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**MONTMORILLONITE FROM MILOWICE
INTERCALATED WITH HYDROXY-ALUMINIUM OLIGOCATIONS
AS VAPOUR AND GAS ADSORBENT**

A b s t r a c t. An interesting type of sorbent was obtained by introducing hydroxy-aluminium oligocations into the interlayer spaces of montmorillonite separated from the Milowice bentonite. Studies of argon, water and benzene sorption on the montmorillonite samples were carried out. From adsorption isotherms specific surface areas were determined, depending on the process mechanism and the sorbate used. The volume of micropores was calculated from the argon adsorption isotherm at low relative pressures. The volume of mesopores was also determined from the argon desorption isotherm. X-ray powder method was used to calculate interlayer spacings. The size of cross-linking oligocations was estimated from DTA, DTG and TG curves.

INTRODUCTION

Many scientific centres are engaged in research on a new type of adsorbents with the properties competing with those of zeolites (Brindley, Sempels 1977; Lahav *et al.* 1978; Vaughan *et al.* 1979; Brindley, Kao-Chun 1980; Fijał, Olkiewicz 1982; Occelli, Tindawa 1983; Pinnavaia 1983, and others). These new "molecular sieves" are montmorillonites with inorganic oligocations or organic-inorganic complexes introduced into their interlayer spaces. The properties of these sorbents depend on the manner of modification of the initial mineral, and on the heat treatment of the resulting product. In the paper presented here, hydroxy-aluminium oligocations were introduced into the interlayer spaces of montmorillonite separated from the Milowice bentonite. As a result of calcination (dehydration and dehydroxylation), the oligocations were transformed into oxide "supports". The most important features of these sorbents from the viewpoint of utilization of their sieve properties are their porosity and the size of pores.

Owing to their thermal stability and large specific surface area, intercalated montmorillonites can be used as molecular sives or catalysts of the reaction of large organic macromolecules (Diddams *et al.* 1984, Kikutchi *et al.* 1985).

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EXPERIMENTAL

The montmorillonite fractions of a particle diameter less than 2 μm were separated by sedimentation from the water suspension of bentonite from Milowice. Then the mineral was mixed with 1 n NaCl to obtain the initial material (Na-form of montmorillonite) for ion exchange with hydroxy-aluminium cations. The solution of hydroxy-aluminium oligocations was prepared by dropping NaOH into AlCl_3 solution and stirring the mixture to prevent local precipitation of $\text{Al}(\text{OH})_3$. The size of oligocations was controlled by dropping in different amounts of NaOH, whereby solutions with the OH/Al ratio of about 1, 2 and 2.5 were obtained (Brindley, Yamanaka 1979). The solutions prepared in this way were mixed directly with the water suspension of montmorillonite, whereupon the modification product was washed with distilled water until the reaction to chloride ions was negative. The samples were then dried on a water bath and heated at 473, 573, 673, 773 and 873 K for 10 hours. The samples were designated respectively as IMAl1, IMAl2, IMAl2.5. The numbers 1, 2 and 2.5 denote the OH/Al ratio in oligocation solution. The number of three figures after the sample symbol, e.g. IMAl2.5 673, denotes calcination temperature in degrees Kelvin.

Argon adsorption isotherms were obtained at 77.5 K using a sorption manostat. Prior to measurements, the samples were outgassed at 383 K to a vacuum of 10^{-5} Torr. Adsorption and desorption isotherms were determined for montmorillonite samples before and after calcination, and for Na-form of montmorillonite. Argon sorption was also measured at low relative pressures. Isotherms for water vapour and benzene sorption were obtained at 298 K using microburettes for liquids. Prior to this, the samples were outgassed at 383 K.

Thermal analysis was carried out on a DERIVATOGRAPH. The instrument settings were: weighed portion – 600 mg; sensitivity: DTA – 250 μV , TG – 200 mg, DTG – 250 μV ; heating rate – $10^\circ/\text{min}$. The samples were heated in ceramic crucibles.

X-ray powder patterns were recorded with a DRON-1.5 diffractometer, using filtered CuK_α radiation and flat samples with the texture parallel to the surface. The samples were dried at room temperature and rehydrated after calcination at 573, 673, 773, 833, 873, 923 and 973 K.

RESULTS

Argon sorption

Barrer's studies (1955, 1963) of the adsorptive properties of montmorillonites with alkyloamines introduced into their interlayer spaces showed that it was possible to make their structure rigid. Adsorptive capacity with respect to argon depends on the manner of preparation of samples.

Figure 1 presents isotherms obtained for argon sorption on untreated montmorillonite and on samples intercalated with hydroxy-aluminium oligocations, not subjected to calcination. The isotherms initially follow the shape of Langmuir isotherms, and sorption is the higher, the larger is the oligocation.

In the calcined sample IMAl1 argon sorption decreases. This decrease is caused by the small size of oligocations, the collapse of the structure, and the diminishing amount of micropores (Fig. 2A). The IMAl2.5 sample shows different behaviour, as argon sorption increases at 673 K. This may be due to the loss of water by hy-

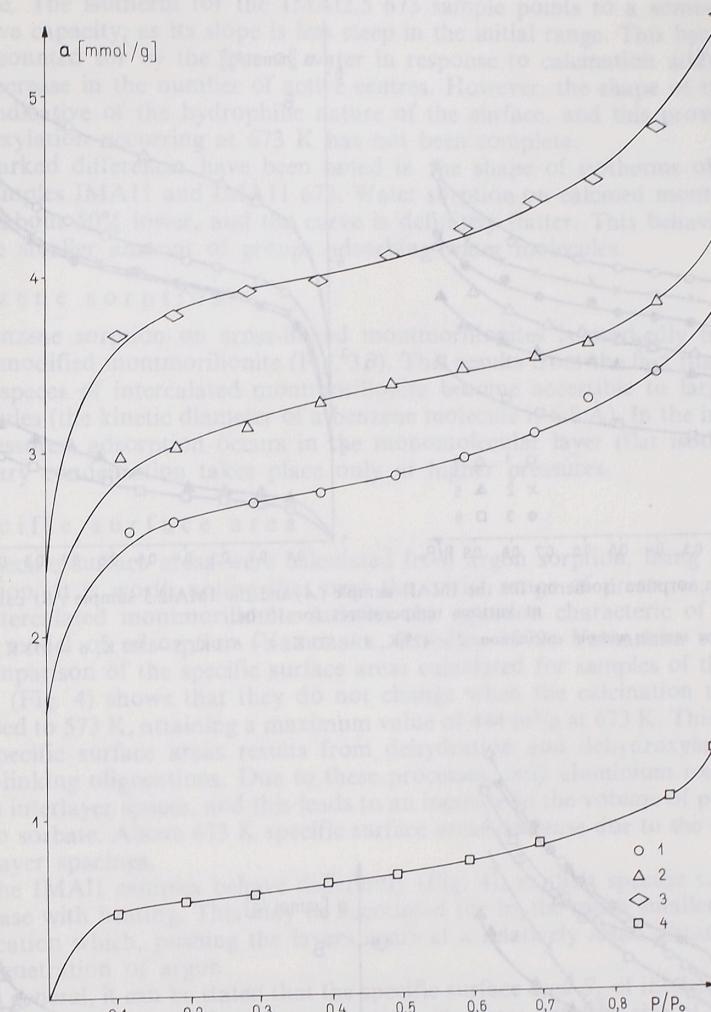


Fig. 1. Sorption isotherms of argon
1 – IMAl1, 2 – IMAl2, 3 – IMAl2.5, 4 – Na-montmorillonite

droxy-aluminium cations, yet this phenomenon is not attended by any significant decrease in the d_{001} spacing.

Water sorption

Isotherms determined for water sorption on Na-montmorillonite and on montmorillonites intercalated with hydroxy-aluminium oligocations are the BET curves of type II (Fig. 3A). The amount of water vapour absorbed by modified samples is markedly greater than that absorbed by sodium montmorillonite. Water sorption on modified montmorillonite is already high over the initial range, which

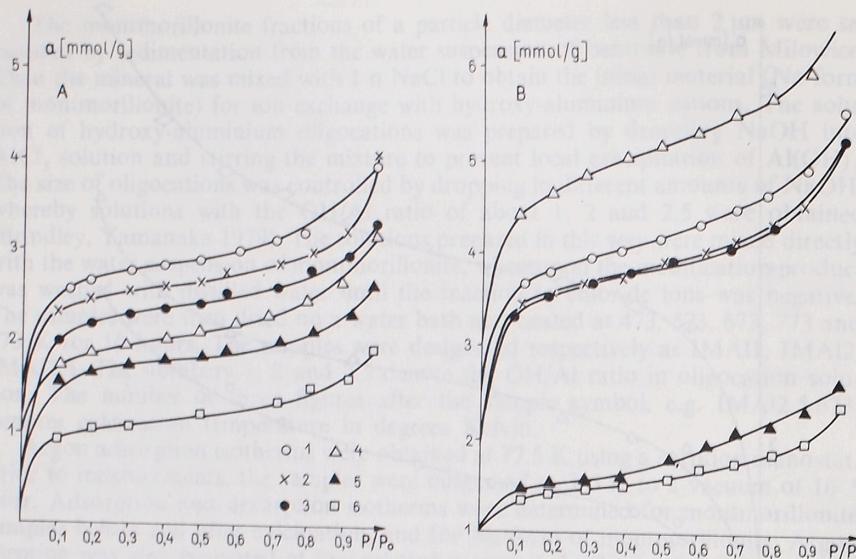


Fig. 2. Argon sorption isotherms for the IMA11 sample (A) and the IMA12.5 sample (B) calcinated at various temperatures for 10 hr.

1 - the sample without calcination, 2 - 473 K, 3 - 573 K, 4 - 673 K, 5 - 773 K, 6 - 873 K

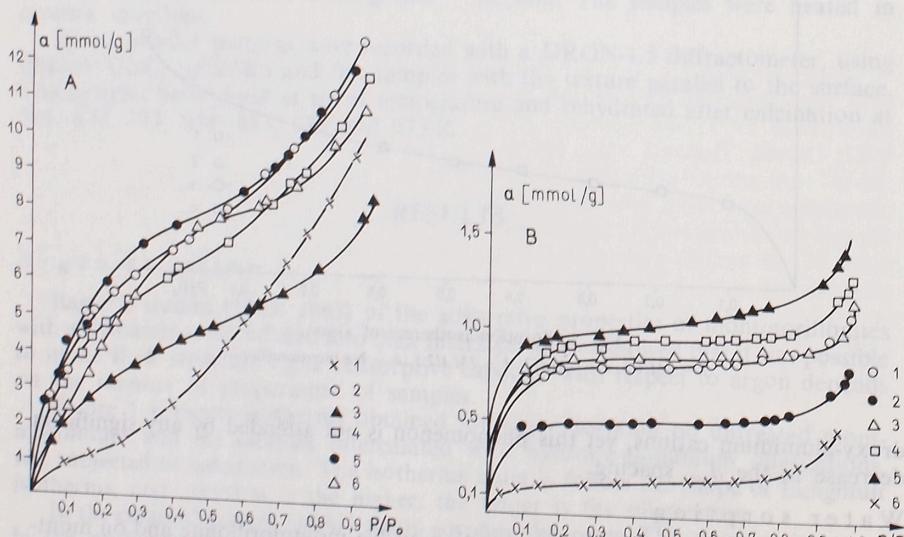


Fig. 3. A - Sorption isotherms of water vapors at 298 K
1 - Na-montmorillonite, 2 - IMA11, 3 - IMA11 673, 4 - IMA12, 5 - IMA12.5, 6 - IMA12.5 673

B - Sorption isotherms of benzene vapors at 298 K
1 - IMA11, 2 - IMA11 673, 3 - IMA12, 4 - IMA12.5, 5 - IMA12.5 673, 6 - Na-montmorillonite

indicates that the number of active centres increases compared with the unmodified sample. The isotherm for the IMA12.5 673 sample points to a somewhat lower sorptive capacity, as its slope is less steep in the initial range. This behaviour can be accounted for by the loss of water in response to calcination and, therefore, the decrease in the number of active centres. However, the shape of the curve is still indicative of the hydrophilic nature of the surface, and this proves that dehydroxylation occurring at 673 K has not been complete.

Marked differences have been noted in the shape of isotherms obtained for the samples IMA11 and IMA11 673. Water sorption on calcined montmorillonite is by about 50% lower, and the curve is definitely flatter. This behaviour is due to the smaller amount of groups adsorbing water molecules.

Benzene sorption

Benzene sorption on cross-linked montmorillonites is markedly higher than on unmodified montmorillonite (Fig. 3B). This results from the fact that the interlayer species of intercalated montmorillonite become accessible to large benzene molecules (the kinetic diameter of a benzene molecule is 6.8 Å). In the initial range of pressures, adsorption occurs in the monomolecular layer (flat isotherm), and capillary condensation takes place only at higher pressures.

Specific surface area

Specific surface areas were calculated from argon sorption, using Langmuir's equation. It is worth noting that over the initial range of pressures, adsorption on intercalated montmorillonite satisfies the equation characteristic of the monolayer model of adsorption (Yamanaka, Brindley 1979; Yamanaka *et al.* 1984). A comparison of the specific surface areas calculated for samples of the IMA12.5 series (Fig. 4) shows that they do not change when the calcination temperature is raised to 573 K, attaining a maximum value of 444 m²/g at 673 K. This high value for specific surface areas results from dehydration and dehydroxylation of the cross-linking oligocations. Due to these processes, only aluminium oxide remains in the interlayer spaces, and this leads to an increase in the volume of pores accessible to sorbate. Above 673 K specific surface areas decrease due to the diminishing interlayer spacings.

The IMA11 samples behave differently (Fig. 4), as their specific surface areas decrease with heating. This may be accounted for by the much smaller size of the oligocation which, pushing the layers apart at a relatively small distance, hinders the penetration of argon.

In general, it can be stated that the specific surface area S_L of intercalated montmorillonites, calculated from argon sorption, increases with the size of oligocation.

Specific surface areas were also calculated from water and benzene sorption isotherms, using Langmuir's or BET equation. The choice of the equation was determined by its ability to satisfy the principle of rectilinearity of the isotherm over the initial range of relative pressures.

X-ray analysis

The interplanar spacings determined from X-ray diffraction data are presented in Figure 4. It is evident from the figure that the oligocations vary in size, depending on the OH/Al ratio in the solution. The d_{001} value decreases after calcination. As appears from the X-ray diffraction patterns, samples calcined after treatment with ethyl glycol do not show swelling properties at temperatures higher than 573 K.

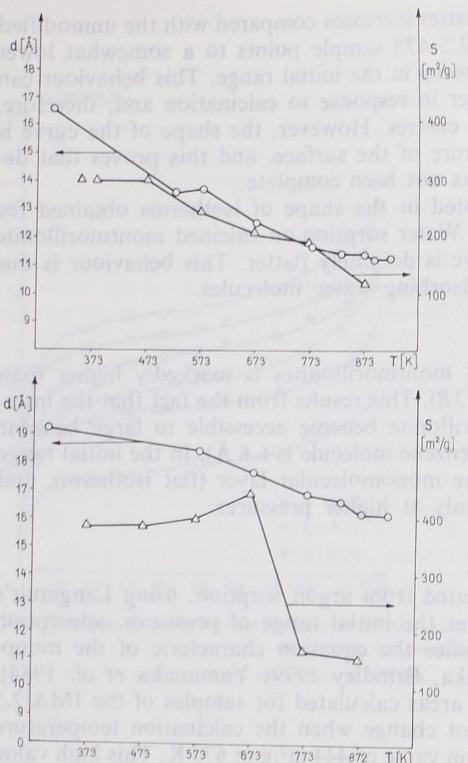


Fig. 4. Relation between interlayer spaces d_{001} and specific surface area (S_L) and the calcination temperature

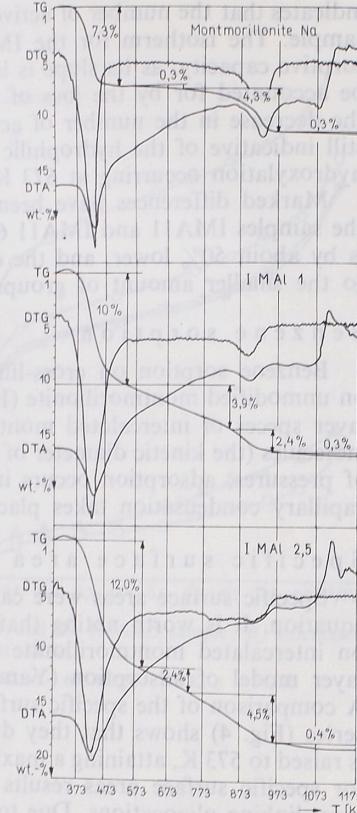


Fig. 5. TG, DTG and DTA plots of the examined samples

Thermal analysis

From the TG, DTG and DTA curves (Fig. 5) the size of oligocations was estimated (different percentage of water loss on TG curves), and the changes occurring in the samples during calcination were determined (Fig. 5). It has been found that the amount of OH groups in the mineral increases with the increase in the OH/Al ratio in the solution of intercalating oligocations.

DISCUSSION

Analysing the dependence of d_{001} on temperature, the process of structural destruction of intercalated montmorillonite was followed (Fig. 4).

In the IMA1 sample, dehydration causes a decrease in the d_{001} spacing from about 16.8 Å at 293 K to 13.6 Å at 523 K. As a result of dehydroxylation, beginning at 673 K, the d_{001} value decreases down to 11 Å at 873 K. In the IMA1.2.5 sample,

the d_{001} value changes respectively from 19.2 Å at 293 K to 18.4 Å at 573 K and to 16 Å at 873 K. It appears from these observations that the thermal stability of the oligocations of both types is the same whereas the height of "pillars" forming after calcination is different.

Figures 6 and 7 present isotherms determined for water, benzene and argon sorption before and after calcination. The amount of adsorbed substance is given in mm^3 of liquid sorbate per 1 g of adsorbent. In uncalcined samples, a fairly large number of water molecules penetrate into the pore structure of the adsorbent due to the specific forces interacting between the water molecules and the polar centres of the adsorbent. Calcination reduces the number of these centres, decreasing in consequence the sorption of water molecules.

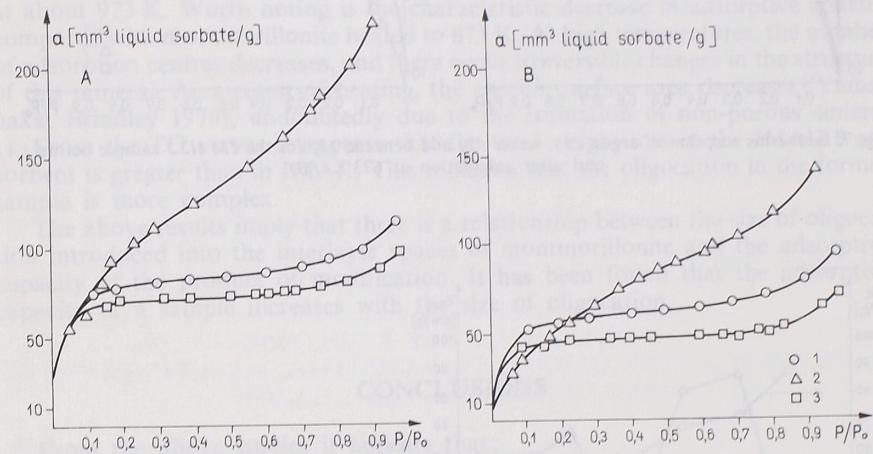


Fig. 6. Isotherms sorption of argon (1), water (2) and benzene (3) for the IMA1 sample before (A) and after calcination at 673 K (B)

Benzene sorption on both calcined and uncalcined samples is high, in the case of IMA1 being not much lower than argon sorption. This testifies to the well-developed mesoporosity of this adsorbent, which promotes adsorption of inert gases.

Argon adsorption isotherms were also determined at very low relative pressures (p/p_0 from 0.001 to 0.1 Torr). From the shape of these isotherms the volume of micropores was calculated, using the equation of Dubinin-Raduszkiewicz. The volume of mesopores was calculated from the desorption part of the argon sorption isotherm. Figures 8A and 8B show the relationship between V_{mic} and V_{mes} and the calcination temperature. For the IMA1.2.5 sample (Fig. 8B) a parallel dependence of the volume of pores of these two kinds and calcination temperature was noted. This dependence also correlates with the changes in the specific surface area S_L as a function of calcination temperature (Fig. 4). The same plots are slightly more complicated for IMA1 (Fig. 8A). Over the initial range of temperatures (up to 573 K), the amount of micropores increases, and above 573 K the content of both kinds of pores decreases. At 773 K the amount of mesopores increases again, but during further calcination (up to 873 K), the contents of both meso- and micro-pores decreases.

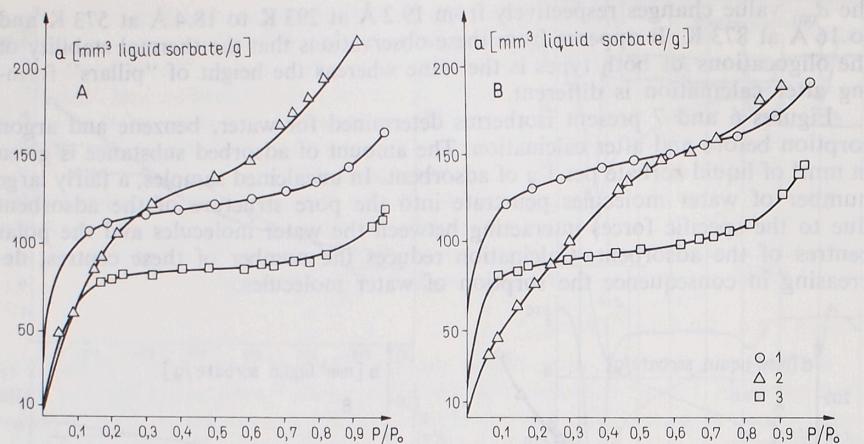


Fig. 7. Isotherms sorption of argon (1), water (2) and benzene (3) for the IMA12.5 sample before (A) and after calcination at 673 K (B)

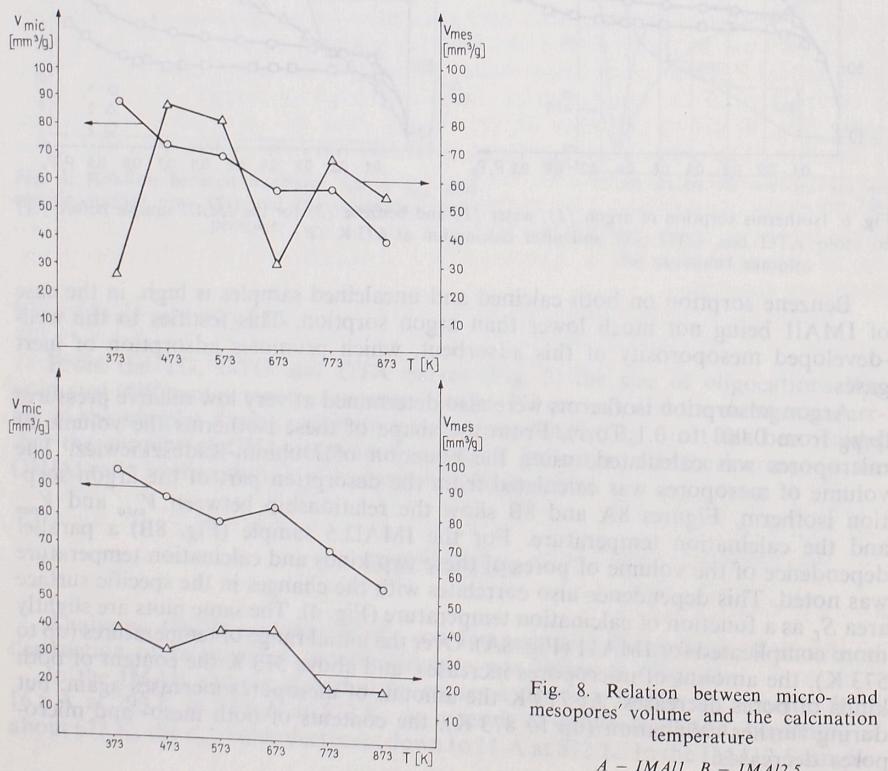


Fig. 8. Relation between micro- and mesopores' volume and the calcination temperature
A - IMA1, B - IMA12.5

The analysis of TG, DTG and DTA curves (Fig. 5) indicates that dehydration and dehydroxylation of the oligocations introduced into the interlayer spaces occur over the temperature range from 293 to about 800 K. The loss of interlayer water is attended by a decrease in interplanar spacings, which has also been confirmed by X-ray analysis. The higher adsorptive capacity of montmorillonite (with respect to argon) after the interlayer water has been given off is presumably due to the increased volume of pores into which diffuse the sorbate molecules.

The loss of water bound to hydroxy-aluminium cations and dehydroxylation of these cations take place in a temperature range between 573 and 773 K (there is no such effect in Na-form of montmorillonite). After they have lost water, the aluminium oligocations change into alumina supporting the layers. The loss of water bound in the structure of montmorillonite begins at about 773 K and ends at about 973 K. Worth noting is the characteristic decrease in adsorptive activity compared with montmorillonite heated to 673 K. At high temperatures, the number of adsorption centres decreases, and there occur irreversible changes in the structure of this mineral. As a result of heating, the specific surface area decreases (Yamanaka, Brindley 1979), undoubtedly due to the formation of non-porous sinters.

From the TG curves it appears that the total weight loss in the IMA12.5 adsorbent is greater than in IMA1. This indicates that the oligocation in the former sample is more complex.

The above results imply that there is a relationship between the size of oligocation introduced into the interlayer spaces of montmorillonite and the adsorptive capacity of the product of modification. It has been found that the adsorptive capacity of a sample increases with the size of oligocation.

CONCLUSIONS

From the above studies it appears that:

- the structure of montmorillonite becomes micro- and mesoporous after modification,
- intercalation causes the specific surface area (determined from argon sorption) to develop to 450 m²/g,
- calcination reduces the number of active centres for sorbed water molecules,
- calcined montmorillonite shows structural stability even at elevated temperatures (up to 773 K),
- the interplanar spacing d_{001} of intercalated montmorillonites have a close relationship with the size of hydroxy-aluminium oligocations.

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MONTMORILLONIT Z MILOWIC INTERKALOWANY OLIGOKATIONAMI HYDROKSYGLINOWYMI JAKO ADSORBENT PAR I GAZÓW

Streszczenie

Montmorillonit z Miłowic interkalowano oligokationami hydroksyglinowymi. Stosunek OH/Al w roztworze interkalujących oligokationów wynosił około 1, 2, 2,5. Otrzymane próbki poddano kalcynacji w temperaturach 473, 573, 673, 773 i 873 K. Na tak przygotowanych sorbentach wykonano badania sorpcyjne, rentgenowskie i termiczne. Wyznaczono izotermy sorpcji argonu, wody i benzenu. Z początkowej części izotermy adsorpcji argonu wyliczono powierzchnię właściwą, która osiągała 450 m²/g i była tym większa im większy był interkalujący oligokation. Z sorpcji argonu przy niskich ciśnieniach względnych wyznaczono objętości mikroporów korzystając z równania Dubinin-Raduszkiewicza.

Do wyliczenia objętości mezoporów posłużyła desorpcyjna gałąź izotermy sorpcji argonu. Krzywe TG, DTG, DTA pozwoliły na szacunkowe określenie wielkości oligokationu. Za pomocą analizy rentgenograficznej wyznaczono rozmiary odległości międzypakietowych d_{001} . Okazało się, że otrzymane sorbenty są stabilnie termiczne do 773 K, a w wyniku kalcynacji tracą własności pęcznięce do około 16 Å w 673 K. Wielkość d_{001} zależy od stosunku OH/Al w roztworze „pórki”. Szeroki przedział temperaturowy, w którym następuje dehydroksylacja w hydroksylationie. Wbudowanie oligokationu w strukturę montmorillonitu

wpływają na zwiększenie ilości centrów aktywnych dla wody. Otrzymane sorbenty mają strukturę mikroporową, która jest także dostępna dla dużych cząsteczek benzenu, zapełniających mezopory.

OBJAŚNIENIA FIGUR

Rys. 1. Izotermy sorpcji argonu

1 – $IMA1I$, 2 – $IMA12$, 3 – $IMA12.5$, 4 – forma sodowa montmorillonitu

Rys. 2. Izotermy sorpcji argonu $IMA1I$ (A) i $IMA12.5$ (B) wygrzewanych w różnych temperaturach przez 10 h:

1 – próbka bez wygrzewania, 2 – 473 K, 3 – 573 K, 4 – 673 K, 5 – 773 K, 6 – 873 K

Rys. 3. A – izotermy sorpcji par wody w temperaturze 298 K

1 – forma sodowa montmorillonitu, 2 – $IMA1I$, 3 – $IMA1I$ 673, 4 – $IMA12$, 5 – $IMA12.5$, 6 – $IMA12.5$ 673

B – izotermy sorpcji par benzenu w temperaturze 298 K:

1 – $IMA1I$, 2 – $IMA1I$ 673, 3 – $IMA12$, 4 – $IMA12.5$, 5 – $IMA12.5$ 673, 6 – forma sodowa montmorillonitu

Rys. 4. Zależność odległości międzypakietowych d_{001} i powierzchni S_L (argon) od temperatury kalcynacji dla próbek $IMA1I$ i $IMA12.5$

Rys. 5. Krzywe TG, DTG i DTA badanych próbek

Rys. 6. Porównawcze zestawienie izoterm adsorpcji argonu (1), wody (2) i benzenu (3) dla próbki $IMA1I$ przed (A) i po kalcynacji w 673 K (B)

Rys. 7. Porównawcze zestawienie izoterm adsorpcji argonu (1), wody (2) i benzenu (3) dla próbki $IMA12.5$ przed (A) i po kalcynacji w 673 K (B)

Rys. 8. Zależność objętości mikro- i mezoporów od temperatury kalcynacji dla:

A – $IMA1I$, B – $IMA12.5$

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МОНТМОРИЛЛОНИТ ИЗ МИЛЁВИЦ С ВНЕДРЕННЫМИ ГИДРОКСИАЛЮМИНИЕВЫМИ ОЛИГОКАТИОНАМИ КАК АДСОРБЕНТ ПАРОВ И ГАЗОВ

Резюме

В монтмориллонит из Милёвиц внедрялись гидроксилалюминиевые олигокатионы. Соотношение OH/Al в растворе внедряющихся олигокатионов составляло около 1, 2, 2,5. Полученные образцы подвергались обжигу в температурах 473, 573, 673, 773 и 873 К. На таким образом полученных сорбентах проведены были сорбционные, рентгеновские и термические исследования. Определены изотермы сорбции аргона, воды и бензола. Из начальной изотермы адсорбции аргона вычислена удельная поверхность, которая достигала 450 м²/г; ее значение тем больше чем больше внедряющийся олигокатион. Из сорбции аргона при низких относительных давлениях, определено объем микропор. Для расчета объема мезопор использовалась десорбционная ветвь изотермы сорбции аргона. Кривые ТГ, ДТГ, ДТА позволили оценить размеры олигокатиона. С помощью рентгенографического анализа определено раз-

Для расчета объема мезопор использовалась десорбционная ветвь изотермы сорбции аргона. Кривые ТГ, ДТГ, ДТА позволили оценить размеры олигокатиона. С помощью рентгенографического анализа определено раз-

меры межпакетных расстояний d_{001} . Оказалось, что полученные сорбенты термически устойчивы до 773 К, а в итоге прокаливания теряют свойства разбухания. Межпакетные расстояния достигают 19,2 Å, а после обжига в температуре 673 К сокращаются до 16 Å. Величина d_{001} зависит от соотношения OH/Al в растворе внедряющихся олигокатионов, а тем самым от разрастания „подпоры“. Широкий температурный диапазон, в котором происходит дегидроксилация гидроксилалюминиевых олигокатионов свидетельствует о разной силе связи групп OH в гидроксилкатионе. Внедрение олигокатиона в структуру монтмориллонита влияет на увеличение количества активных центров для воды. Полученные сорбенты обладают микропоровой структурой, которая тоже доступна для крупных молекул бензола, заполняющих мезопоры.

ОБЪЯСНЕНИЯ К ФИГУРАМ

Фиг. 1. Изотермы сорбции аргона

1 – IMAI1, 2 – IMAI2, 3 – IMAI2,5, 4 – натриевая разновидность монтмориллонита

Фиг. 2. Изотермы сорбции аргона IMAI1 (A) и IMAI2,5 (B), прогреваемых в разных температурах в течении 10-и часов

1 – образец без обжига, 2 – 473 К, 3 – 573 К, 4 – 673 К, 5 – 773 К, 6 – 873 К

Фиг. 3. А – изотермы сорбции паров воды в температуре 298 К

1 – натриевая разновидность монтмориллонита, 2 – IMAI1, 3 – IMAI1 673, 4 – IMAI2, 5 – IMAI2,5, 6 – IMAI2,5 673

В – изотермы сорбции паров бензола в температуре 298 К

1 – IMAI1, 2 – IMAI1 673, 3 – IMAI2, 4 – IMAI2,5, 5 – IMAI2,5 673, 6 – натриевая разновидность монтмориллонита

Фиг. 4. Зависимость межпакетных расстояний d_{001} и поверхности S_L (аргон) от температуры прокаливания образцов IMAI1 и IMAI2,5

Фиг. 5. Кривые ТГ, ДТГ и ДТА изучаемых образцов

Фиг. 6. Сопоставление изотерм адсорбции аргона (1), воды (2) и бензола (3) для образца IMAI1 до (A) и после (B) прокаливания в 673 К

Фиг. 7. Сопоставление изотерм адсорбции аргона (1), воды (2) и бензола (3) для образца IMAI2,5 до (A) и после (B) прокаливания в 673 К

Фиг. 8. Зависимость объема микро- и мезопор от температуры прокаливания для IMAI1 (A) и IMAI2,5 (B)