

## Confinement and Storage of Cyanate in the Nano Scale via Nanolayered Structures

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### ABSTRACT

Cyanate is anion that is formed during protein poisoning in the body and was studied widely in the field of biochemistry because of its toxicity. The present research aims at confinement and storage of cyanate in the nano scale. It was attained by the intercalation of cyanate into nanolayered structures of Ni-Al LDH. In addition, the influence of aging time on the intercalation of cyanate was studied using X-ray diffraction and scanning electron microscopy. Furthermore, the effect of metals on the affinity towards the intercalation of cyanate anions inside LDH structure was studied by replacement of tetra-valent cations  $Ti^{4+}$  instead of the tri-valent cations  $Al^{3+}$  during the preparation of LDH structure. X-ray diffraction results of the Ni-Ti LDH indicated that the inter-layered space was 0.73 nm. This space was smaller than that of Ni-Al LDH suggesting that the interlayered anions inside Ni-Ti LDH are different from those into Ni-Al LDH. Thermal analysis (TG, DTG and DTA) and Infra-red spectrum showed the existence of only cyanate anions inside Ni-Ti LDH while, in case of Ni-Al LDH, both cyanate and carbonate anions were observed. SEM images showed plate-like structure for both Ni-Ti and Ni-Al LDHs although the shapes of their plates are not similar. Our results suggested that the LDH structures containing titanium cations have higher affinity for cyanate anions than those containing aluminum cations. Therefore, this choice for cyanate in the interlayered spacing widens the applicability to study the effect of the confinement on the toxicity of cyanate by bio researchers.

**Key Words:** Intercalation of cyanate anions, Nanolayered structures, Ni-Al LDH, Ni-Ti LDH, Urea hydrolysis.

### INTRODUCTION

a variety of nano-hybrid materials such as bio-inorganic (Kong *et al.*, 2010), inorganic-inorganic (Li *et al.*, 2010), and organic-inorganic (Saber *et al.*, 2011) systems have created attention for researchers because of their abnormal physicochemical properties that cannot be realized by usual reactions. This trend is very creative, where it shows new compounds with a huge range of organic and inorganic properties.

Cyanate has been investigated widely in many areas of biochemistry where it is formed during protein poisoning in the body. In the other trend, cyanate species have been found to be particularly useful in the preparation of electrical and structural laminates. Accordingly, the relations of molecules containing the cyanate functionality with different substrates should be understood and studied.

It is known that nanolayered structures which have two dimensions confine and

fix compounds in only one dimension. The confinement produce from the intercalation of organic or inorganic species inside interlayered space between two rigid layers, where the interlayered space can adjust and control the thickness and orientation of the organic or inorganic groups. According to the structures of layered materials, layered double-metal hydroxides are a family of 2-dimensional materials having nanolayered structures. The sheets of layered double hydroxides composed of di- and tri-valent metals. The presence of trivalent cations besides divalent cations create positive charge which attract the organic or inorganic species as anions to neutralize the layers (El-Brolossy *et al.*, 2013). In general, layered double hydroxides (LDHs) are expressed by the formula  $[M^{II}_{1-n}M^{III}_n(OH)_2]^{n+}(A^{y-})^{n/y} \cdot mH_2O$ ; di- and tri-valent cations are represented by  $M^{II}$  and  $M^{III}$  respectively; the molar fraction of  $M^{2+}/(M^{2+} + M^{3+})$  can be expressed by  $n$  and  $A^{y-}$  is anion. LDHs that

have ion exchange properties (Basile *et al.*, 2010) can be used as stabilizer for polymeric products or as antacid or container for medical molecules (Holtermuller *et al.*, 1992 and Aguzzi *et al.*, 2007, Saber 2013). Moreover, there are different reports for LDHs to use as adsorbents or photocatalysts for treatment of waste water (Liu *et al.*, 2009 and Saber *et al.*, 2011).

Because of the acute toxicity of cyanate ions, the studies of LDH containing cyanate are rare. Therefore, the intercalation of cyanate among the rigid layers of LDHs expands the applicability to investigate the consequence of the confinement on the toxicity and behavior of cyanate by bio-researchers.

In this contribution, we focus on the intercalation of cyanate anions into Ni-Al and Ni-Ti LDHs through urea hydrolysis. In addition, the result of aging time of LDH particles on the confinement and interaction of cyanate anions with the layers of LDH is studied. Also, the result of cations exchange from trivalent cations to tetravalent cations on the storage of cyanate anions among the layers of LDHs is investigated. Furthermore, this study indicates that the construction of LDHs is not restricted to the processes between di- and tri-valent metals, although these are the only results obtained by several authors.

## MATERIALS AND METHODS

Nickel nitrate, Aluminum nitrate, Titanium chloride and urea were supplied by Sigma-Aldrich. The Ni-Ti and Ni-Al LDHs were prepared by co-precipitation of nickel, aluminum and titanium salts from a homogeneous solution (Saber *et al.*, 2011). Solutions of nickel nitrate, aluminum chloride or titanium chloride (0.047 mol) were mixed with a urea solution (0.5 mol) under vigorous stirring. The mixture was heated at high temperature (90 °C) at different aging times. After filtration and washing several times in deionized and distilled water, the products were dried under vacuum at room temperature.

X-ray diffraction (XRD) of powder was measured on Rigaku, RINT 2200 using

CuK $\alpha$  radiation ( $\lambda = 0.154$  nm ). Thermal analyses of powder were achieved using a Seiko SSC 5200 apparatus by heating samples 10 °C /min up to 800 °C under flow of nitrogen or oxygen. Fourier Transform Infrared spectra were carried out on a Horiba FT-720. Scanning Electron Microscope was executed with JEOL: JSM-6330F.

## RESULTS

### X-ray Powder Diffraction

Powder X-ray diffraction patterns of Ni-Al layered double hydroxides at diverse aging times are revealed in Fig.1. After aging time 6 h, three weak peaks at spacing 0.79, 0.39, and 0.26 nm were observed. With increasing the aging time, these peaks grow and become stronger at 18 h. In the range of aging time from 18 h to 28 h, the main peaks are sharp and their intensity did not change indicating that the crystalline form of the structure became stable after 18 h. It means that the aging time 18 h is critical for the formation of LDH structure. The strong and weak peaks at low and high 2theta values; respectively indicated some familiar properties of layered materials.

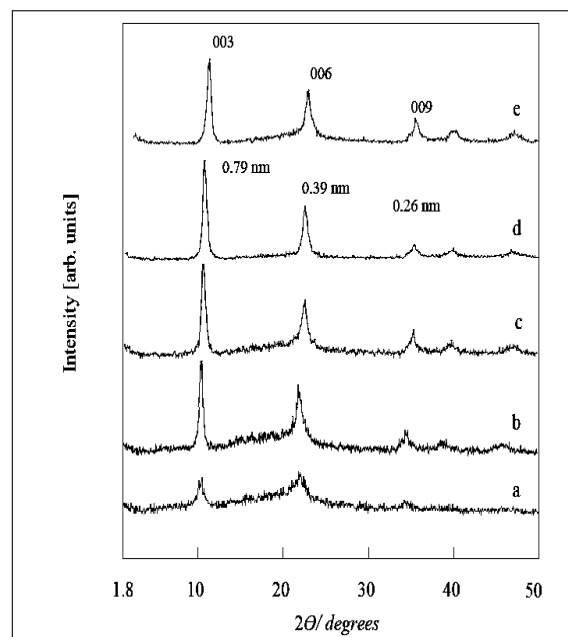


Fig. 1. XRD diagrams of Ni-Al LDH at diverse aging time: (a) 6h, (b) 12h, (c) 18h, (d) 25h and (e) 28h.

The main reflections (0.79nm, 0.39nm, and 0.26nm) matched with diffraction by h k l

planes [003], [006], and [009], respectively. It means that the measured XRD patterns fit well to layered structure. The good agreement between the values corresponding to successive diffractions by basal planes, i.e.  $d(003) = 2d(006) = 3d(009)$  reveals highly packed stacks of the layers ordered along axis c. Dimension c is calculated as three times the spacing for planes (003), i.e. 2.37 nm. The c dimension is very close to that reported for natural and synthetic hydrotalcite, 2.31 nm (Saber *et al.*, 2011). The reflection at 0.79 nm is considerably higher than the reflection 0.76 nm which published for hydrotalcite containing carbonate as the guest (Leroux *et al.*, 2001; You *et al.*, 2002). This value 0.79 nm associated to the width of the brucite-like sheets 0.48 nm in addition to the orientation and size of the anion. This means that the interlayered spacing is not compatible with the size of carbonate anion suggesting existence of cyanate anions. In the case of using titanium instead of aluminum in the LDH structure, the main reflections [003], [006] and [009] in addition to non-basal reflections [100], [101], [012] and [104] were observed with small shift for their reflections to higher (2 $\theta$ ) related to natural hydrotalcites. It means that the width of the layer d003 reduces with the inclusion of tetra-valent cations (Ti) instead of trivalent cations (Al) in layered double hydroxides. Gastuche *et al.*, (1967) reported that the non-basal peaks at  $d = 0.273$  nm, 0.265 nm, 0.259 nm and 0.246 nm in synthetic hydrotalcite are related to the planes [100], [101], [012] and [104] and the existence of diffuse non-basal reflections are considered as sign of a moderately dis-ordered structure. While, the main peak at 0.73 nm was observed for Ni-Ti LDH which matched to the inter-layer space of the LDH-structure, as revealed in Fig.2a. The reflection at 0.734 nm slightly reduced to 0.726 nm by the heating at 150°C indicating that the interlayered spacing of Ni-Ti LDH decreased with the removal of interlayered water. This inter-layer space, 0.73 nm of Ni-Ti LDH, was small compared to 0.79 nm of Ni-Al LDH. It suggests that

the inter-layered anion in the structure of Ni-Ti is unlike that in Ni-Al structure. This speculation is clarified by FT-IR results and thermal analyses.

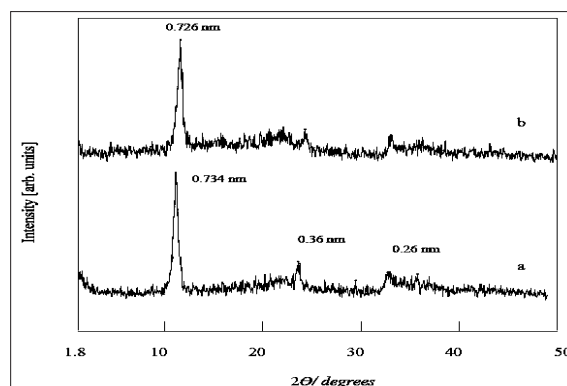


Fig. 2. XRD diagrams of Ni-TiLDH after heating at:(a) 70°C and (b) 150°C.

Although, the x-ray diffraction results are not adequately optimum technique to determine model construction for LDH. However, by using the inter-layer space, the dimensions and orientation of the intercalated ions among the layers was determined. From the layer dimensions of hydrotalcite structure, 0.48 nm (You *et al.*, 2002), the inter-layer space for the anion was determined as 0.29 nm (Fig.3).

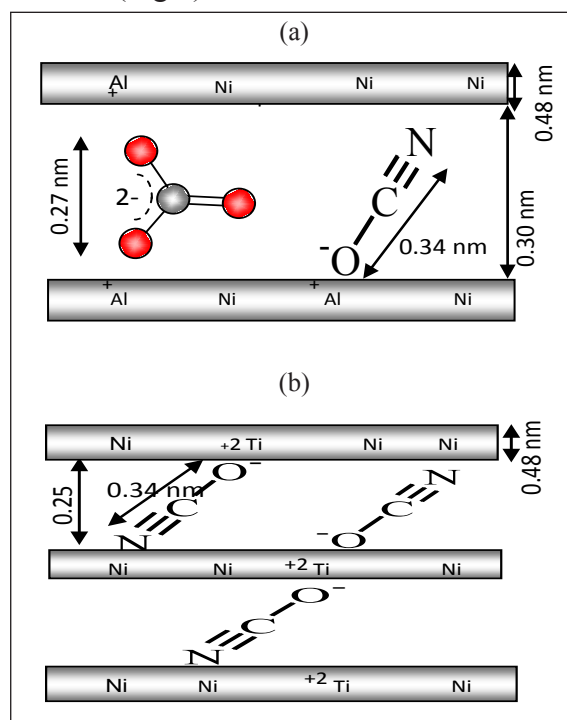


Fig. 3. Schematic representation of: (a) Ni-Al-CO<sub>3</sub>-CNOLDH and (b) Ni-Al-CNOLDH.

By using the dimensions of carbonate and cyanate anions, 0.27 and 0.34nm; respectively, and agreeing to our XRD results, two schematic representations for Zn-AILDH and Zn-TiLDH were suggested as revealed in Fig.3.

### Fourier Transform Infrared Spectroscopy

The nature and symmetry of functional groups of Ni-Al and Ni-Ti LDHs have been identified by the FT-IR technique as shown in Figs. 4, 5. The appearance of the vibrations of  $\nu_1$ ,  $\nu_2$ ,  $\nu_3$  in the different ranges of 2230 – 2180  $\text{cm}^{-1}$ , 600–650 $\text{cm}^{-1}$  and 1190–1220 $\text{cm}^{-1}$ ; respectively, confirmed the intercalation of cyanate ions ( $\text{NCO}^-$ ) into Ni-Al and Ni-Ti LDHs (Nakamoto 1986).

In case of Ni-TiLDH, Figure 4 showed strong band at 2223 $\text{cm}^{-1}$  that due to symmetrical vibration form of NCO. Also, the strong peak observed at 636 $\text{cm}^{-1}$  could be attributed to the  $\nu_2$  form of cyanate ion. An important phenomenon is observed for the attendance of NCO ions inside the inter-layered beside OH groups (Miyata 1995). It means that there is strong effect for the electron density of NCO on the vibration of OH groups. The broad OH vibration band which resolved into two bands in case of Ni-Ti LDH was ascribed to the existence of the NCO group beside OH groups. The unaffected OH groups showed absorption band at 3642  $\text{cm}^{-1}$  as reported for usual hydroxyl groups. Also, there is another band was observed at low value 3467  $\text{cm}^{-1}$ . The shifting of the band of the hydroxyl group due to the lowering of the O–H bond electron density because of the effect of cyanide group. This phenomenon is in agreement with the results of Xu *et al.*, (2001) that indicated the outcome of existence of nitrate ions on the bands of LDH. Also, this phenomenon agrees with the proposed model (Fig.3b) which indicate that cyanate anions look like bridge between two layers through ionic bond with one layer and hydrogen bond with the other layer. The shoulder peaks recorded at 2925 and 2854  $\text{cm}^{-1}$  indicate the stretching OH bonds that

due to the interlayer water molecules and/or produced from the hydrogen-bonds with cyanate ions (Saber *et al.*, 2005). The peak at 1637  $\text{cm}^{-1}$  was due to the bending band of water molecules (PerezRamirez *et al.*, 2001).

In case of Ni-Al LDH, the peaks at 2181 $\text{cm}^{-1}$  and 642 $\text{cm}^{-1}$ , which were ascribed to the stretching vibration of cyanate, were weak as revealed in Fig. 5. In addition, the presence of carbonate anions was suggested by the existence of weak peaks at 1382 $\text{cm}^{-1}$  and 1457 $\text{cm}^{-1}$  (Nakamoto 1986; Miyata 1995). With noticing that the band of free carbonate is reported at 1450  $\text{cm}^{-1}$ , however it divides and shifts with lowering of symmetry (Saber *et al.*, 2005).

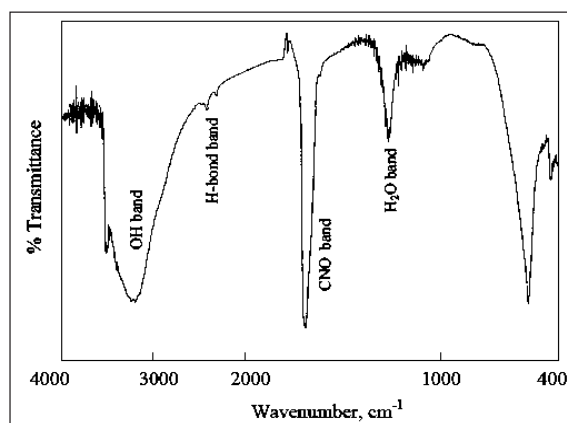


Fig. 4. IR Spectrum of Ni-Ti LDH at aging time 18 h.

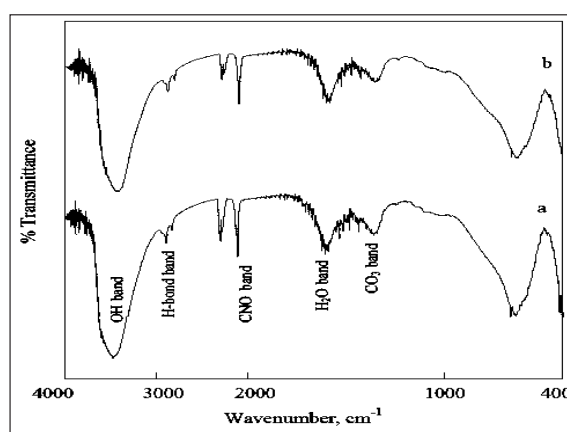


Fig. 5. IR Spectrum of Ni-Al LDH at diverse aging time: (a) 18h, (b) 28h.

The vibration modes of carbonate ( $\nu_1$  and  $\nu_4$ ) are in charge of bands at 1029 $\text{cm}^{-1}$  and 669 $\text{cm}^{-1}$ ; respectively (Perez-Ramirez *et al.*,

2001; Saber *et al.*, 2005).

Generally, these results concluded that both Ni-Ti LDH and Ni-Al LDH have a comparable structure with the common layered double-metal hydroxides structure. It confirmed that Ni-Ti LDH has cyanate as interlayered anions while Ni-Al LDH has two interlayered anions; cyanate and carbonate. This can explain the difference between both the interlayered spacing ( $d_{003}$ ) of Ni-Al and Ni-Ti LDHs. The IR absorption spectra of Ni-Al LDH at different aging time; 18 h and 28 h are similar as shown in Fig. 5. It confirmed that 18 h is enough to build Ni-Al-CNO-CO<sub>3</sub> LDH agreeing with XRD results.

### Thermal Analyses

Thermal analyses of the Ni-Al layered double hydroxide in presence of nitrogen and oxygen were measured by thermogravimetry (TG), differential thermogravimetry (DTG) and differential thermal analysis (DTA), as revealed in Figs. 6, 7.

TG diagram of Ni-Al LDH in presence of oxygen revealed that the mass loss up to 180°C is 10.5wt.% agreeing with a broad band in the DTG diagram of Ni-Al LDH. It due to the removal of surface and interlayered water. The main mass loss occurred in the range of 236 °C - 322 °C coincidence with a clear peak at 299 °C in DTG diagram. It was 15 wt.% and corresponds to the decay and oxidation of both cyanate and carbonate anions.

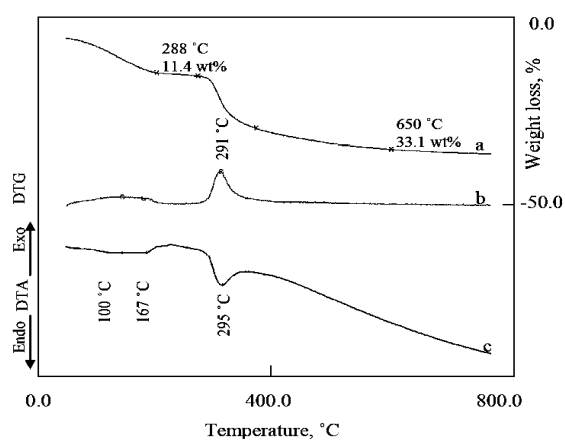


Fig. 6. TG, DTG and DTA of Ni-AlLDH at nitrogen gas

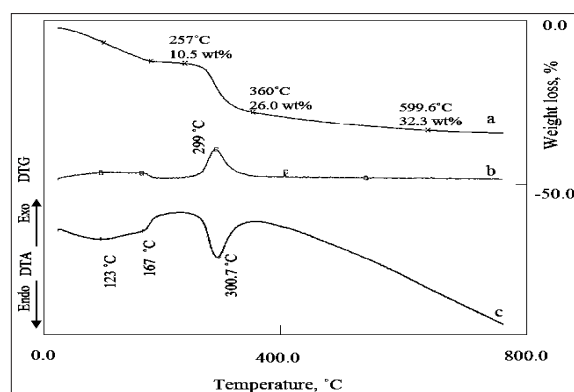


Fig. 7. TG, DTG and DTA of Ni-AlLDH at oxygen gas

The slow mass loss from 322 °C to 650 °C has been reported by many other researchers suggesting different processes taking place between metal oxide hydroxide and the decomposed ions in the interlayer area (Velu *et al.*, 1996; Rives *et al.*, 2000; PerezRamirez *et al.*, 2001).

DTA diagram confirmed TG results through appearance of three endo-thermic peaks at 95°C, 166°C and 290°C as revealed in Fig.6c. When the thermal analyses of Ni-Al LDH were performed at nitrogen gas, the TG and DTG diagrams were similar to that performed at oxygen gas and the peak of DTA diagram became more clear as revealed in Fig. 8.

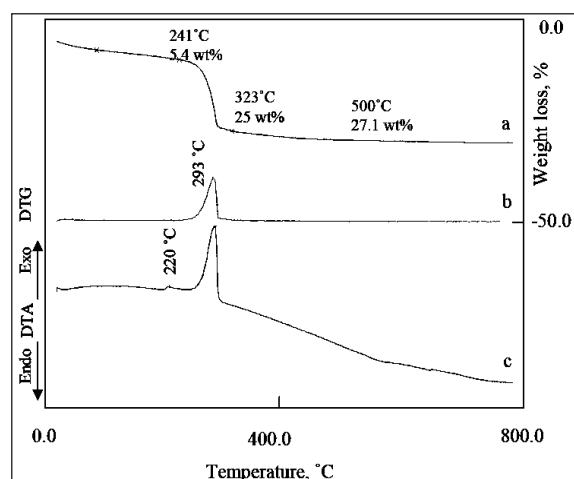


Fig. 8. TG, DTG and DTA of Ni-TiLDH at oxygen gas.

The thermal analysis of Ni-Ti LDH at oxygen is shown in Fig.8. The TG curve revealed that the mass loss of Ni-Ti LDH at 236°C



is only 5 wt.%. It suggests that the quantity of inter-layered water of Ni–TiLDH is lesser than that of Ni–AlLDH, and Ni–Ti LDH has no enough space for water molecules. The major loss of Ni-Ti LDH (20 wt.%) occurred from 236 °C to 322 °C. Agreeing with the main weight loss, the DTA curve of Ni–TiLDH revealed two exothermic peaks at 222°C and 295°C, as revealed in Fig.8c. The weak exothermic peaks could be explained according to a group of thermal events such as evaporation of interlayered water (endothermic event), the decomposition and oxidation of CNO anions (exo-thermic event) in addition to the dehydroxylation process. The comparison between both Ni–TiLDH and Ni–AlLDH in DTA diagram confirmed that the interlayered anions in Ni–Al LDH are carbonate with cyanate anions and the interlayered anions in Ni-Ti LDH are only cyanate anions.

These thermal results pointed to that the cyanate inside Ni–TiLDH have stronger contact with the sheets of the LDH structures than those in Ni–AlLDH. It agrees with the small inter-layered space of Ni–TiLDH related to that of Ni–AlLDH.

### Scanning Electron Microscopy

The crystals of layered double metal hydroxides possess hexagonal platelike morphology if they crystallize, (Ogawa *et al.*, 2000). The natural pyroaurite crystals show plate-like structures with thickness and width for plate in millimeters. These plates seem plane and are often appeared to be grown in irregular orientations producing opened structure. A comparable morphology was observed for Ni-Al LDH showing plates in the range 100nm in width and 1-2  $\mu\text{m}$  as revealed in Fig. 9. SEM images of Ni-AlLDH at low aging time; 12 h, showed plate-like morphology but, in some locations, the plates was not complete. This means that this sample need enough time to complete the construction of LDH as shown in Fig.9 agreeing with XRD data.

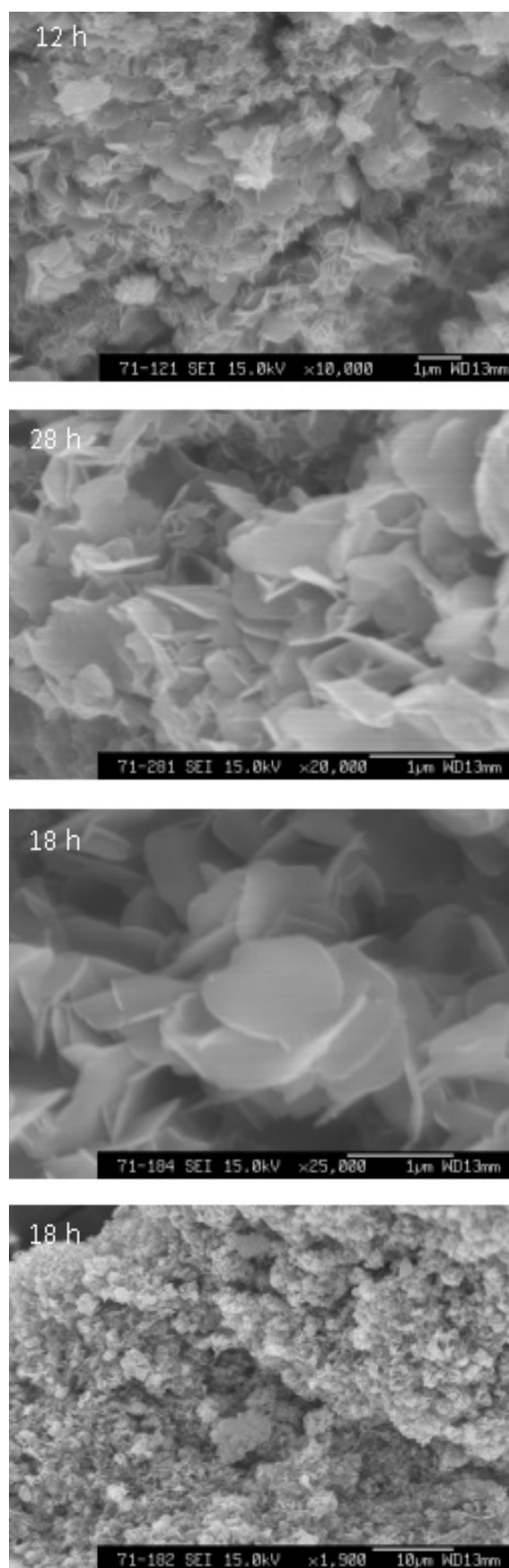


Fig. 9. Scanning Electron Microscopy images of Ni-AlLDH at diverse aging time 12-28 h.

At aging time 18 h and above, SEM images illustrated clear plate like morphology. A disc-shape was observed for the discrete plates of Ni-AlLDH with slightly curved and rounded at the edge.

In the case of Ni-Ti LDH, SEM images revealed plate-like structure. However, the individual plates are flat. it seems as sheets with irregular size and are stacking with each other as shown in Fig.10.

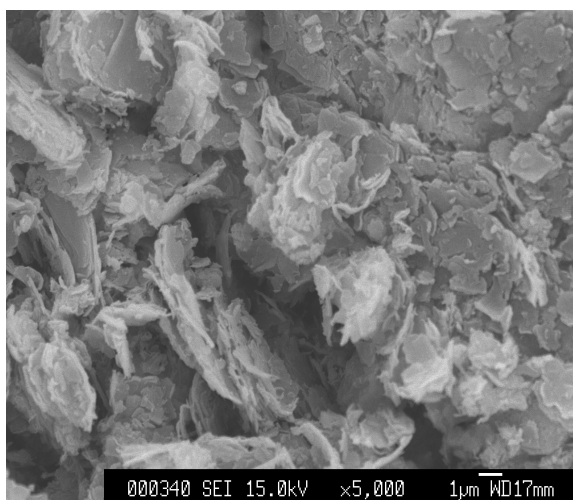
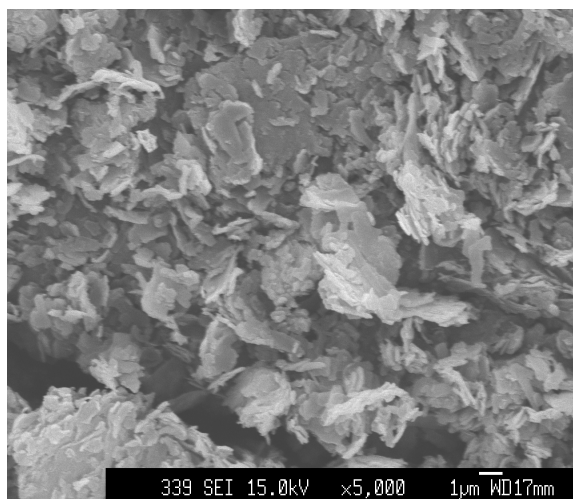
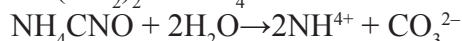
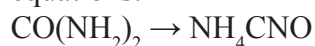


Fig. 10. Scanning Electron Microscopy images of Ni-TiLDH at aging time 18 h.

## DISCUSSION

Urea is soluble in water and it is hydrolyzed through varying the mixture temperature. Urea hydrolysis of takes place through two stages. The production of ammonium cyanate ( $\text{NH}_4\text{CNO}$ ) is the controlling step during hydrolysis. Fast decomposition of the cyanate occur and convert to ammonium

carbonate as shown in the following equations:



Because of conversion of ammonium ions to ammonia and carbonate, pH increase to be 9 and the solution become alkaline.

The earlier researchers (Shaw *et al.*, 1955) reported that during decomposition of urea the cyanate anions are produced as intermediate in the reaction. Koebel *et al.*, (1993) proclaimed that the hydrolysis of urea produce range of products such as isocyanic acid and ammonia. During the slow hydrolysis of urea, the cyanate anions release and start to react. Therefore, the trapping of cyanate anions inside LDH structure could be occurred after long time 18h. Also, the ageing time has an important role in the intercalation reaction of cyanate anions because of the slow decay of urea. The existence of tetravalent cations (Ti) having positive charges (+2) speed up the trapping of cyanate anions (+1) because one titanium cation neutralize with two cyanate anions. Therefore, the withdrawing of cyanate anions from the decomposition products of urea through trapping inside LDH directs the reaction toward production of cyanate anions instead of carbonate anions. It may clarify the complete intercalation of cyanate anions inside Ni-Ti LDH.

Finally, optimization synthesis conditions are necessary for intercalation of cyanate anions inside LDH because of its instability in the aqueous solutions. The present study concluded that it is possible to synthesis LDH with cyanate instead of carbonate in the interlayered space through urea hydrolysis. Where, Ni-Ti LDH with cyanate anions has been successfully prepared using urea hydrolysis. In addition, Ni-Al LDH with a mixture of cyanate and carbonate anions has been synthesized using the same technique. The experimental results showed that layered double hydroxides which containing titanium metal have greater affinity for cyanate than LDH structures having aluminum metal.

Consequently, this existence for cyanate in the inter-layered space expands the applicability to investigate the influence of the confinement on the toxicity of cyanate by bio researchers.

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## حصار وتخزين أيونات السيانيت في المقياس النانوي من خلال التركيبات الطبقيّة النانوية

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استلام 6 أكتوبر 2015م – قبول 14 يناير 2016م

### الملخص

أيونات السيانيت هي الأيونات التي تتكون أثناء تسمم البروتين في جسم الإنسان ولذلك تم دراستها باتساع في مجال الكيمياء الحيوية بسبب سميتها. الدراسة الحالية تهدف إلى حصار وتخزين أيونات السيانيت في المقياس النانوي. وتم ذلك من خلال تداخل أيونات السيانيت بين التركيب الطبقي النانوي المحتوي على النيكل والألمونيوم. بالإضافة إلى توضيح تأثير الزمن العمري لعملية التداخل في وجود درجة الحرارة بواسطة تحليل العينات المحضرة باستخدام حيود أشعة إكس والميكروسكوب الإلكتروني. وكذلك تم دراسة تأثير نوع العناصر الموجودة في التركيب الطبقي النانوي على قابلية تداخل أيونات السيانيت داخل التركيب الطبقي باستبدال عنصر التيتانيوم رباعي التكافؤ بدلا من الألمونيوم ثلاثي التكافؤ. حيود أشعة إكس أظهر أن المسافة البينية النانوية للنيكل والتيتانيوم هي 0.73 نانومتر وهذه المسافة تكون أصغر من مثيلتها في مركب النيكل والألمونيوم. وهذا يقترح أن الأيونات الموجودة داخل النيكل والتيتانيوم تكون مختلفة إلى حد ما عن الموجودة داخل النيكل والألمونيوم. وأظهرت التحاليل الحرارية والأشعة تحت الحمراء وجود أيونات السيانيت فقط داخل التركيب الطبقي النانوي لمركب النيكل والتيتانيوم بينما أظهرت وجود أيونات الكربونات بالإضافة إلى أيونات السيانيت داخل المركب الطبقي للنيكل والألمونيوم. وكذلك أظهرت صور الميكروسكوب الإلكتروني أن تركيب كل من مركب النيكل والتيتانيوم ومركب النيكل والألمونيوم على شكل طبقات.

واقترحت النتائج أن التركيب الطبقي النانوي المحتوي على عنصر التيتانيوم له قدرة عالية على اصطياد أيونات السيانيت لداخل التركيب النانوي أكثر من المحتوي على عنصر الألمونيوم. ولذلك فإن إدخال أيونات السيانيت سوف يتيح الفرصة لتوسيع تطبيقات السيانيت بعد دراسة تأثير حصارها في التركيب النانوي على سميتها بواسطة الباحثين في العلوم الحيوية. الكلمات المفتاحية: التحليل المائي لليوريا، تداخل أيونات السيانيت، التركيب الطبقي النانوي، طبقات ثنائية الهيدروكسيد لعناصر النيكل والألمونيوم، طبقات ثنائية الهيدروكسيد لعناصر النيكل والتيتانيوم.