

## PREPARATION OF MONTMORILLONITE-NYLON COMPLEXES AND THEIR THERMAL PROPERTIES

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**Abstract**—Montmorillonite-aminocaproic acid complexes (monomer complexes) were prepared by the intercalation of 6-aminocaproic acid to various homoionic ( $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Co}^{2+}$ , and  $\text{Cu}^{2+}$ ) montmorillonites. Infrared spectra of the monomer complexes indicated that the interaction between the exchangeable cations and the 6-aminocaproic acid increased in the following order:  $\text{Na-} < \text{Ca-} < \text{Mg-} < \text{Co-} < \text{Cu-}$  montmorillonite-aminocaproic acid complex. Montmorillonite-nylon complexes (polymer complexes) were prepared by thermal treatment of the monomer complexes, which was confirmed by X-ray powder diffraction and infrared spectroscopy the results of which indicated the condensation of 6-aminocaproic acid in the interlayer space.

Thermal degradation of montmorillonite-nylon complexes was studied by thermogravimetry. It was found that the thermal stability of the polymer complexes increased in the following order:  $\text{Cu-} < \text{Co-} < \text{Na-} < \text{Mg-} < \text{Ca-}$  montmorillonite-nylon complex.

It was suggested that the difference in thermal stability depended upon the length of the polymer chain which might be influenced by the interaction between the exchangeable cations and the 6-aminocaproic acid. The activation energy for the thermal degradation of each montmorillonite-nylon complex was obtained, and the value for Cu-montmorillonite-nylon complex was smaller than that for the other cation-exchanged montmorillonite-nylon complexes.

**Key Words**—Aminocaproic Acid, Intercalation, Montmorillonite, Nylon, Polymer

### INTRODUCTION

It is well known that clay reacts with organic reagents to give clay-organic complexes. Giesecking (1939), Hendricks (1941), MacEwan (1944), and other investigators studied the clay-organic complexes systematically, and many studies on the clay-organic polymer complexes have been also reported (Imoto, 1963; Solomon and Rosser, 1965; Solomon, 1968; Solomon and Loft, 1968; Blumstein, 1965a, 1965b; Blumstein and Billmeyer, 1966; Blumstein *et al.*, 1969, 1970, 1971, 1972). However, there have been few studies on the thermal properties of the clay-polymer complexes, though Blumstein (1965b) stated the isothermal degradation of montmorillonite-poly(methyl methacrylate) complex.

In the present work, montmorillonite-aminocaproic acid complexes (monomer complexes) were prepared by the reaction of homoionic ( $\text{Na-}$ ,  $\text{Ca-}$ ,  $\text{Mg-}$ ,  $\text{Co-}$ , and  $\text{Cu-}$ ) montmorillonites with 6-aminocaproic acid. Further, each monomer complex was polymerized to yield montmorillonite-nylon complex (polymer complex), and the thermal properties of the polymer complexes were investigated.

### EXPERIMENTAL

#### *Preparation of materials*

**Homoionic montmorillonites.** Montmorillonite from Yamagata, Japan, was used to prepare homoionic phases by the ion-exchange treatment with 1 N solutions of

$\text{NaCl}$ ,  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{CoCl}_2$ , and  $\text{CuCl}_2$ . These samples were washed with methyl alcohol until free of  $\text{Cl}^-$ .

**Montmorillonite-aminocaproic acid complexes (monomer complexes).** Ten grams of homoionic montmorillonite was immersed in every 6-aminocaproic acid solution (50, 100, 200, 300, 500, and 700 meq/100 g clay) and allowed to stand for several days. After drying in vacuo, the complexes were ground to pass 100 mesh and washed with n-hexane in a Soxhlet extractor for 6 hours.

**Montmorillonite-nylon complexes (polymer complexes).** Polymer complexes were prepared by the thermal treatment of monomer complexes at 240–250°C for 1 hour in  $\text{N}_2$  flow.

#### *Identification and analyses*

**X-ray powder diffraction.** The basal spacings of all complexes were determined by the X-ray powder diffraction data which were obtained by a Shimadzu VD-11 diffractometer ( $\text{CuK}\alpha$ , Ni filter).

**Infrared spectroscopy.** Infrared spectra of the self-supporting films, which were prepared by air-drying 1% suspensions on a mirror plate, were recorded on a Shimadzu IR-400 spectrometer.

**Thermogravimetry.** Thermogravimetric data were obtained by a Shimadzu MTG-1. The operating conditions were as follows: heating rate 10°C/min; sample

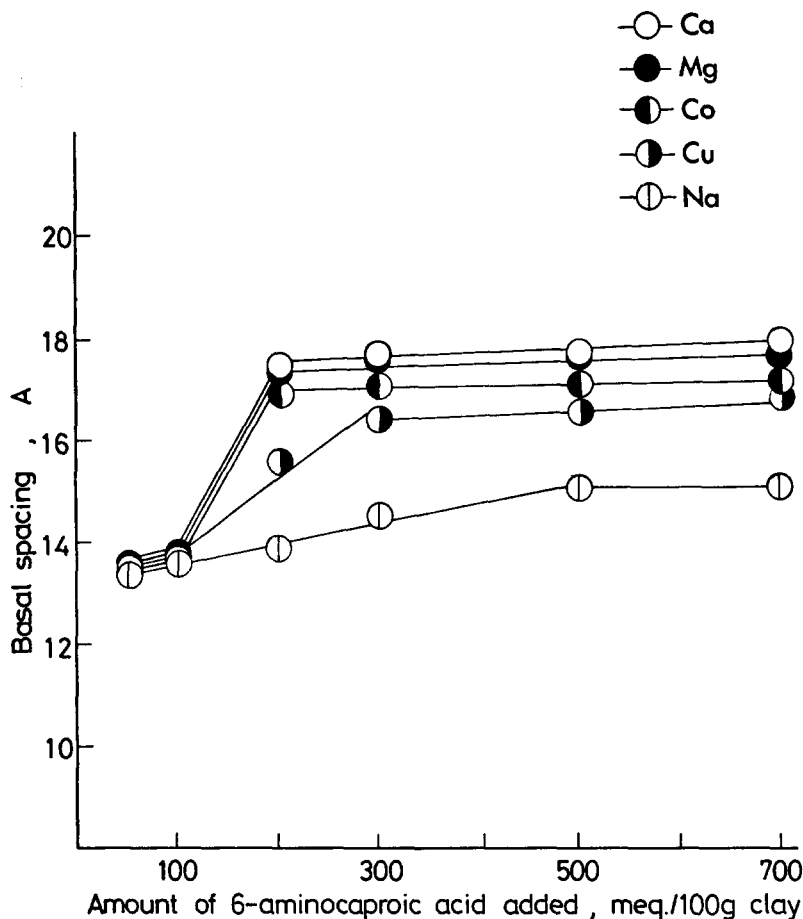


Figure 1. Basal spacings of montmorillonite-aminocaproic acid monomer complexes.

weight 200 mg;  $N_2$  flow. The activation energy for the decomposition of each sample was determined by Reich's equation (Reich *et al.*, 1963) expressed as follows:

$$\ln \frac{T_1^2}{\phi_1} - \frac{E}{RT_1} = \ln \frac{T_2^2}{\phi_2} - \frac{E}{RT_2}$$

where  $E$  = activation energy,  $R$  = gas constant,  $\phi$  = heating rate, and  $T_1$  and  $T_2$  = temperature at same weight loss at different heating rates  $\phi_1$  and  $\phi_2$ .

## RESULTS AND DISCUSSION

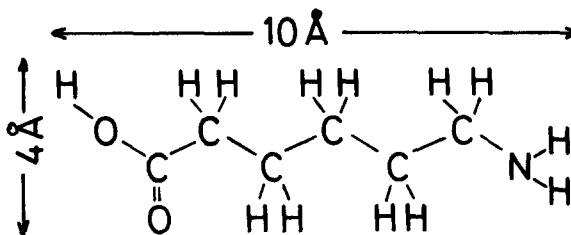
### Formation of montmorillonite-aminocaproic acid monomer complexes

Figure 1 shows the variation in the basal spacings of montmorillonite-aminocaproic acid monomer complexes against the amount of the 6-aminocaproic acid added. The interlayer spacings of the monomer complexes are listed in Table 1. Infrared spectral results in the region of  $1800$ – $1200\text{ cm}^{-1}$  are shown in Figure 2, and their assignments are given in Table 2.

Figure 1 indicates that the  $d(001)$  spacings of the

monomer complexes increased with an increase of the amount of aminocaproic acid. From these findings and IR data, it was proven that 6-aminocaproic acid was intercalated in the interlayer space of each homoionic montmorillonite.

The molecular size of 6-aminocaproic acid is roughly illustrated below.



Considering the results in Table 1, the interlayer spacing of the Na-montmorillonite-aminocaproic acid complex increased gradually, and it was considered that aminocaproic acid was adsorbed as a monomolecular layer and that the increase of the amount of the reagent

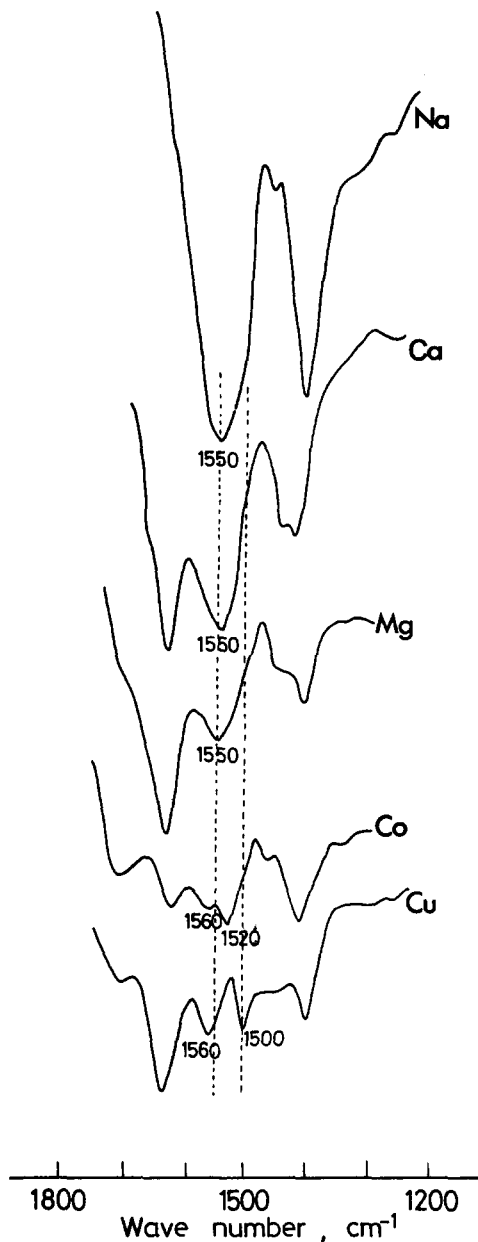


Figure 2. Infrared spectra of montmorillonite-aminocaproic acid monomer complexes.

added might result in the orientation of the molecules inclined to the oxygen surface.

However, in the case of the other cation-exchanged montmorillonite-aminocaproic acid complexes, the distinct changes of  $\Delta d$  were recognized between the range of 100 meq/100 g clay and 200 meq/100 g clay.

Estimating the size of the 6-aminocaproic acid, it is suggested that a monomolecular layer was formed in the range less than 100 meq/100 g clay and that a double layer was produced beyond 200 meq/100 g clay. The

Table 1. Interlayer spacings of montmorillonite-aminocaproic acid monomer complexes.

6-aminocaproic acid (meq/100 g clay)	Interlayer spacing $\Delta d = d(001) - 9.6 \text{ \AA}$				
	Na-mont.	Mg-mont.	Ca-mont.	Cu-mont.	Co-mont.
50	3.6	3.8	3.8	3.8	3.8
100	3.8	3.8	3.8	3.8	3.8
200	4.1	7.7	7.7	5.6	7.2
300	4.7	7.7	7.9	6.6	7.4
500	5.2	7.9	7.9	6.9	7.5
700	5.2	7.9	8.2	7.2	7.5

variation of the spacings in Ca-, Mg-, and Co-montmorillonite-aminocaproic acid complexes showed the typical tendency.

It is worth noting that the band around  $1550 \text{ cm}^{-1}$  showed various shapes with the kind of cations, as shown in Figure 2. The spectra of the Na-, Ca-, and Mg-montmorillonite-aminocaproic acid complexes showed the strong band at  $1550 \text{ cm}^{-1}$  assigned to  $\nu\text{-COO}^- + \delta\text{-NH}_3^+$ , whereas the spectrum of Co-montmorillonite-aminocaproic acid complex produced the resolved peaks at  $1560 \text{ cm}^{-1}$  and  $1520 \text{ cm}^{-1}$ . The two clear peaks at  $1560 \text{ cm}^{-1}$  and  $1500 \text{ cm}^{-1}$  were recognized in the spectrum of the Cu-montmorillonite-aminocaproic acid complex. The peak at  $1560 \text{ cm}^{-1}$  in Co- and Cu-complexes was due to  $\nu\text{-COO}^-$  and the peaks at  $1520 \text{ cm}^{-1}$  in Co-complex and  $1500 \text{ cm}^{-1}$  in Cu-complex were attributed to  $\delta\text{-NH}_3^+$ . Therefore, it was found that the band due to  $\delta\text{-NH}_3^+$  shifted to a lower frequency in the case of Co- and Cu-montmorillonite-aminocaproic acid complexes. This fact may be explained by the idea that the Cu or Co cation has the capability of coordinate bond formation and that its linking force to  $\text{NH}_2$  groups is stronger than that of other cations.

From the results described above, it may be concluded that the bonding strength of each homoionic montmorillonite to 6-aminocaproic acid is in the following order:  $\text{Cu} > \text{Co} > \text{Na}$ -, Ca-, Mg-montmorillonite-aminocaproic acid complexes.

#### Formation of montmorillonite-nylon polymer complexes

Figure 3 shows the X-ray powder diffraction data of the samples obtained by the thermal treatment of mont-

Table 2. Main features of the infrared spectra of montmorillonite-aminocaproic acid monomer complexes.

Absorption band ( $\text{cm}^{-1}$ )					
Na-mont.	Mg-mont.	Ca-mont.	Co-mont.	Cu-mont.	Assignment
	1640	1640	1640	1640	$\delta\text{-OH}$
1550, br	1550, br	1550, br	1560	1560	$\nu\text{-COO}_{(\text{as})}^-$
			1520	1500	$\delta\text{-NH}_3^+$
1450, w	1440, sh	1440, sh	1450, w	1450, w	$\delta\text{-CH}$
1400	1410	1410	1410	1400	$\nu\text{-COO}_{(\text{s})}^-$

br: broad; sh: shoulder; w: weak.

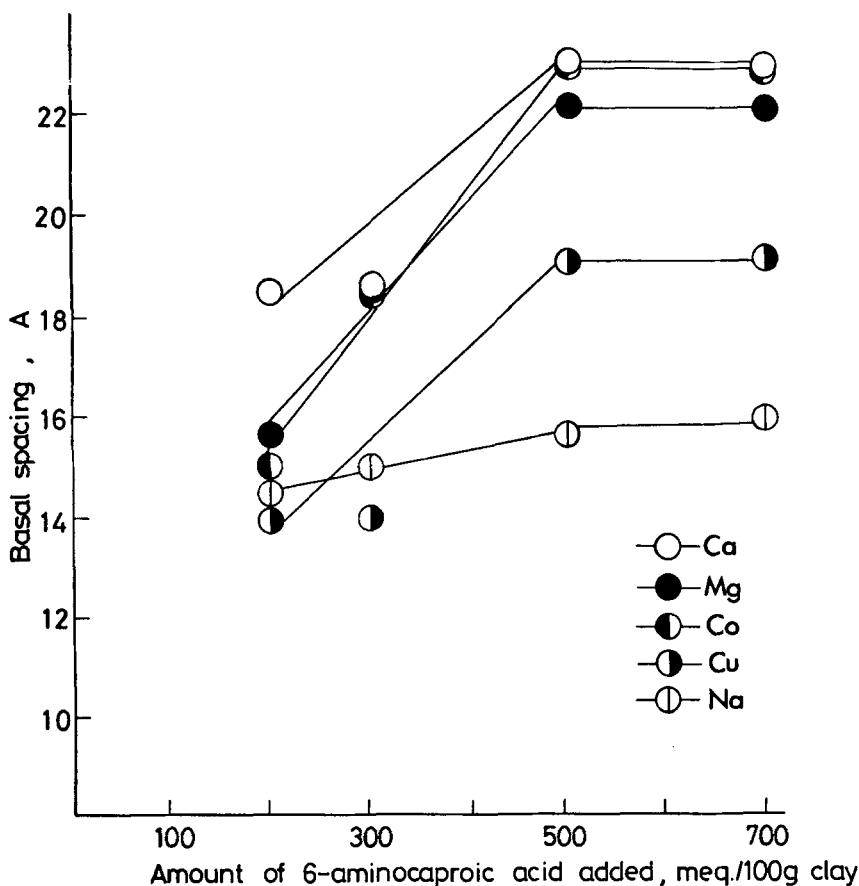


Figure 3. Basal spacings of heat-treated montmorillonite-aminocaproic acid complexes.

morillonite-aminocaproic acid monomer complexes. The interlamellar spacings calculated by the subtraction of 9.6 Å (the basal spacing of montmorillonite) are listed in Table 3. As shown in Table 3, the thermal treatment of monomer complexes caused the expansion of the interlayer spacings rather than the reduction of them. These findings suggested that 6-aminocaproic acid was condensed to yield nylon in the interlayer space. The influence of water on the spacings was neglected because the complexes had been heated at 240–250°C. The degree of increase of the interlayer spacings varied with the kind of the exchangeable cations. The

Table 3. Interlayer spacings of the heat-treated montmorillonite-aminocaproic acid complexes.

6-aminocaproic acid (meq/100 g clay)	Interlayer spacing $\Delta d$ (Å)				
	Na-mont.	Mg-mont.	Ca-mont.	Cu-mont.	Co-mont.
200	4.6	5.9	8.6	4.2	5.2
300	5.2	8.6	8.8	4.2	8.6
500	5.9	12.4	13.2	9.4	13.2
700	6.1	12.4	13.2	9.4	13.2

phenomena will be discussed below. Figure 4 gives the infrared spectra of (1) the Na-montmorillonite-aminocaproic acid complex and (2) the complex treated thermally at 240°C for 1 hour. Other cation-exchanged montmorillonite-organic complexes showed similar patterns. The assignment of the absorption peaks is given in Table 4. As Figure 4 shows, the disappearance of the band at 1400  $\text{cm}^{-1}$  due to  $\nu_{\text{S}}\text{-COO}^-$  and the appearance of the band at 1610  $\text{cm}^{-1}$  assigned to amide I ( $\nu\text{-CO}$ ) and also the band at 1540  $\text{cm}^{-1}$  due to amide II ( $\delta\text{-NH} + \nu\text{-CN}$ ) in the spectrum of the heat-treated

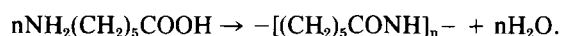
Table 4. Main features of the infrared spectra of monomer complex and the heat-treated complex (Na-montmorillonite-aminocaproic acid complex).

Absorption band ( $\text{cm}^{-1}$ )		
Monomer complex	Heat-treated complex	Assignment
—	1610	Amide I ( $\nu\text{-CO}$ )
1550	—	$\nu\text{-COO}_{(\text{as})}^-$ , $\delta\text{-NH}_3^+$
—	1540	Amide II ( $\delta\text{-NH}$ , $\nu\text{-CN}$ )
1450	1450	$\delta\text{-CH}$
1400	—	$\nu\text{-COO}_{(\text{s})}^-$

Table 5. Peak temperature of DTG curves.

6-aminocaproic acid (meq/100 g clay)	Temperature (°C)				
	Na-mont.	Mg-mont.	Ca-mont.	Co-mont.	Cu-mont.
200	420	470	495	420	350, 405
300	420	465	500	420	350, 405
500	430	460	450, 510	420	360, 415
700	430	470	460, 520	420	365

product indicated that aminocaproic acid was polymerized to give nylon by the thermal treatment as expressed in the following formula:



From the X-ray data and the infrared spectral results described above, it was found that a montmorillonite-nylon complex (polymer complex) was formed by the condensation of aminocaproic acid in the interlayer space of the montmorillonite-aminocaproic acid complex (monomer complex).

#### Thermal properties of polymer complexes (montmorillonite-nylon complexes)

Figure 5 shows TGA curves of  $\text{M}^{n+}$ -montmorillonite-nylon complexes obtained by thermal treatment of  $\text{M}^{n+}$ -montmorillonite-aminocaproic acid complexes prepared in the ratio of 200 meq aminocaproic acid/100 g clay. Though the TGA curves of the complexes showed similar thermal behavior, the initial temperature of the weight loss shifted to a higher temperature in the following order: Cu-, Co-, Na- = Mg-, Ca-montmorillonite-nylon complex.

The DTG curves obtained by the differentiation of the TGA curves at every 10°C interval are given in Figure 6. These curves reveal that the maximum degradation temperature of polymer complexes depended upon the kind of interlayer cations. Thus, the thermal stability of Ca- and Mg-montmorillonite-nylon complexes was relatively higher than that of other complexes. On the contrary, Cu- and Co-montmorillonite-nylon complexes showed relatively lower stability. The relation between the kind of exchangeable cation and the peak temperature of DTG curves is summarized in Table 5.

As is shown in Figure 3, X-ray powder diffraction results indicated that the interlayer spacings of  $\text{M}^{2+}$ -montmorillonite-nylon complexes were larger than that of the Na-montmorillonite-nylon complex. The number of divalent cations which exist in the interlayer space

Table 6. Activation energy of the polymer complexes.

Activation energy (kcal/mol)				
Na-mont.	Mg-mont.	Ca-mont.	Co-mont.	Cu-mont.
35	34	37	31	26

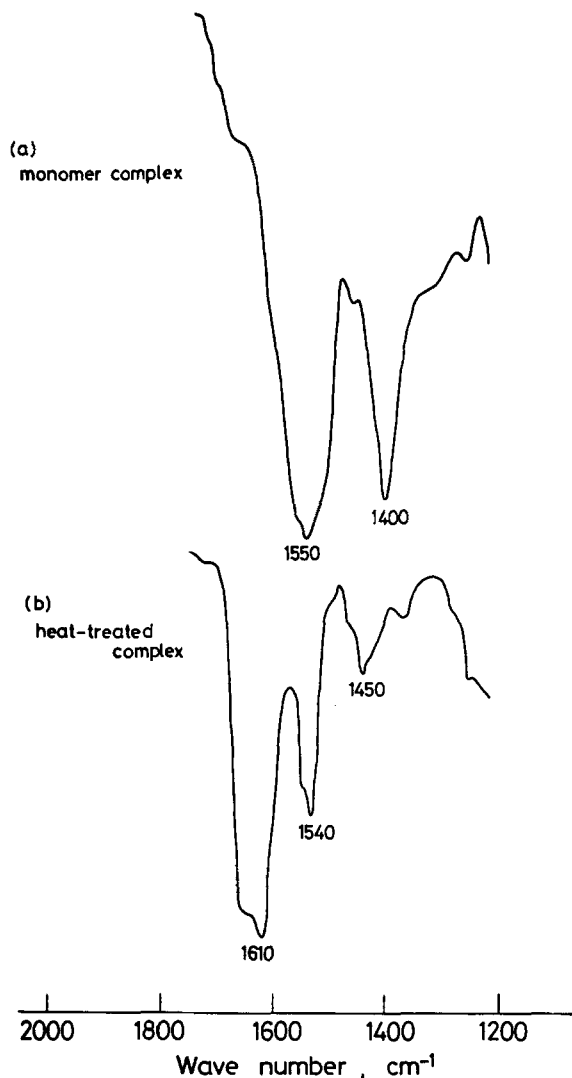


Figure 4. Infrared spectra of (a) Na-montmorillonite-aminocaproic acid complex and (b) the complex treated thermally at 240°C for 1 hr.

is half the number of monovalent cations theoretically. So the inhibition of the formation of the polymer chain by the interlayer cation might be reduced compared with that of the monovalent complex. Consequently, a longer polymer chain was thought to be formed, and it is suggested that the thermal stability was increased.

However, in comparing the divalent montmorillonite-nylon polymer complexes, the Co-complex decom-

Table 7. Peak temperature of DTG curves of the organic compounds after the HF treatment of the polymer complexes.

Temperature (°C)		
Na-mont.	Ca-mont.	Cu-mont.
370	410	340

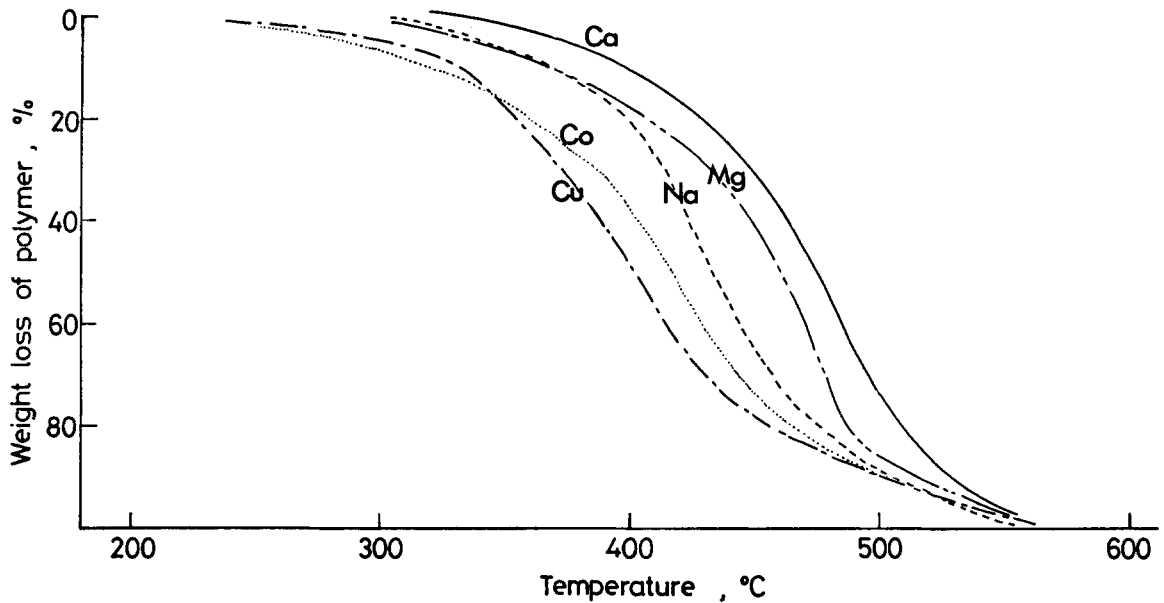


Figure 5. TGA curves of montmorillonite-nylon polymer complexes.

posed at a relatively lower temperature, and the degradation temperature of the Cu-complex was lower than that of the Na-complex. Thus, the transition metals Co and Cu may have played a catalytic role in the

degradation. In order to clarify these phenomena, the activation energy for the degradation of polymer complexes was determined by Reich's equation (1963), and the results are given in Table 6. The activation energy

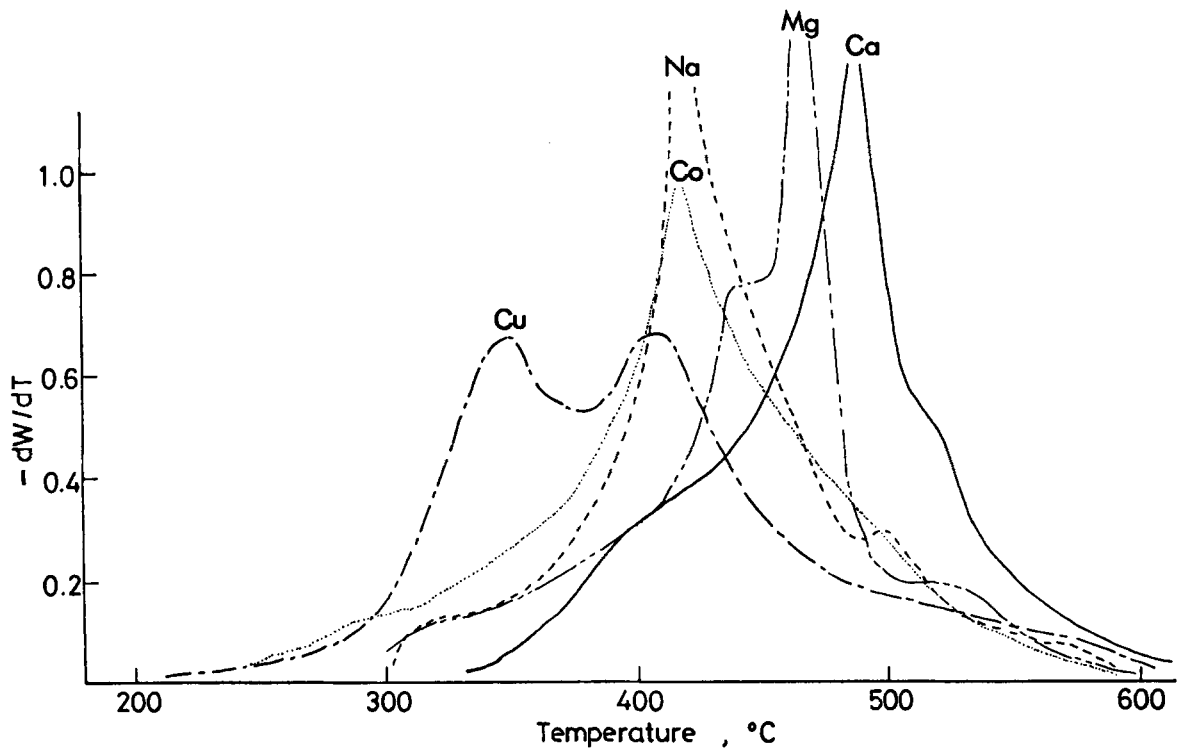


Figure 6. DTG curves of montmorillonite-nylon polymer complexes.

in the case of Co- and Cu-montmorillonite-nylon complexes was lower than that of other complexes. Shirai *et al.* (1974) concluded that the degradation of PVA was accelerated by the complexing of PVA with Cu and that the activation energy was lowered. In the present work, the infrared spectral results (Figure 2) indicate that Co and Cu were bonded to aminocaproic acid more strongly, suggesting that similar thermal behavior was observed because of this effect.

Interlayer polymers were liberated from the clay crystal by HF treatment of montmorillonite-nylon polymer complexes, and the extracted nylon samples were analyzed by thermogravimetry. Table 7 gives the maximum degradation temperature obtained from the DTG curves. The extracted samples decomposed at temperatures lower than those in polymer complexes. From this finding, it became apparent that the thermal stability of nylon was improved by complex formation with clay.

### CONCLUSION

6-aminocaproic acid was intercalated to the interlayer space of Na-, Ca-, Mg-, Co-, or Cu-montmorillonite to yield montmorillonite-aminocaproic acid complexes. It was found that Cu in Cu-montmorillonite-aminocaproic acid complex was strongly bonded to the aminocaproic acid. Thermal treatment of montmorillonite-aminocaproic acid complexes caused the condensation of the aminocaproic acid in the interlayer space, and montmorillonite-nylon complexes were formed. From the thermogravimetric results for the polymer complexes, it was found that the thermal stability of the complexes decreased in the following order: Ca-, Mg-, Na-, Co-, Cu-montmorillonite-nylon complex. It was also clarified that the thermal degradation in Co- and Cu-montmorillonite-nylon complexes which inhibited the formation of polymer chains because of the strong bonding of cations to the aminocaproic acid occurred at lower temperature, whereas the thermal stability of Ca- and Mg-montmorillonite-nylon complexes which inhibited the formation to a lesser degree was relatively higher. This finding was supported by the results for activation energy, in which the Cu-montmorillonite-nylon complex had the smallest.

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**Резюме**—Комплексы монтмориллонит-аминокапроновая кислота (мономерные комплексы) были приготовлены интеркаляцией 6-аминокапроновой кислоты в различные гомоионные ( $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Co}^{2+}$ , и  $\text{Cu}^{2+}$ ) монтмориллониты. Инфракрасные спектры мономерных комплексов указывали, что взаимодействие между обменными катионами и 6-аминокапроновой кислотой возрастает в следующем порядке:  $\text{Na}$ -,  $\text{Ca}$ -, и  $\text{Mg}$ - $\text{Co}$ - $\text{Cu}$ -монтмориллонит-аминокапроновый кислотный комплекс. Комплексы монтмориллонит-нейлон (полимерные комплексы) приготавливались термальной обработкой мономерных комплексов, наличие которых подтверждалось порошковым методом рентгеноструктурного анализа и инфракрасной спектроскопией, которые указывали на конденсацию 6-аминокапроновой кислоты в межслойных промежутках.

Термальная деградация комплексов монтмориллонит-нейлон изучалась с помощью термогравиметрии. Было обнаружено, что термальная стабильность полимерных комплексов возрастает в следующем порядке:  $\text{Cu}$ - $\text{Co}$ - $\text{Na}$ - $\text{Mg}$ - $\text{Ca}$ -монтмориллонит-нейлоновый комплекс.

Предполагается, что различие в термальной стабильности зависит от длины полимерной цепи, на которую должно влиять взаимодействие обменных катионов и 6-аминокапроновой кислоты. Была получена активационная энергия для термальной деградаций каждого комплекса монтмориллонит-нейлон, и ее значение для комплекса  $\text{Cu}$ -монтмориллонит-нейлон оказалось меньше, чем для других катионно-обменных комплексов монтмориллонит-нейлон.

**Resümee**—Montmorillonit-Aminocapronsäure Komplexe (monomere Komplexe) wurden durch die Interkalation von 6-Aminocapronsäure in verschiedene homoionische ( $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Co}^{2+}$ , und  $\text{Cu}^{2+}$ ) Montmorilloniten hergestellt. Infrarotspektren der monomeren Komplexe deuteten an, daß der Einfluß, den die austauschbaren Kationen und die 6-Aminocapronsäure aufeinander haben, in der folgenden Richtung zunimmt:  $\text{Na}$ -,  $\text{Ca}$ -, und  $\text{Mg}$ - $\text{Co}$ - $\text{Cu}$ -Montmorillonit-Aminocapronsäurekomplex. Montmorillonit-Nylonkomplexe (polymere Komplexe) wurden durch die thermische Behandlung der monomeren Komplexe präpariert, was durch Röntgenpulverdiagramme und Infrarotspektroskopie, welche die Kondensation von 6-Aminocapronsäure in den Zwischenschichträumen andeuten, bestätigt wurde. Thermische Degradation von Montmorillonit-Nylonkomplexen wurde mittels Thermogravimetrie untersucht. Es wurde gefunden, daß die thermische Stabilität der polymeren Komplexe folgendermaßen zunimmt:  $\text{Cu}$ - $\text{Co}$ - $\text{Na}$ - $\text{Mg}$ - $\text{Ca}$ -Montmorillonit-Nylonkomplex. Es wurde vorgeschlagen, daß der Unterschied in den thermischen Stabilitäten von der Länge der polymeren Kette abhängt, welche durch die Einwirkung der austauschbaren Kationen auf die 6-Aminocapronsäure beeinflußt sein könnte. Die Aktivierungsenergie für die thermische Degradation für jeden Montmorillonit-Nylonkomplex wurde erhalten; der Wert für den  $\text{Cu}$ -Montmorillonit-Nylonkomplex war niedriger als für die anderen Kation-Montmorillonit-Nylonkomplexe.

**Résumé**—Des complexes montmorillonite-acide aminocaproïque (complexes monomères) ont été préparés par intercalation d'acide aminocaproïque-6 à diverses montmorillonites ( $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Co}^{2+}$ , et  $\text{Cu}^{2+}$ ) homoioniques. Les spectres infrarouges des complexes monomères ont indiqué que l'action entre les cations échangeables et l'acide aminocaproïque-6, croît dans l'ordre suivant; le complexe acide aminocaproïque-montmorillonite- $\text{Na}$ ,  $\text{Ca}$ ,  $\text{Mg}$  <  $\text{Co}$  <  $\text{Cu}$ . Les complexes montmorillonites-nylon (complexes polymères) ont été préparés par traitement thermique de complexes monomères, ce qui a été confirmé au moyen de la diffraction aux rayons-X et la spectroscopie infrarouge dont les résultats ont indiqué la condensation de l'acide aminocaproïque-6 dans l'espace interfeuillet. La dégradation thermique des complexes montmorillonite-nylon a été étudiée par thermogravimétrie. On a trouvé que la stabilité thermique des complexes polymères croît dans l'ordre suivant; les complexes nylon-montmorillonite- $\text{Cu}$  <  $\text{Co}$  <  $\text{Na}$  <  $\text{Mg}$  <  $\text{Ca}$ . Il a été suggéré que la différence de stabilité thermique dépendait de la longueur de la chaîne polymère qui pourrait être influencée par l'interaction entre les cations échangeables et l'acide aminocaproïque-6. L'énergie d'activation pour la dégradation thermique de chaque complexe nylon-montmorillonite a été obtenue et la valeur du complexe nylon-montmorillonite- $\text{Cu}$  était plus petite que celles des complexes nylon-montmorillonite à cations échangés.