0.1	0.117	6.91

Table 3 Hinckley index and intercalation ratio of three kaolinites

Location	Anhui Xiaoxian(X)	Hebei Zhangjiakou(Z)	Shanxi Datong(T)
Hinckley index (HI)	1.03	1.31	1.35
Intercalation ratio (%)	56	84	93

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Fig.1 XRD patterns of kaolinite intercalation complex by (A) KAc, (B) NH_4Ac, (C) AM and (D) KCl

Fig. 2 X-ray diffraction (XRD) patterns for (a) kaolinite/potassium acetate complex and (b) the intercalation ratio curve

K: Kz；A、B、C、D、E：solution concentration at 5%, 10%, 30%, 50% and 75%，respectively. Reaction 24h

Fig.3 the changes of intercalation ratio with reaction time in different concentration of potassium acetate

A、B、C、D: Solution concentration at 5%, 10%, 30%, 50% respectively; kaolinite: Kz

Fig.4 XRD patterns for (a) three kaolinites and (b) three kaolinite-potassium acetate intercalation complexes

C-Kd、C-Kx、C-Kz: Intercalation complexes of Kd, Kx and Kz

Fig.5 XRD patterns of (a) kaolinite/ potassium acetate intercalation composite in different temperature and (b) the curve of intercalation ratio

A、B、C、D: Temperatures at 20, 40, 60, 80 °C respectively; Reaction heating in a water bath at different temperature for 24h; kaolinite: Kz

Fig.6 XRD patterns of (a) kaolinite/ potassium acetate intercalation composite in different pH and (b) the curve of intercalation ratio

A、B、C、D、E、F: pH=2、4、6、8、10、12 respectively. Reaction 24h; kaolinite: Kz

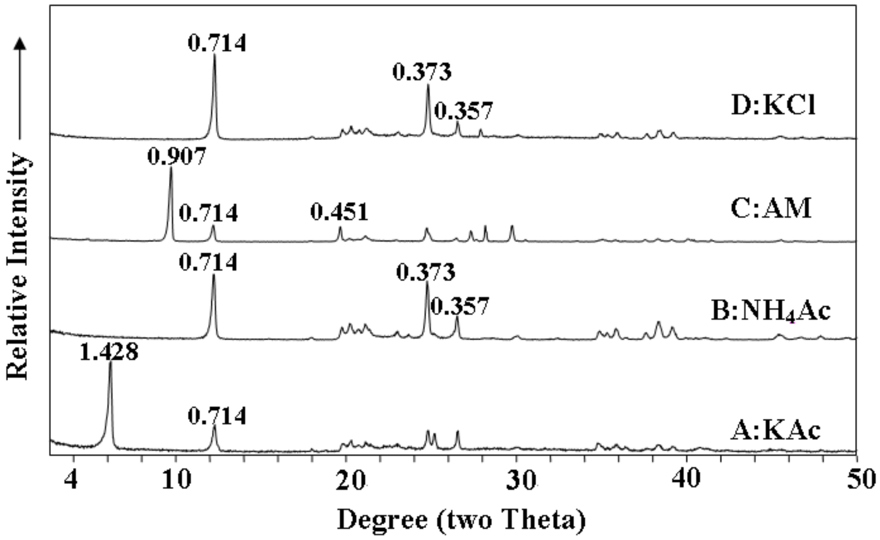
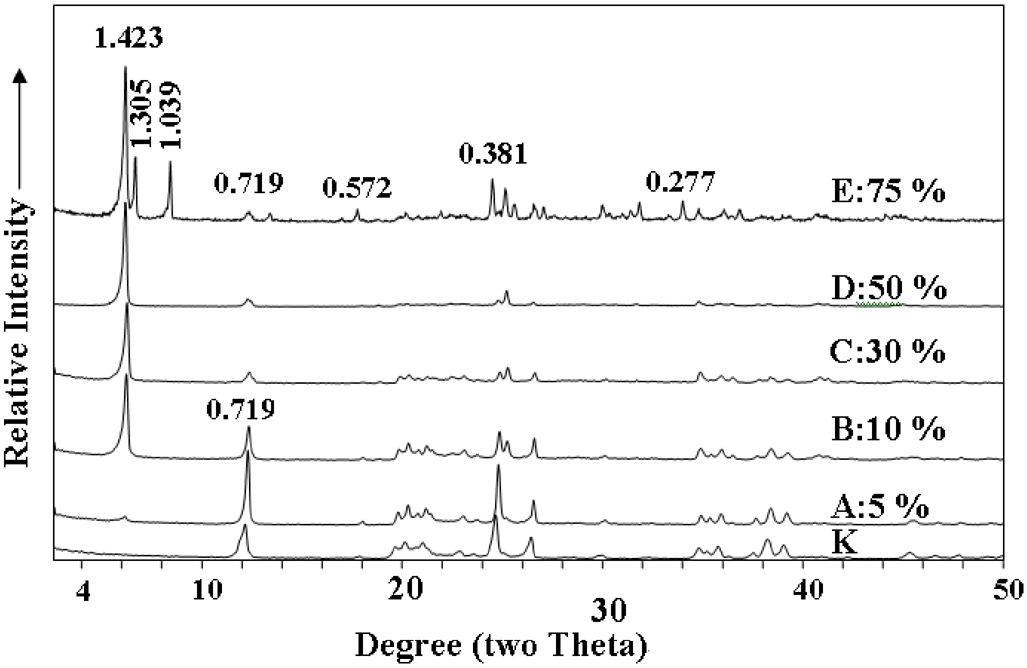

Relative Intensity
0.714
0.373
0.357
D:KCl
0.907
0.714
0.451
C:AM
0.714
0.373
0.357
B:NH$_4$Ac
1.428
0.714
A:KAc
4
10
20
30
40
50
Degree (two Theta)

Fig. 1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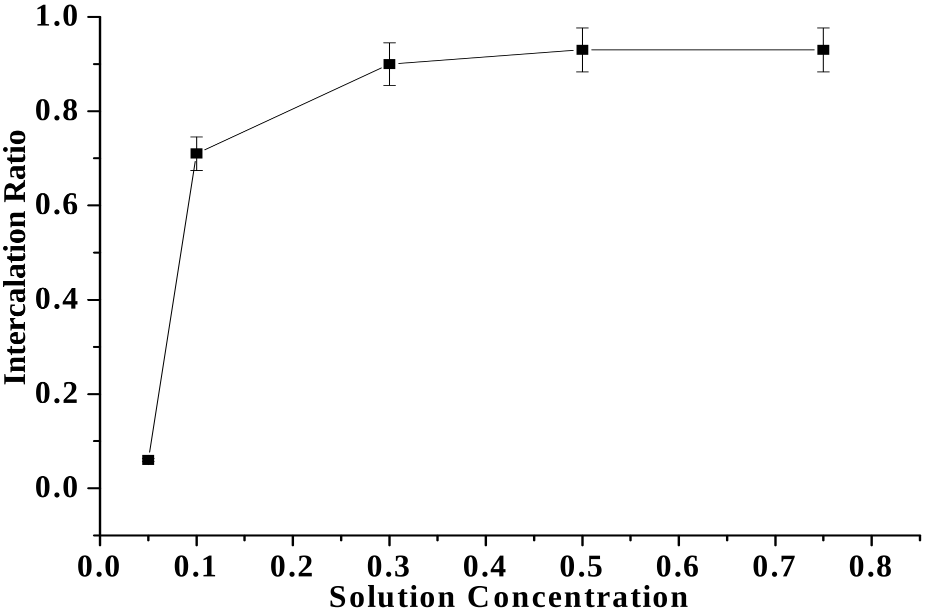
1.305
1.039
0.719
0.572
0.381
0.277
E:75 %
D:50 %
C:30 %
0.719
B:10 %
A:5 %
K
Relative Intensity
4
10
20
30
40
50
Degree (two Theta)

Fig. 2a


1.0
0.8
0.6
0.4
0.2
0.0
Intercalation Ratio
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0.2
0.3
0.4
0.5
0.6
0.7
0.8
Solution Concentration

Fig. 2b

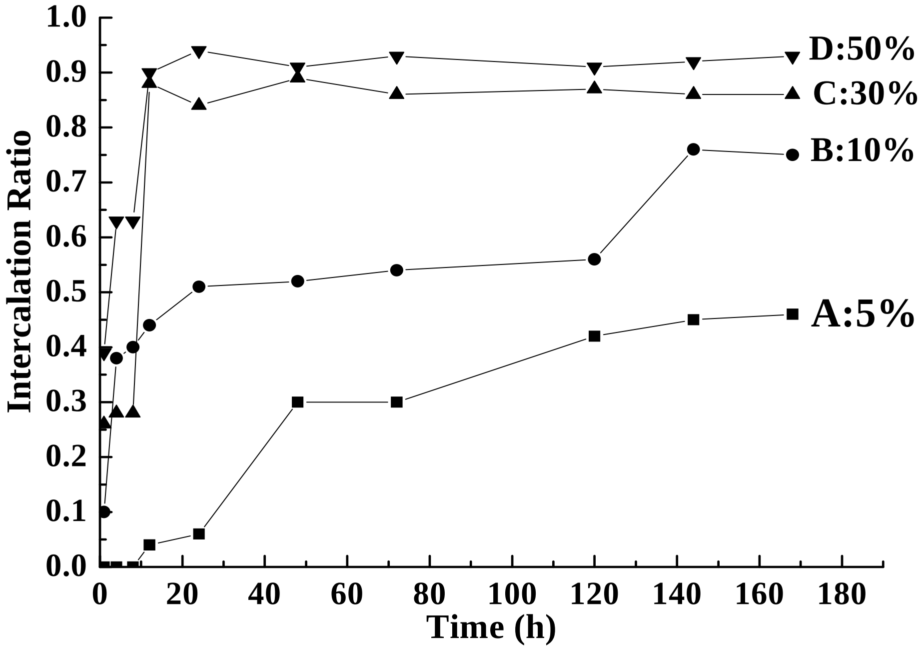

1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
Intercalation Ratio
0
20
40
60
80
100
120
140
160
180
Time (h)
D:50%
C:30%
B:10%
A:5%

Fig. 3

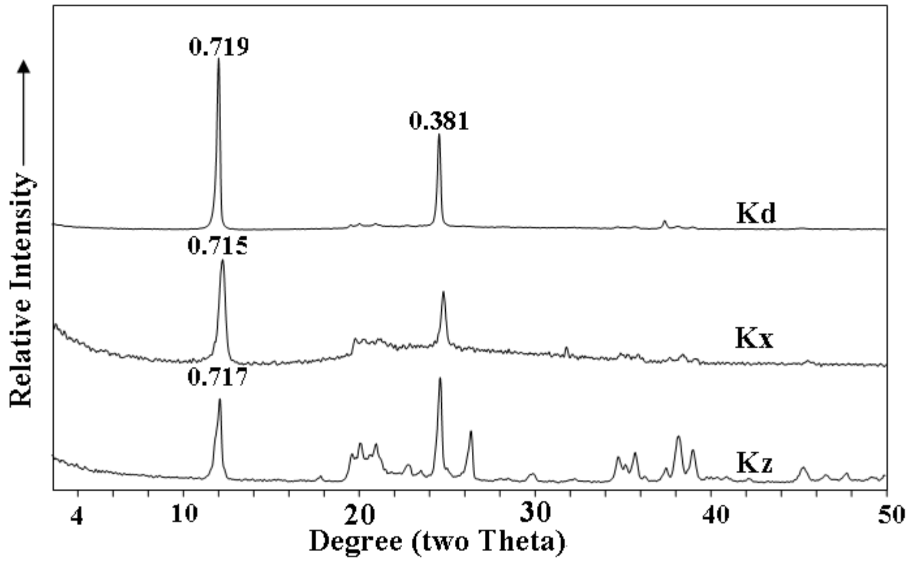

0.719
0.381
Kd
0.715
Kx
0.717
Kz
Relative Intensity
4
10
20
30
40
50
Degree (two Theta)

Fig.4a

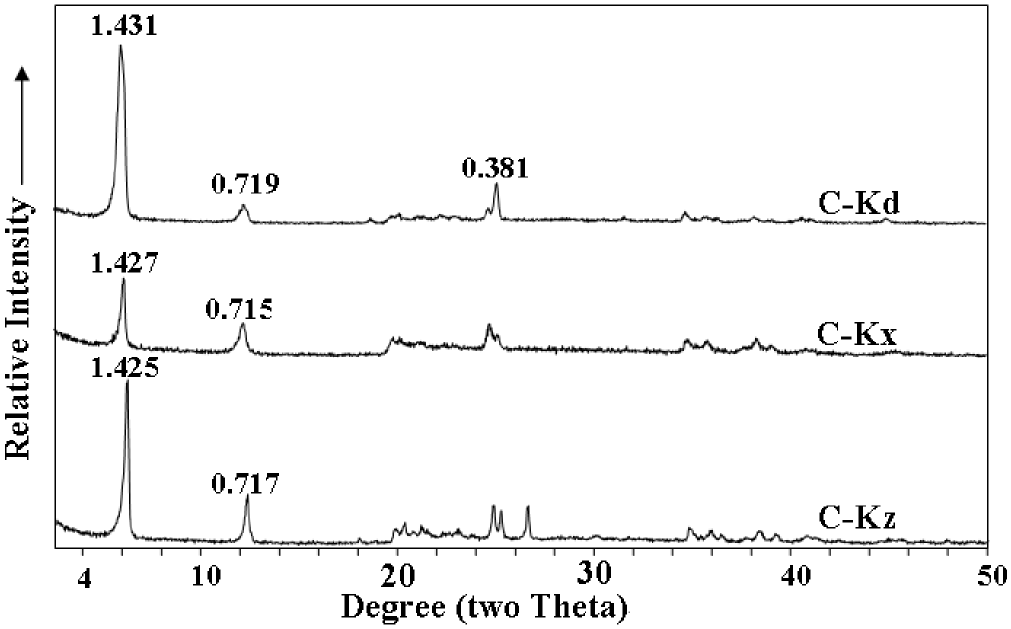

1.431
0.719
0.381
C-Kd
1.427
0.715
C-Kx
1.425
0.717
C-Kz
Relative Intensity
4
10
20
30
40
50
Degree (two Theta)

Fig.4b

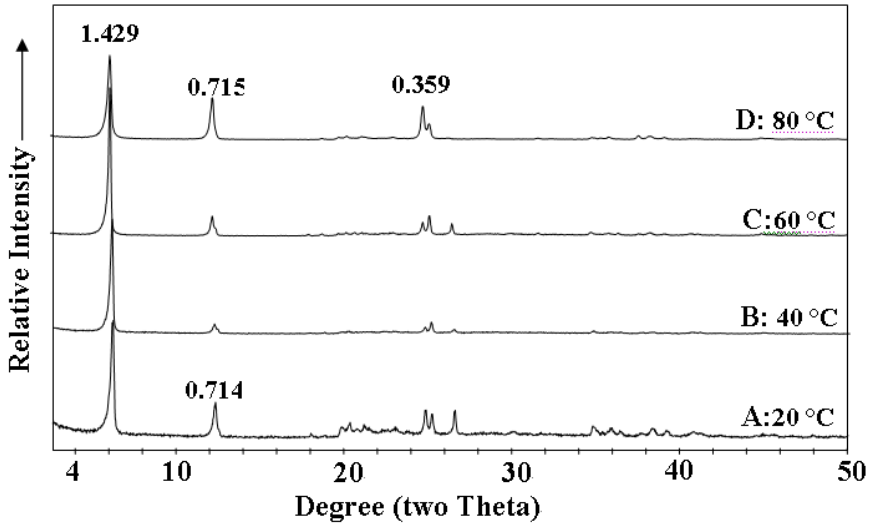

1.429
0.715
0.359
0.714
D: 80 °C
C:60 °C
B: 40 °C
A:20 °C
Relative Intensity
4
10
20
30
40
50
Degree (two Theta)

Fig.5a

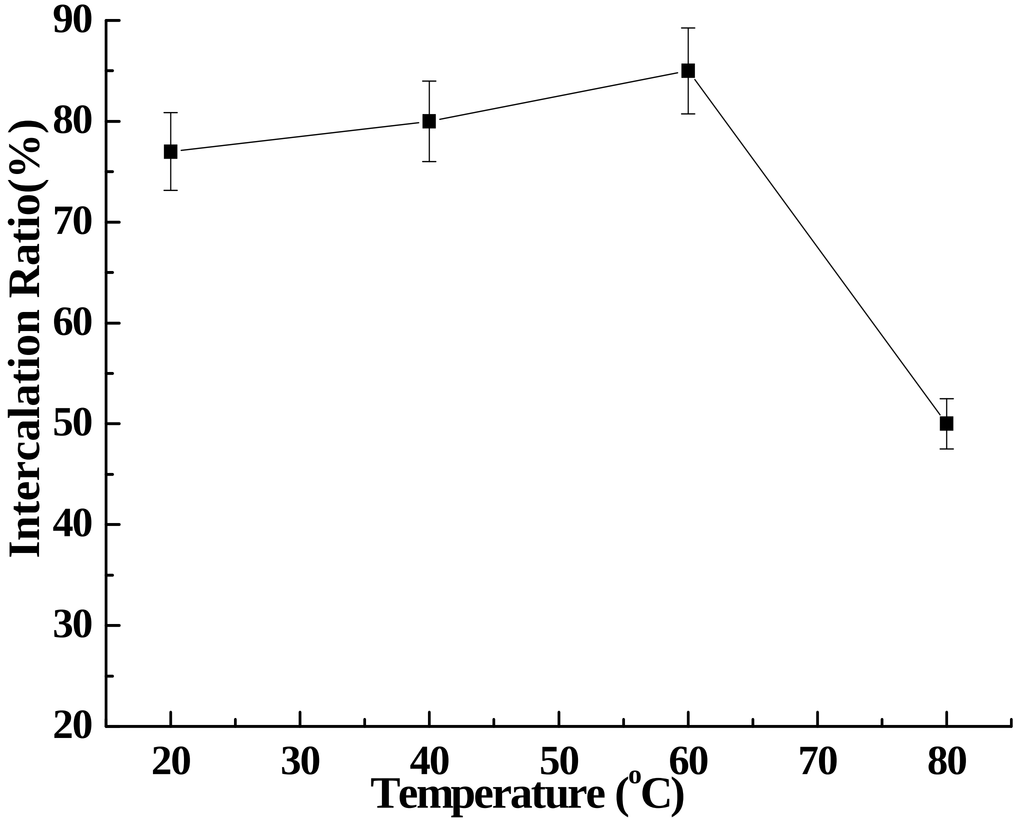
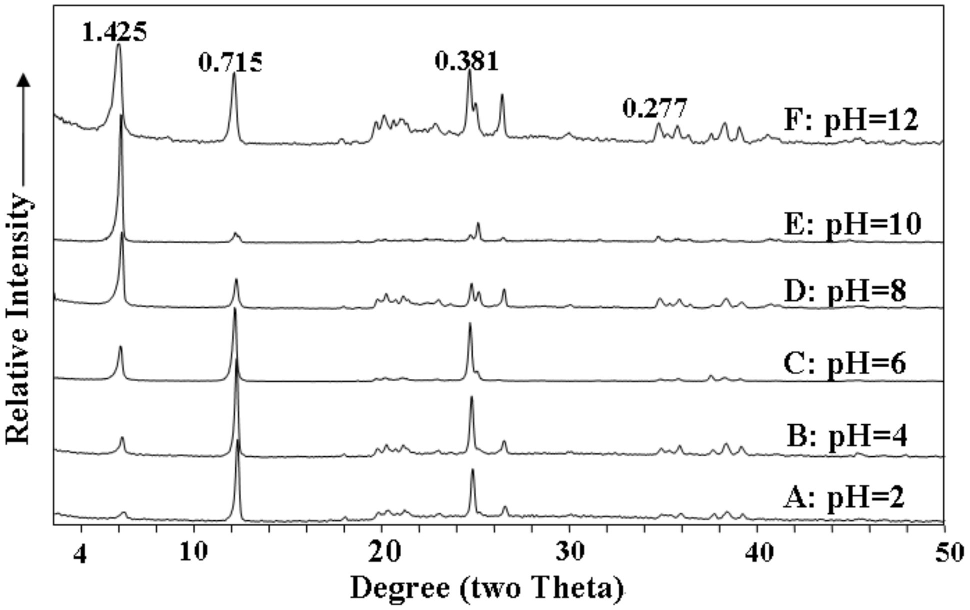
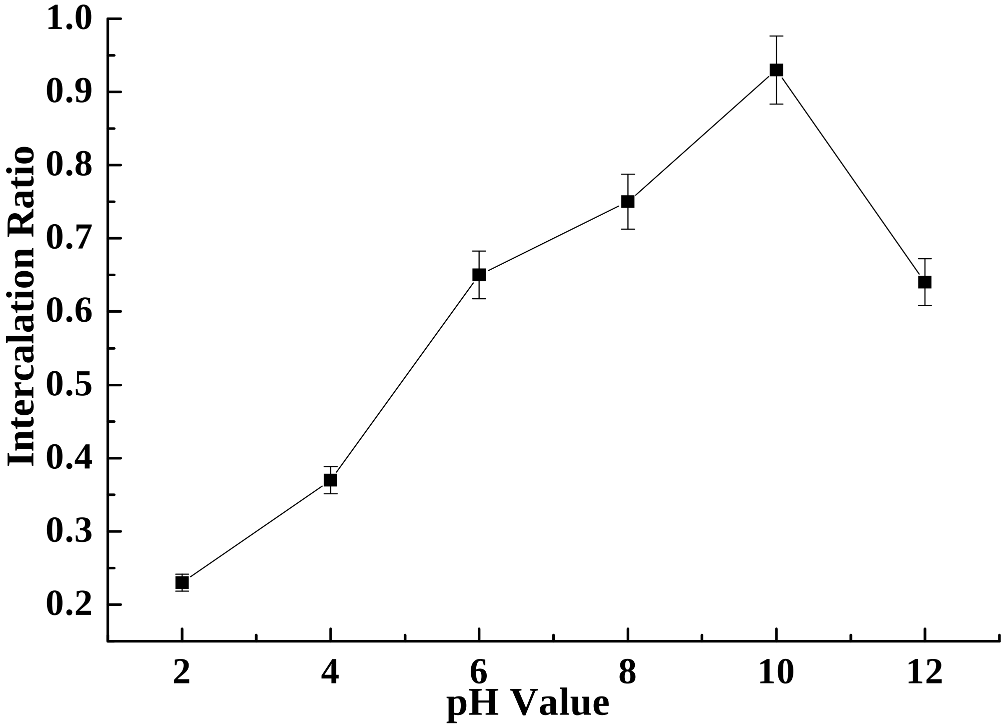

Intercalation Ratio(%)
90
80
70
60
50
40
30
20
20
30
40
50
60
70
80
Temperature (°C)

Fig. 5b



334

335 **Fig.6a**



336

337 **Fig. 6b**

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