

## Molecular assembly in synthesized hydrotalcites of formula $\text{Cu}_x\text{Zn}_{6-x}\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$ –a vibrational spectroscopic study

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### Abstract:

Infrared and Raman spectroscopy have been used to characterize synthetic hydrotalcites of formula  $\text{Cu}_x\text{Zn}_{6-x}\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$ . The spectra have been used to assess the molecular assembly of the cations in the hydrotalcite structure. The spectra may be conveniently subdivided into spectral features based (a) upon the carbonate anion (b) the hydroxyl units (c) water units. The Raman spectra of the hydroxyl-stretching region enable bands to be assigned to the CuOH, ZnOH and AlOH units. It is proposed that in the hydrotalcites with minimal cationic replacement that the cations are arranged in a regular array. For the  $\text{Cu}_x\text{Zn}_{6-x}\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcites, spectroscopic evidence suggests that 'islands' of cations are formed in the structure. In a similar fashion the bands assigned to the interlayer water suggest that the water molecules are also in a regular well-structured arrangement. Bands are assigned to the hydroxyl stretching vibrations of water. Three types of water are identified (a) water hydrogen bonded to the interlayer carbonate ion (b) water hydrogen bonded to the hydrotalcite hydroxyl surface and (c) interlamellar water. It is proposed that the water is highly structured in the hydrotalcite as it is hydrogen bonded to both the carbonate anion and the hydroxyl surface.

*Key Words:* hydrotalcite, infrared spectroscopy, Raman microscopy, molecular assembly, structured water.

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### 1. Introduction

Hydrotalcites both natural and synthetic have been known for an extended period of time [1-5]. Hydrotalcites, or layered double hydroxides (LDH) are fundamentally known as anionic clays, and are less well-known and more diffuse in nature than cationic clays like smectites [6-10]. The structure of hydrotalcite can be derived from a brucite structure ( $\text{Mg}(\text{OH})_2$ ) in which e.g.  $\text{Al}^{3+}$  or  $\text{Fe}^{3+}$  (pyroaurite-

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sjögrenite) substitutes a part of the  $Mg^{2+}$ . This substitution creates a positive layer charge on the hydroxide layers, which is compensated by interlayer anions or anionic complexes. In hydrotalcites a broad range of compositions are possible of the type  $[M^{2+}_{1-x}M^{3+}_x(OH)_2][A^{n-}]_{x/n}.yH_2O$ , where  $M^{2+}$  and  $M^{3+}$  are the di- and trivalent cations in the octahedral positions within the hydroxide layers with  $x$  normally between 0.17 and 0.33.  $A^{n-}$  is an exchangeable interlayer anion. Commonly the  $M^{3+}$  ion is  $Al^{3+}$  but may be any one of a number of trivalent cations including Ga, Fe, Cr [11-18].

Likewise the characterisation of these types of minerals by infrared spectroscopy goes back in time [17, 19-24]. More recently infrared emission spectroscopy has been used to study the thermal behaviour of hydrotalcites [6, 7]. The question arises as to the assembly of the cations in the hydrotalcite structures. Are the cations of Cu and Zn randomly arranged in the hydrotalcite structure? Or do the cations form lakes of individual cations in the structure? What are the effects of increasing substitution of the Cu by the Zn in the  $Cu_xZn_{6-x}Al_2(OH)_{16}(CO_3).4H_2O$  hydrotalcites? In this paper we report the changes in the structure and molecular assembly of copper based aluminium hydrotalcites using infrared spectroscopy as the Cu in the hydrotalcite  $Cu_xZn_{6-x}Al_2(OH)_{16}(CO_3).4H_2O$  is replaced by Zn.

## 2. Experimental

### 2.1 *Synthesis of hydrotalcite samples*

Hydrotalcites with a composition of  $Cu_xZn_{6-x}Al_2(OH)_{16}(CO_3).4H_2O$  where  $x$  varied from 6 to 0, were synthesised by the coprecipitation method. Three solutions were prepared, solution 1 contained 2M NaOH and 0.125M  $Na_2CO_3$ , solution 2 contained 0.75M  $Cu^{2+}$  ( $Cu(NO_3)_2.6H_2O$ ) together with 0.25M  $Al^{3+}$  ( $Al(NO_3)_3.9H_2O$ ), solution 3 contained 0.75M  $Zn^{2+}$  with 0.25M  $Al^{3+}$  ( $Al(NO_3)_3.9H_2O$ ). Solution 2 and 3 in the appropriate ratio were added to solution 1 using a peristaltic pump at a rate of 40  $cm^3/min.$ , under vigorous stirring, maintaining a pH of 10.

### 2.2 *Raman microprobe spectroscopy*

The crystals of the synthesised hydrotalcite minerals were placed and orientated on a polished metal surface on the stage of an Olympus BHSM microscope, which is equipped with 10x and 50x objectives. The microscope is part of a Renishaw 1000 Raman microscope system, which also includes a monochromator, a filter system and a Charge Coupled Device (CCD). Raman spectra were excited by a Spectra-Physics model 127 He-Ne laser (633 nm) at a resolution of 2  $cm^{-1}$  in the range between 100 and 4000  $cm^{-1}$ . Repeated acquisition using the highest magnification were accumulated to improve the signal to noise ratio in the spectra. Spectra were calibrated using the 520.5  $cm^{-1}$  line of a silicon wafer. Spectra at liquid nitrogen temperature were obtained using a Linkam thermal stage (Scientific Instruments Ltd, Waterfield, Surrey, England).

### 2.3 *Infrared Spectroscopy*

The samples were dried to remove any adsorbed water and stored in a desiccator before measurement in the FT-IR spectrometer. The sample (1mg) was finely ground for one minute, combined with oven dried spectroscopic grade KBr having a

refractive index of 1.559 and a particle size of 5-20  $\mu\text{m}$  (250mg) and pressed into a disc using 8 tonnes of pressure for five minutes under vacuum. The spectrum of each sample was recorded in triplicate by accumulating 64 scans at 4  $\text{cm}^{-1}$  resolution between 400  $\text{cm}^{-1}$  and 4000  $\text{cm}^{-1}$  using the Perkin-Elmer 1600 series Fourier transform infrared spectrometer equipped with a LITA detector.

Spectroscopic manipulation such as baseline adjustment, smoothing and normalisation were performed using the Spectralcalc software package GRAMS (Galactic Industries Corporation, NH, USA). Band component analysis was undertaken using the Jandel 'Peakfit' software package, which enabled the type of fitting, function to be selected and allows specific parameters to be fixed or varied accordingly. Band fitting was done using a Gauss-Lorentz cross-product function with the minimum number of component bands used for the fitting process. The Gauss-Lorentz ratio was maintained at values greater than 0.7 and fitting was undertaken until reproducible results were obtained with squared correlations of  $r^2$  greater than 0.995.

### 3. Results and discussion

#### 3.1 Hydroxyl Stretching Region

Hydrotalcites of formula  $\text{Cu}_x\text{Zn}_{6-x}\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  contain 16 moles of hydroxyl units from the three cations and 8 moles of hydroxyl units of water. Thus the vibrational spectra of this hydrotalcite will show both the stretching bands of these two types of hydroxyl units. Water hydroxyl-stretching vibrations are intense in infrared spectroscopy because of the large change in dipole moment. However the water bands in the Raman spectra are less intense as water is a very weak Raman scatterer. The infrared and Raman spectra of the hydroxyl-stretching region of the  $\text{Cu}_x\text{Zn}_{6-x}\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcites are shown in Figures 1 and 2. If the moles of Zn is zero, then the hydrotalcite is purely a copper hydrotalcite of formula  $\text{Cu}_6\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$ . Whereas if the moles of Zn is 6 then the hydrotalcite is purely a zinc hydrotalcite of formula  $\text{Zn}_6\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$ . Thus the spectra in Figures 1 and 2 show the spectra of the end members of the hydrotalcite series and the mixed cation hydrotalcites.

For the high zincian hydrotalcite three infrared hydroxyl-stretching bands are observed at around 3078, 3240 and 3442  $\text{cm}^{-1}$ . For the cuprian hydrotalcite four bands are observed at 3214, 3398, 3519 and 3608  $\text{cm}^{-1}$ . For the  $\text{Cu}_3\text{Zn}_3\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcite five bands are observed at 3078, 3286, 3432, 3544 and 3592  $\text{cm}^{-1}$ . The assignment of the bands is difficult because of the initial complex band profile and the overlap of the bands. One possible model is that the band with the lowest wavenumber at around 3078  $\text{cm}^{-1}$  is attributed to a very strongly hydrogen bonded water molecule. Such a strong bond is formed when the water hydrogen bonds to the carbonate ion. The second band is assigned to strongly hydrogen bonded water, which coordinates the cation hydroxyl surface. Figure 3a shows the variation of the band position with increasing replacement of the Cu by Zn. In general there is a shift of the band positions towards lower wavenumbers as the Cu is replaced by Zn. The band at 3608  $\text{cm}^{-1}$  for the  $\text{Cu}_6\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcite may be ascribed to the AlOH stretching vibration and the band at 3519  $\text{cm}^{-1}$  to the CuOH hydroxyl stretching vibration. The ratio of the relative intensities

of these two bands is 0.35, which is close to the theoretical value. The observation of two bands suggests that the cupric cations and aluminium cations are separate entities. In contrast for the  $\text{Cu}_4\text{Zn}_2\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcite a broad band is observed at  $3488\text{ cm}^{-1}$ . One possibility is that this band is formed from a continuum of states of all three cationic hydroxyl units, namely CuOH, ZnO and AlOH. A similar result is observed for the  $\text{Cu}_2\text{Zn}_4\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  and  $\text{Zn}_6\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcites. For the  $\text{Cu}_3\text{Zn}_3\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcite, bands are observed at  $3592$  and  $3544\text{ cm}^{-1}$ . One possible assignment is to attribute the first band to the AlOH stretching vibration and the second to the CuOH and ZnOH stretching bands. If this description is true, then this is vibrational spectroscopic evidence for the formation of cationic lakes in the hydrotalcite structure. In other words the Cu cations form groups in the structure.

The overall spectra profiles are different between the infrared and the Raman spectra. The Raman spectra appear more broad and with a relative lower intensity. The infrared spectra appear to maximise at around  $3440\text{ cm}^{-1}$ . In the Raman spectra the low wavenumber hydroxyl-stretching band at around  $3078\text{ cm}^{-1}$  is not observed. The bands, which appear around  $3220\text{ cm}^{-1}$  are attributed to the hydroxyl stretching unit of water, which coordinates the cationic hydroxyl surface of the hydrotalcite. The second Raman band appears to vary in position according to the cationic ratio. Figure 3b illustrates this variation. This band is by far the most intense band in the infrared spectrum. This band is assigned to water which is hydrogen bonded to other water molecules in the hydrotalcite interlayer space. As for the assignment of the infrared bands the band at  $3602\text{ cm}^{-1}$  is attributed to the AlOH hydroxyl stretching vibration and the band at  $3519\text{ cm}^{-1}$  to the CuOH hydroxyl stretching vibration. The bands appear to shift to higher wavenumbers as the Cu is replaced by the Zn in the hydrotalcite structure (Figure 3b).

### 3.2 Carbonate vibrations

The structure of hydrotalcites depends on the balance of the positive surface charge on the cationic hydroxyl surfaces by the negative charges of the anion in this case carbonate. Hence double layer hydroxides are termed anionic clays. The unperturbed carbonate ion is a planar triangle with point symmetry  $D_{3h}$ . Group theoretical analysis of the carbonate ion predicts four normal modes the  $\nu_1$  symmetric stretch of  $A_1'$  symmetry normally observed at  $1063\text{ cm}^{-1}$ , the asymmetric stretch of  $E'$  symmetry observed at  $1415\text{ cm}^{-1}$ , the  $\nu_2$  out of plane bend at  $879\text{ cm}^{-1}$  and the in-plane bend at  $680\text{ cm}^{-1}$ . All modes are both Raman and infrared active except for the  $\nu_2$  mode, which is IR active only.

Figure 4 presents the infrared spectra of the CO antisymmetric stretching region. Two bands are observed at around  $1403$  and  $1344\text{ cm}^{-1}$ . The asymmetric stretching mode of the free carbonate is normally observed at  $1415\text{ cm}^{-1}$ . The observation of two or more vibrations for the E mode is an indication of the loss of symmetry from  $D_{3h}$  to a lower symmetry species such as  $C_{2v}$ . Such a result can be modelled by the carbonate and water forming a single unit. The two hydrogens of water hydrogen bonding to the two carbonate oxygens forms such a model. The hydroxyl stretching vibration around  $3060\text{ cm}^{-1}$  is assigned to the water hydroxyl stretching vibrations of this unit. Figure 5 displays the variation of the band centres of the CO stretching vibrations as a function of increasing Zn content. The  $1405\text{ cm}^{-1}$

band remains constant in band position and is independent of the hydrotalcite composition. The  $1344\text{ cm}^{-1}$  band does shift to higher wavenumbers with increasing Zn content. A conclusion that may be made is that this band is actually composed of two component bands with the  $1344\text{ cm}^{-1}$  band assigned to the carbonate anion associated with CuOH units and the band at  $1363\text{ cm}^{-1}$  associated with ZnOH units. In the Raman spectra of the Cu/Zn hydrotalcites a low intensity complex band is observed centred upon  $1405\text{ cm}^{-1}$ . The antisymmetric stretching vibration is very weak in the Raman spectrum. Two bands may be resolved at  $1405$  and  $1345\text{ cm}^{-1}$ .

In the infrared spectra of the hydrotalcites bands are observed at around  $1049$  and  $1065\text{ cm}^{-1}$  and are assigned to the forbidden symmetric stretching vibrations. The band at  $1065\text{ cm}^{-1}$  appears to increase in intensity as the Cu is replaced by Zn. The Raman spectra of the Cu/Zn based hydrotalcites are difficult to measure as these materials are coloured and absorb the incident excitation of the laser beam. The samples tend to decompose under the laser beam even though low intensities of the incident excitation radiation are used. Nevertheless Raman bands are observed at  $1053$ ,  $1065$  and  $1085\text{ cm}^{-1}$ .

### 3.3 *Water OH deformation vibrations*

One means of studying the molecular assembly of water molecules in the hydrotalcites is to study the water HOH deformation modes. Minerals containing physically adsorbed water give strong infrared hydroxyl stretching bands at  $3450\text{ cm}^{-1}$ , the water hydroxyl stretching vibration, and at  $\sim 1630\text{ cm}^{-1}$ , the water bending vibrations. The position of these bands is influenced by the amount of adsorbed water, the mineral type and the exchangeable cation to which the water is bonded. For monomeric non-hydrogen bonded water as occurs in the vapour phase, these bands are found at  $3755$  and  $1595\text{ cm}^{-1}$ . For liquid water the bands occur at  $3455$  and  $1645\text{ cm}^{-1}$  and for water molecules in ice, the bands are at  $3255$  and  $1655\text{ cm}^{-1}$ . When water molecules are very tightly bound to the mineral surface as may occur with hydrotalcites, then bands occur in the  $3200$  to  $3250\text{ cm}^{-1}$  region. What is being distinguished here is the formation of strong and weak hydrogen bonds. The hydroxyl stretching modes of weak hydrogen bonds occur in the  $3580$  to  $3500\text{ cm}^{-1}$  region and the hydroxyl stretching modes of strong hydrogen bonds occurs below  $3420\text{ cm}^{-1}$ . When the water is coordinated to the cation in the clays as occurs in certain minerals, then the water OH stretching frequency occurs at  $3220\text{ cm}^{-1}$ . A simple observation can be made that as the water OH stretching frequency decreases then the HOH bending frequency increases. Thus the water hydroxyl stretching and the water HOH bending  $1610\text{ cm}^{-1}$  frequencies provide a measure of the strength of the bonding of the water molecules either chemically or physically to the hydrotalcite surfaces or to the interlayer anions. Likewise the position of the water bending vibration also provides a measure of this strength of water hydrogen bonding. Bands that occur at frequencies above  $1650\text{ cm}^{-1}$  are indicative of coordinated water and chemically bonded water. Bands that occur below  $1630\text{ cm}^{-1}$  are indicative of water molecules that are not as tightly bound. In this case the hydrogen bonding is less as the frequency decreases.

Figure 7 displays the water HOH bending modes for the  $\text{Cu}_x\text{Zn}_{6-x}\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcites. For the cupric hydrotalcite, three bands are observed at  $1622$ ,  $1643$  and  $1670\text{ cm}^{-1}$ . The first band is not observed for

the other hydrotalcites. The band at around  $1643\text{ cm}^{-1}$  is observed in all the spectra. Although some variation in the position of the  $1670\text{ cm}^{-1}$  band is observed, the high wavenumber component is observed in all the spectra. The band at  $1622\text{ cm}^{-1}$  is attributed to adsorbed water. The band at  $1643\text{ cm}^{-1}$  is assigned to water which is strongly hydrogen bonded. Such water molecules may be hydrogen bonded to the cationic hydroxyl surface or to adjacent water molecules. The observation of bands in the high wavenumber region of the water HOH bending region is significant. It means that the water is highly structured. The water molecules are in well defined positions in the interlayer of the hydrotalcites. The water may be termed “structured water”. Indeed the molecules of water are in a molecular assembly of water molecules and anions in a regular array.

#### 4. Conclusions

Insight into the unique structure of hydrotalcites has been obtained using a combination of infrared and Raman spectroscopy. The hydroxyl-stretching units of CuOH, ZnOH and AlOH, are identified by unique band positions. The identification of unique bands or lack thereof for these units makes it possible to assess whether there is a unique and regular arrangement of cations in the hydrotalcite structure. It is proposed that for  $\text{Cu}_x\text{Zn}_{6-x}\text{Al}_2(\text{OH})_{16}(\text{CO}_3)\cdot 4\text{H}_2\text{O}$  hydrotalcites when  $x$  is less than or equal to two, that the cations are randomly assembled. However when  $x=3$ , separate bands for each of the cationic hydroxyls can be identified. This means that ‘lakes’ or ‘islands’ of the cations are formed in the hydrotalcite structures

Water plays a unique role in the stabilisation of the hydrotalcite structure. The position and intensity of the vibrational spectroscopic bands in the hydroxyl-stretching region indicates that the water is highly structured. The position of the bands in the hydroxyl deformation region of the infrared spectrum supports the concept of structured water between the hydrotalcite layers. Three types of water are identified (a) water hydrogen bonded to the interlayer carbonate ion (b) interlamellar water (c) water hydrogen bonded to the hydroxyl units of the hydroxyl surface. It is proposed that the water is highly structured within the hydrotalcite layers and is in a regular structure such that the water is hydrogen bonded to the carbonate anion and to the cationic hydroxyl surface.

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