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ELECTRON MICROSCOPIC STUDY OF TUBULAR HALLOYSITE FORMS

(24 figures, 1 table)

Abstract: Application of electron microscope in the study of tubular halloysite forms in Neogene basins of the West Carpathians has made possible to create a picture of their morphological variability and indicated the existence of some relation between the shape of particles and structure order. At the same time it has shown that the value shape parameter "λ" may be a manifestation of conditions, under which they originate.

Резюме: Применение электронного микроскопа для изучения трубчатых форм галлуазита из пород неогеновых котловин Западных Карпат дало возможность получить картину их морфологической вариабильности и показало на присутствие некоторой взаимосвязи между формой частиц и упорядоченностью структуры. Одновременно было определено то, что величина параметра форм может являться отражением условий их образования.

Introduction

Shape variability of crystals of kaolinite group minerals is nearly as large as variability of shapes in the whole clay mineral group.

The fundamental shapes are sphere, cylinder and pseudohexagonal (hexagonal) plate. The extent of imperfection and deformation of these shapes is, however, so large that onion-like scales from spheres or ultrathin flakes from plates appear as rolled scrolls to tubes. At another place the cylinders are splitting and get equalized and so give the impression of apparent appurtenance to substances with onedimensional lengthening of pseudohexagonal plates, and the same plates piled up above one antoher into books or bunches may form equidimensional triclinic crystals, bordered by prisms and pinacoids. If we also include clusters of particles and irregular fragments formed due to mechanical impact with laboratory treatment of the sample, then variability of shapes is almost complete.

In up to present investigation M. Harman (1964), I. Kraus et al. (1967), I. Kraus et al. (1972) have found out that in Neogene volcanogenic-sedimentary complexes of the West Carpathians very different types of tubular forms of halloysite with variously ordered structure occur. Having considered the facts mentioned, we started to devote more intensely to methods, which might give a more objective view of morphological development of tubular halloysite crystals from various localities, identified mineralogically.

For the study under electron microscope we prepared preparations by the method of suspensions. The observed samples we scattered in little concentration by ultrasound in redistilled water and carried on collodion membrane.

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We studied morphology of the particles under electron microscope Tesla BS 242 E.

Geological position and mineral composition of studied samples Clays with halloysite come from Neogene basins of the West Carpathians (Fig. 1) where they usually originated with weathering of products of andesite and rhyolite volcanism. Here belong the inner basins in the region of Central Slovakian neovolcanics, especially the Slatinská, Lučenecká and Bátovská kotlina basins. The second particular region in the East Slovakian Neogene basin. For comparison we also studied halloysite from the Karlové Vary area in the Bohemian massif and from the locality Douaria (Tunisia).

In the Slatinská kotlina basin we have established the presence of halloysite in the complex of altered volcanoclastics of the second andesite phase in the sense of M. Kuthan (1963), assigned to the upper Badenian, occuring south and southeast of the village Hrochoť.

Concerned is dark-brown clay with unpreserved texture of mother rock (sample No. 1, Sk-18/82,5 m), which most probably represents a redeposited product of alteration of andesite volcanoclastics. With X-ray analysis exclusively the presence of halloysite was confirmed. In the original sample the hydrated form of halloysite (reflex 10,1 A°) predominates over the form of partially dehydrated halloysite (diffusion reflex within the range 7,3—7,8 A°). Halloysite particles display extremely fine-dispersed development, are imperfectly delimited and partly unfolded (Fig. 2). They usually attain length of several tenths of micrometers only.

A morphologically different halloysite type is present in the Slatinská kotlina basin in intensively decomposed andesite volcanoclastics where probably an

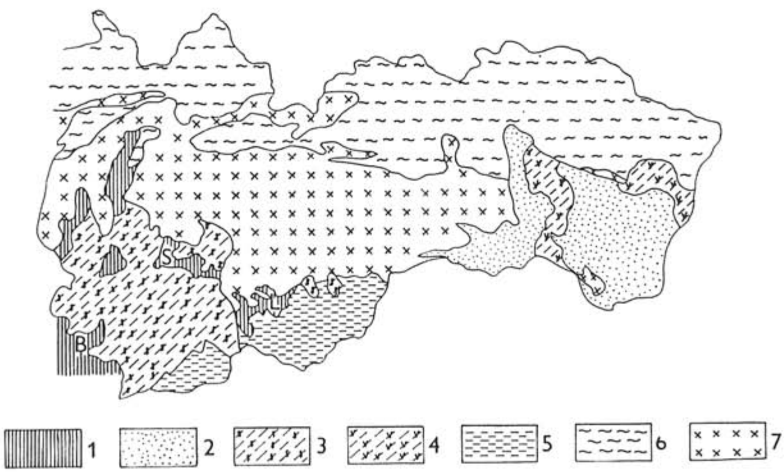


Fig. 1. Sketch-map of the geological structure of Neogene Basins in the West Carpathians. 1 — Inner basins, S — Slatinská kotlina, L — Lučenecká kotlina basin, in fresh-water development of the so called Poltár formation, B — Bátovská kotlina basin. 2 — East Slovakian Basin. 3 — Central Slovakian Neovolcanics. 4 — East Slovakian Neovolcanics. 5 — Ipeľská and Lučenecká kotlina basins in brackish and marine development. 6 — Outer and inner flysch belt. 7 — Central West Carpathians.

Fig. 2. Electron micrograph of halloysite, Slatinská kotlina basin, borehole SK-18/ 82,5 m, magnif. 18 000×.

Fig. 3. Electron micrograph of halloysite and montmorillonite, Slatinská kotlina basin, borehole SK-25/27,2 m, magnif. 18 000×.

alteration in situ is concerned (sample No. 2, Sk-25, 27,2 m). Tre X-ray analysis verified the presence of dehydrated halloysite and montmorillonite. Halloysite is characterized by perfect delimitation of tubular particles (Fig. 3).

In the Lučenecká kotlina basin at the western border in the underlier of the Poltár formation of Pliocene age decomposed pyroxenic andesites occur, belonging to the first andesite phase in the sense of M. Kuthan (1963). They belong to the Lower Badenian and according to I. Kraus and V. Hano (1976) their alteration was taking place at the boundary of the Upper Sarmation and Lower Pannonian (sample No. 3, Halič-THV 18). By means of X-ray analysis and electron microscope was verified the presence of halloysite and kaolinite with disordered structure. Halloysite is of shape of narrow and long tubes, which are disrupted and often shifted above one another. Well developed, undisturbed particles have a preserved circular crossection (Fig. 4).

In the Lučenecká kotlina basin, mainly in clays of the Poltár formation, halloysite is often present, especially in the south-western part where the influence of the volcanogenic source area is evident. A typical representative is the sample No. 4, Halič-Kopáň, composed of perfectly delimited, short tubes, approximately of equal length (Fig. 5).

Particular forms of halloysite occurrence in the sequence of the Poltár formation, near Kalinovo-Hrabovo, in form of so called armoured clay galls, were

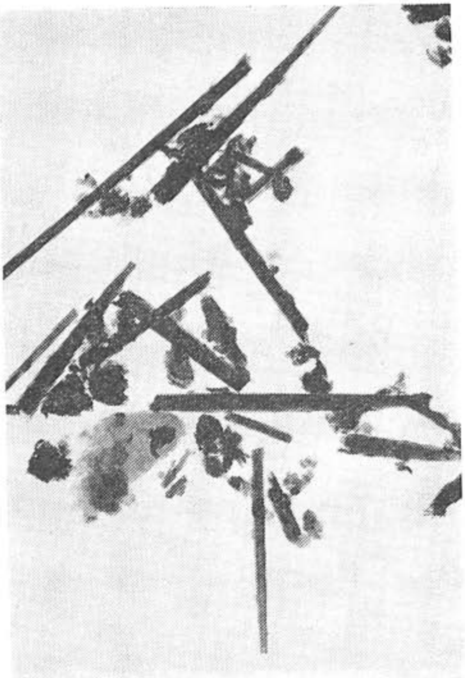


Fig. 4. Electron micrograph of halloysite with kaolinite, Lučenecká kotlina basin, Halič, borehole THV-18, magnif. 18 000×.

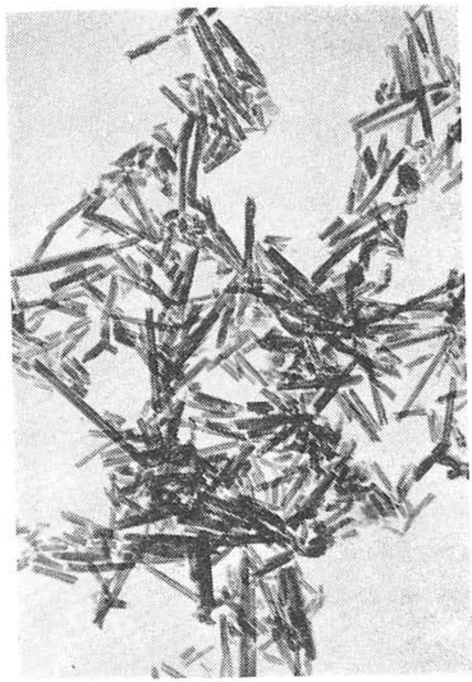


Fig. 5. Electron micrograph of halloysite, Lučenecká kotlina basin, Halič-Kopáň, magnif. 18 000×.

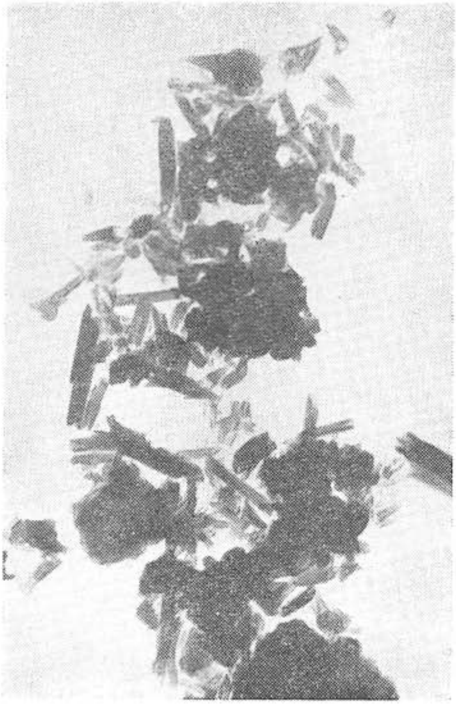
described in detail by I. Kraus et al. (1966). The hydrated form of halloysite is represented by the sample No. 5, halloysite $4H_2O$. It is found in form of spherical shapes in the lower part of the gravelous layer. In electron microscope halloysite prevailingly display tubular development (Fig. 6).

With erosion of gravels releasing and displacement of galls from the primary place of occurrence take place at a distance of several tens of metres. In this process partial dehydration of halloysite occurs. We did not succeed in distinguishing the dehydrated halloysite from the hydrated form on the basis of morphological properties of tubules under electron microscope (samples No. 6,7 Fig. 7).

T. F. Bates and J. J. Comer (1959) state that during the dehydration of halloysite the tubules are splitting at the borders, on the whole they get flattened and partly unfold into rolls of concial form. All these marks we may trace in our case in the hydrated but also partially dehydrated form of halloysite.

The clay cement of gravels, in which are found armoured galls of halloysite, consists of dehydrated halloysite with admixture of kaolinite (sample No. 8). The tubes of halloysite are essentially wider in comparison with samples No. 5, 6, 7.

In the Bátovská kotlina basin, similarly as in the Slatinská kotlina basin clays with halloysite originate with weathering of volcanoclastics of the second ande-



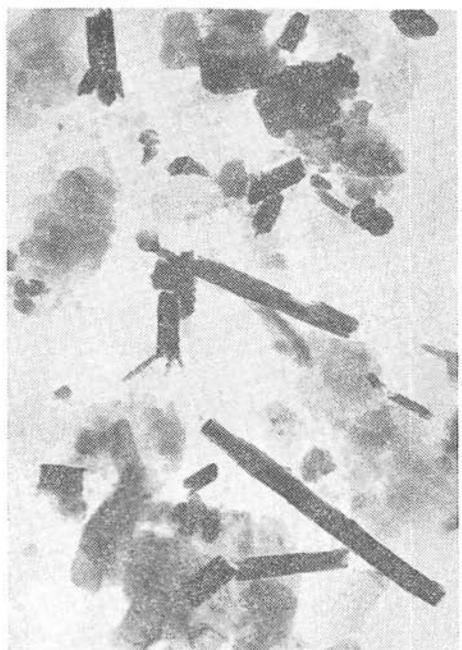


Fig. 6. Electron micrograph of halloysite ($4H_2O$), Lučenecká kotlina basin, Kalinovo-Hrabovo, magnif. 50 000×.

Fig. 7. Electron micrograph of halloysite ($2H_2O$), fraction 1–2 micrometers, Lučenecká kotlina basin, Kalinovo-Hrabovo, magnif. 20 000×.

Fig. 8. Electron micrograph of halloysite and kaolinite, clayey cement, Lučenecká basin, Kalinovo-Hrabovo, magnif. 20 000×.

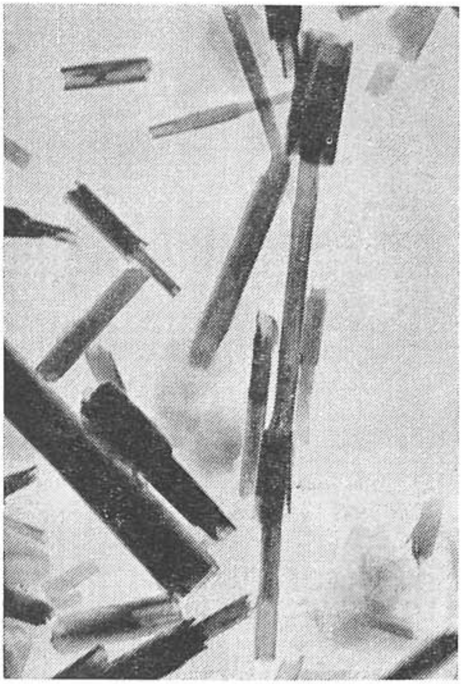


Fig. 9. Electron micrograph of halloysite, Bátovská kotlina basin, Tekovská Nová Ves, magnif. 40 000×.

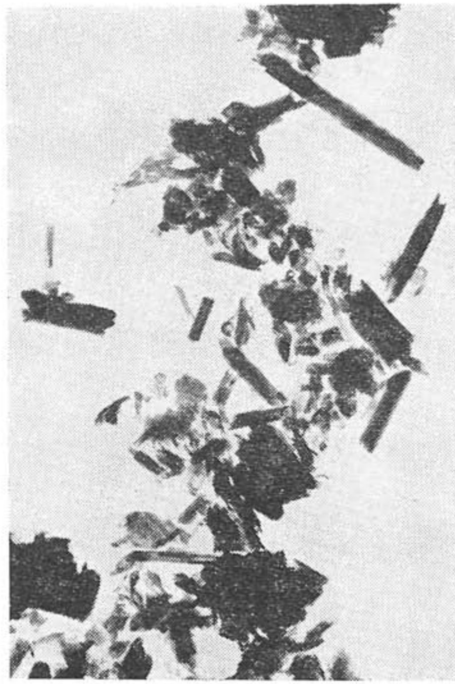
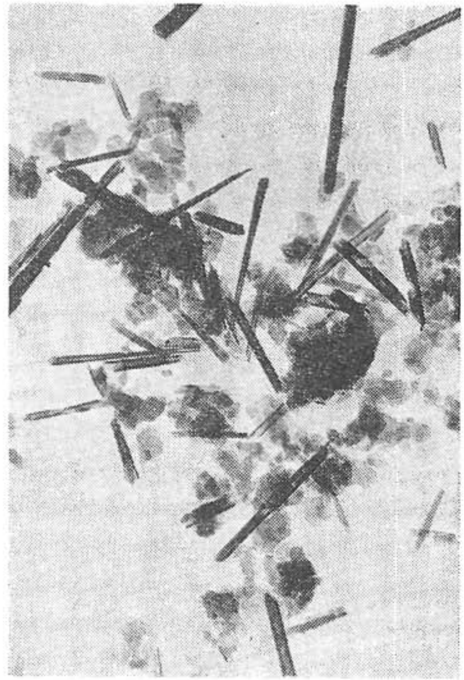


Fig. 10. Electron micrograph of halloysite, East Slovakian Neogene basin, Michalovce, Biela Hora, magnif. 18 000×, suspension.

site phase in the sense of M. Kuthan (1963). The studied clay from the area of Tekovská Nová Ves (sample No. 9) is prevailingly formed by halloysite, less by kaolinite. The halloysite particles are of the shape of narrow long tubules, often shifted above one another. In well developed undisturbed individuals we see preserved circular cross sections (Fig. 9). Pseudohexagonal kaolinite crystals gradually pass into irregularly delimited scales. Noteworthy is that in the sample studied halloysite displays a morphology of almost ideal tubes on the one hand and of tubes with apparent rolling up of basal planes on the other hand.

In the East Slovakian Neogene basin, at the known locality Michalovce-Biela Hora, an accumulation of halloysite of economic importance, seldom in Europe, is found. It formed by alteration of rhyolites and their volcanoclastics of Sarmatian age. The partly preserved texture of mother rocks in the lower part of the halloysite layer points to an origin in situ. In up to present study views of a hypergene but also hydrothermal origin were expressed. Basides other authors, it was defined mineralogically by A. Kochanovská (1955), J. Konta (1957) and E. G. Kukovský (1966). Determined by X-ray analysis was dehydrated halloysite with basal reflex 7,2 A°, then cristoballite and considerable admixture of kaolinite with disordered structure (fireclay).



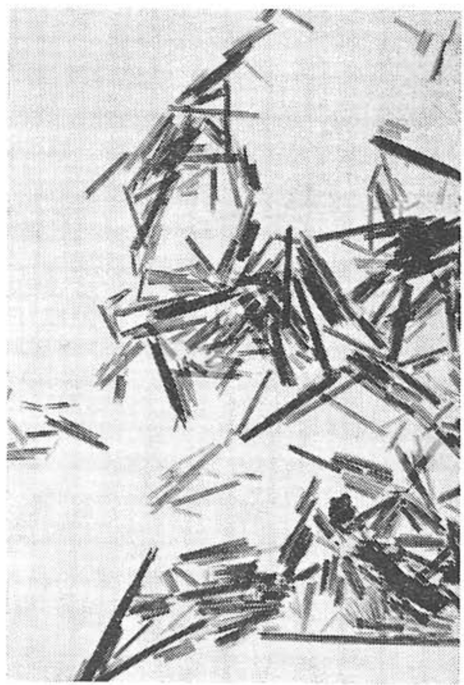


Fig. 11. Electron micrograph of halloysite, East Slovakian Neogene basin, Michalovce, Biela Hora, magnif. 12 000×, extraction replica.

Fig. 12. Electron micrograph of halloysite and kaolinite, Karlove Vary area – Jimlikov, magnif. 12 000×.

Fig. 13. Electron micrograph of halloysite, Douaria-Tunisia, magnif. 20 000×.

For observation under the electron microscope we prepared halloysite from Michalovce-Biela Hora by suspension method (Fig. 10) and by the methods of extraction replica (Fig. 11). On preparations made by suspension method we may observe tubular halloysite crystals of relatively great width as well as finely dispersed tabular fireclay kaolinite, together with fragments of irregular clay mineral grains.

On the extraction replica of fracture plane we may trace halloysite crystals of similar dimensions in length, what signifies that desintegration of the sample with preparation of the suspension was of no influence on the length of tubules. The average dimension in width is, however, lesser. This supports the view that with preparation of suspension the contact of sample with water causes a change in the width of tubules, propably by their partial unfolding.

From several known kaolin deposits in the Karlove Vary area (the western part of the Bohemian massif), which originated by weathering of Karlove Vary granite in situ, is at the locality Jimlikov besides kaolinite represented a considerable amount of halloysite (sample No. 11. Fig. 12).

Halloysite from the Douaria deposit in Tunisia was described by J. Ilavský (1969). The studied clay forms irregular lenticles of thickness 0,2—1,0 m in dacite volcanoclastics of Miocene age, probably originated in situ. It is characterized by massive structure, rarely very finely stratified. The study under electron microscope has confirmed that particles of halloysite are of the shape of narrow, medium-long tubes. An evidence of the tubular shape darker, impenetrable for electrons rims of tubes and a lighter, more translucent part in their middle, parallel with the longer side, together with the rounded off tubes ends of halloysite (Fig. 13).

Study of the particles size

The study under electron microscope makes us possible to measure the size of individual crystals (in our case halloysite besides other minerals). This method offers further the possibility of direct measurement of several dimensions of particles (for example, the width and length of tube) and so makes possible take into consideration the shape in relation to the measurement of size.

The basis of up to present evalution of electron micrographs was rather qualitative judgement of the record, often influenced by the subject of observation. For this reason we were focused on objectivization of quantitative properties, by the method of statistical treatment of the complex of measured values. As electron micrographs with various magnification were available to us, we modified all the dimensions to uniform 18 000 times magnification. In order to verify the influence of subjective factors in the measurement, mainly of tube widths, we performed controlling measurements of particles from electron micrographs with 18 000 times and 40 000 times magnification (Fig. 9, 20). Final comparison of the results has shown agreement of the values measured.

Characteristic dimensions of tubules, length "L" and projection of the tube diameter, appearing as width "B", we recorded in tables for magnification 18 000 times. The material obtained this way we ordered further and on the basis of it we constructed the order of frequency division. In each sample we evaluated about 400 particles. This basis we then ranged into intervals and represented graphically by histogram and frequency distribution polygon (see Figs 14 to 24).

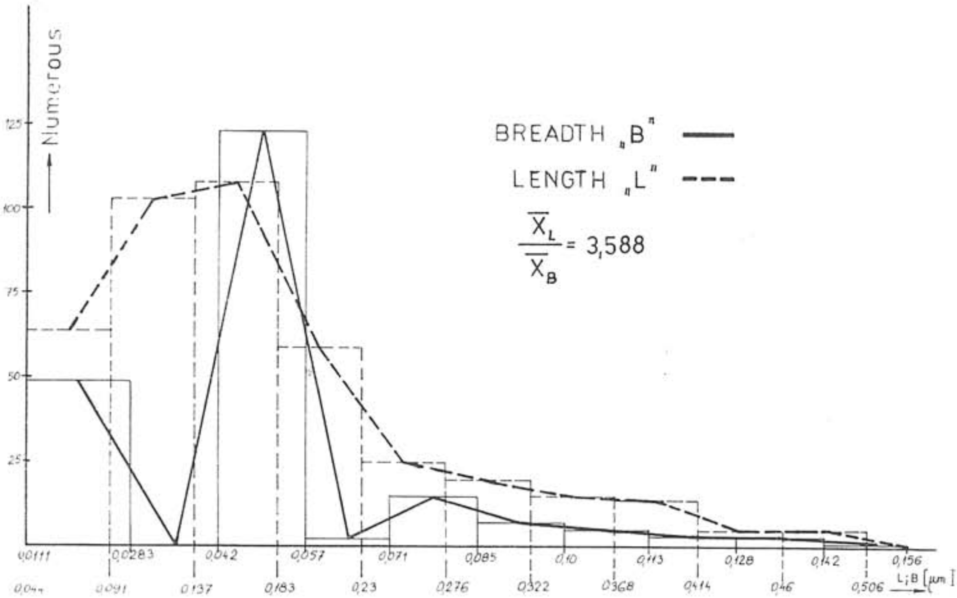


Fig. 14. Frequency distribution of particle dimensions, sample Slatinská kotlina basin, SK-18/82,5 m.

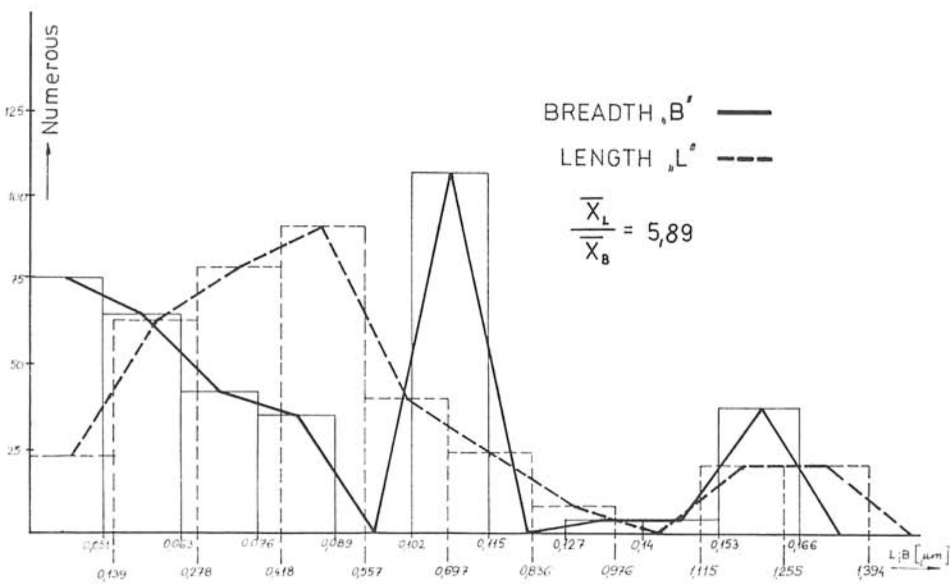


Fig. 15. Frequency distribution of particle dimensions, sample Slatinská kotlina basin, SK-25/27,2 m.

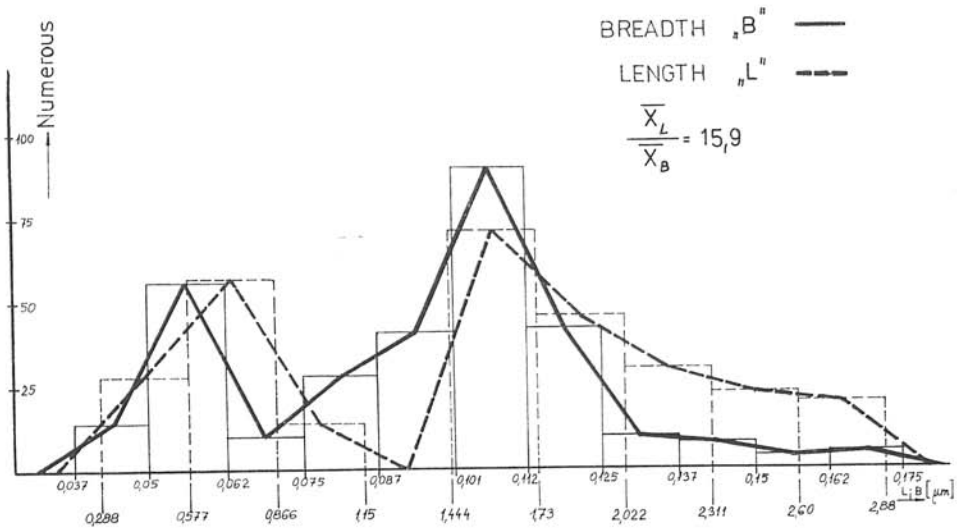


Fig. 16. Frequency distribution of particle dimensions, sample Lučenecká kotlina basin, Halič THV-18.

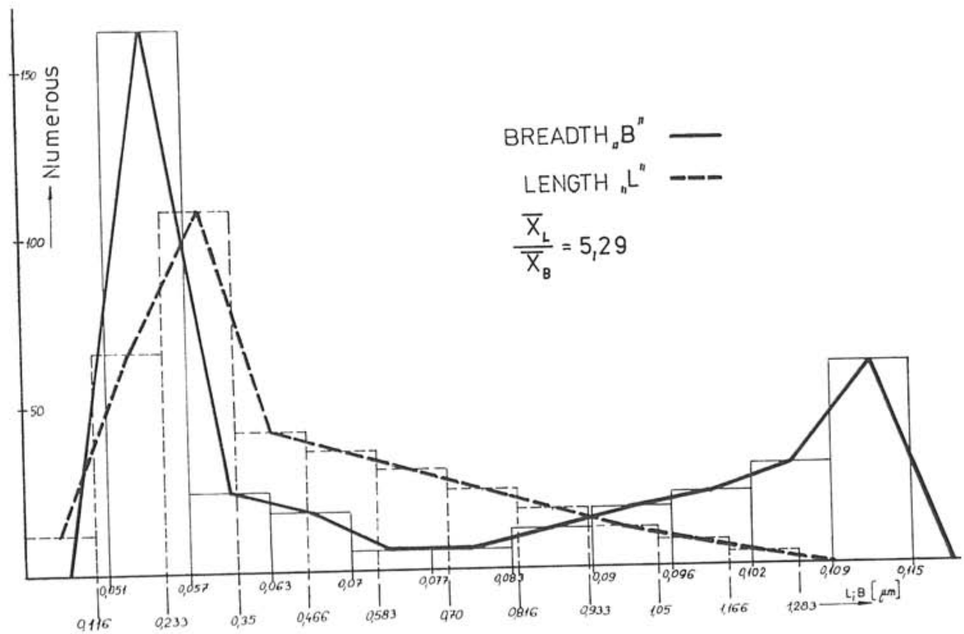


Fig 17. Frequency distribution of particle dimensions, sample Lučenecká kotlina basin, Halič-Kopáň.

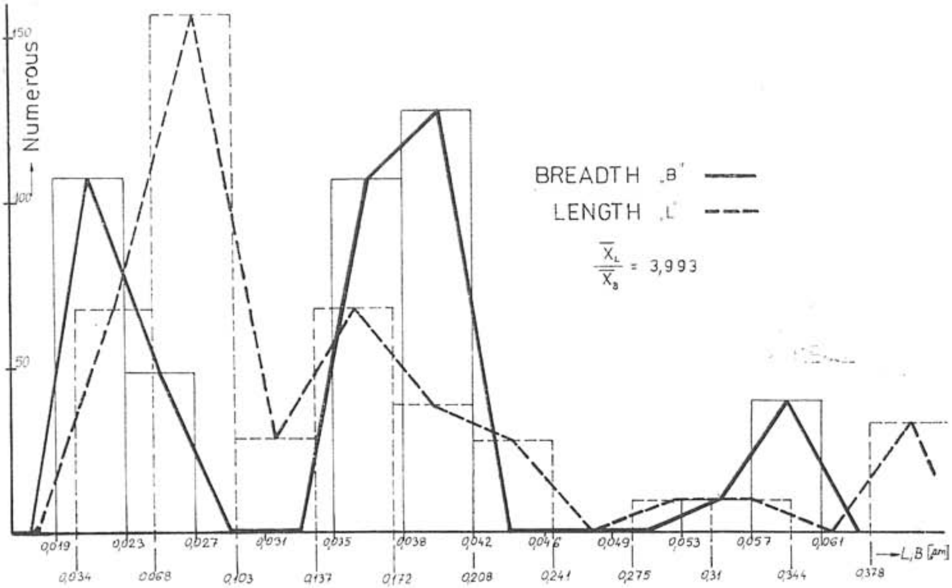


Fig. 18. Frequency distribution of particle dimensions, sample Lučenecká kotlina basin, Kalinovo-Hrabovo, halloysite $4H_2O$.

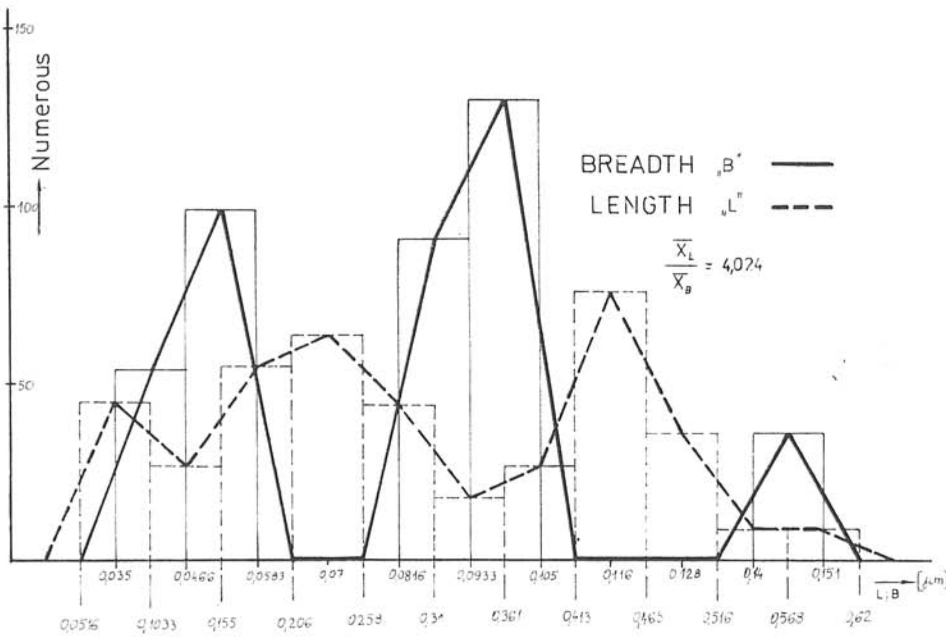


Fig. 19. Frequency distribution of particle dimensions, sample Lučenecká kotlina basin, Kalinovo-Hrabovo, halloysite $2H_2O$, fraction less than 1 micrometer.

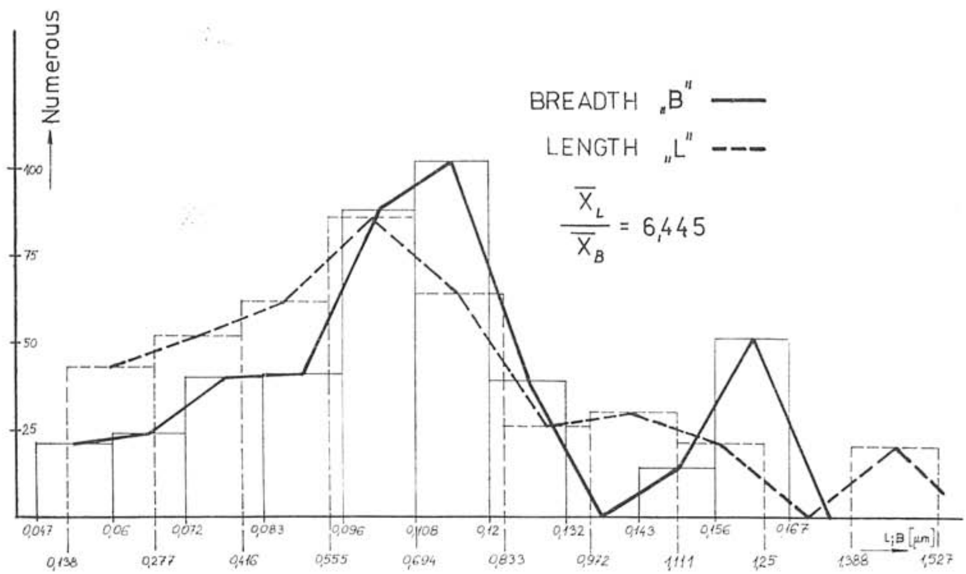


Fig. 20. Frequency distribution of particle dimensions, sample Bátovská kotlina basin. Tekovská Nová Ves.

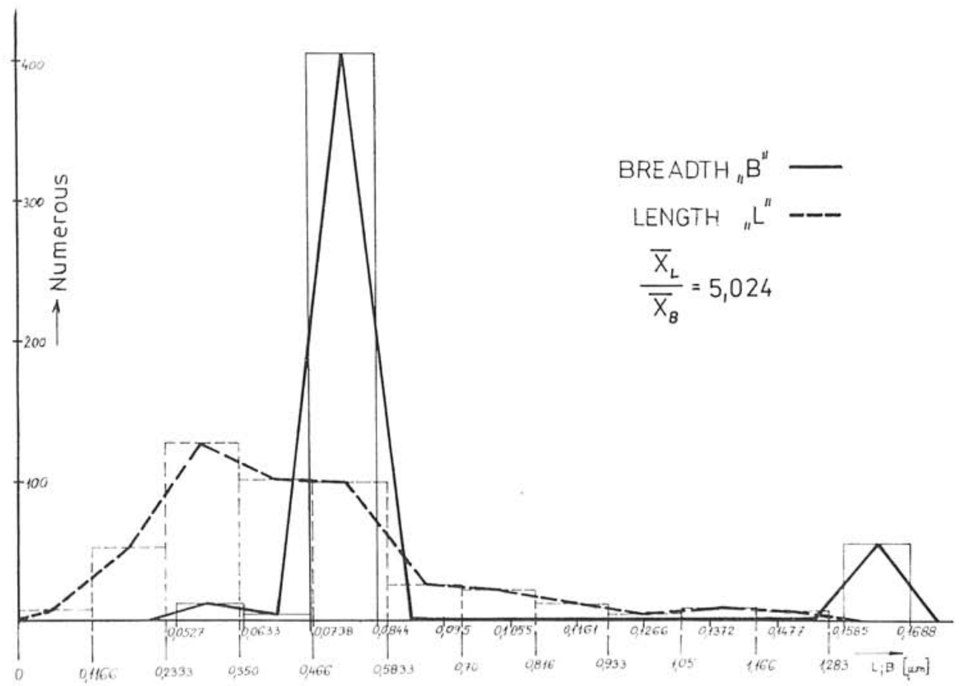


Fig. 21. Frequency distribution of particle dimensions, sample East Slovakian Neogene basin, Michalovce-Biela Hora (suspension).

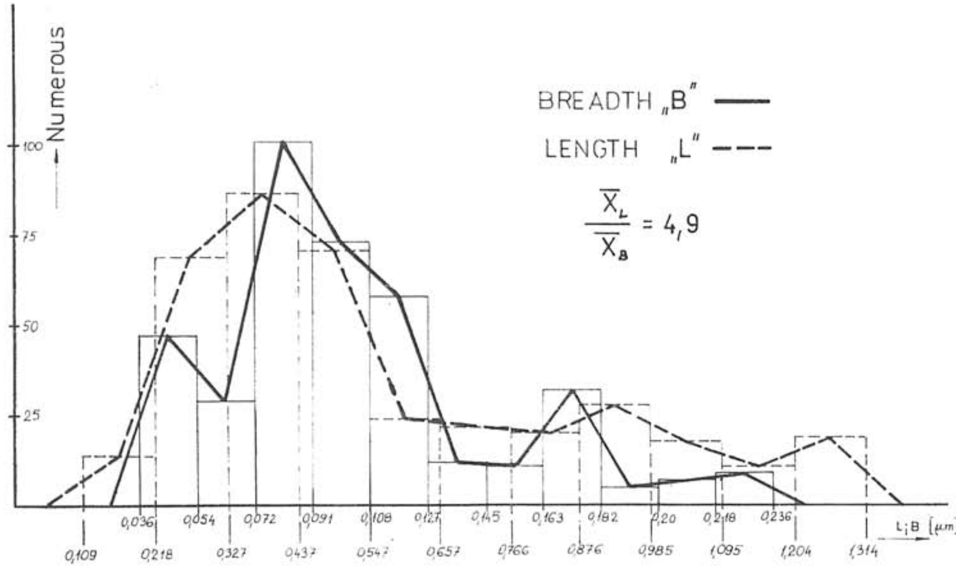


Fig. 22. Frequency distribution of particle dimensions, sample East Slovakian Neogene basin, Michalovce-Biela Hora (replica).

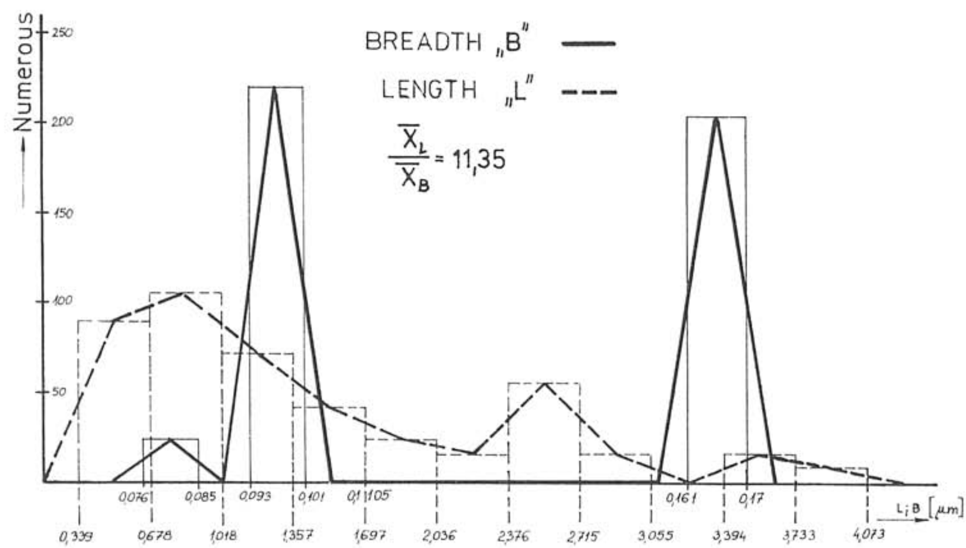


Fig. 23. Frequency distribution of particle dimensions, sample Karlove Vary area — Jimlikov.

The magnitude of interval we established on the basis of relation recommended by H. A. Sturges:

$$i = \frac{X_{max} - X_{min}}{1 + 3{,}322 \log N}$$

where i – width of interval
N – number of measured particles
$X_{max,\ min}$ – maximal and minimal value of measured parameter
The orders of frequency distribution treated this way we analysed then by means of arithmetic mean "$\overline{X}$", not only for characteristic parameters "L" and "B" but also their mutual ratio, characteristic parameter of shape,

$$\lambda = \frac{\overline{X}_L}{\overline{X}_B}$$

The results obtained by measurements of 12 samples are given in Table 1. Graphical records in Figs 14 to 24 make possible to have some idea about the size and distribution of particles. From 5 250 measurements of particles the widths varied within the range 0,019 to 0,25 micrometers and lengths from 0,07 to 2,80 micrometers.

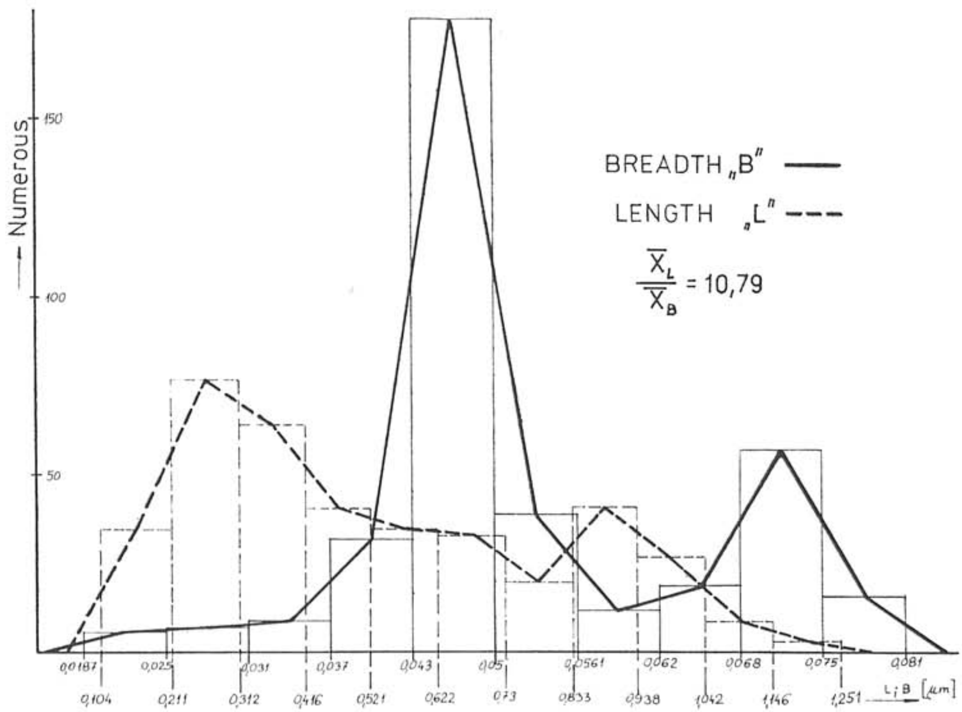


Fig. 24. Frequency distribution of particle dimensions, sample Douaria-Tunisia.

Table 1.
DIMENSION CHARACTERISTICS OF SAMPLES

Sample No.	Designation of samples	Maximum length of tubes	Minimum length of tubes	Maximum width of tubes	Minimum width of tubes	Most frequent length of tubes	Most frequent width of tubes	Ratio of arithmetic means of lengths and widths
1	Slatinská kotlina basin Sk-18/82,5 m	0,48	0,07	0,149	0,019	0,15	0,049 (0,02)	3,588
2	Slatinská kotlina basin Sk-25/27,2 m	1,33	0,07	0,16	0,039	0,48	0,108 (0,045) (0,158)	5,89
3	Lučenecká kotlina basin Halič THV-18	2,80	0,28	0,172	0,039	1,59 (0,71)	0,107 (0,056)	15,9
4	Lučenecká kotlina basin Halič-Kopáň	1,25	0,09	0,115	0,052	0,29	0,054 (0,112)	5,29
5	Lučenecká kotlina basin Kalinovo-Hrabovo halloysite $4H_2O$	0,40	0,038	0,06	0,019	0,085 (0,150) (0,39)	0,18 (0,021) (0,059)	3,993
6	Lučenecká kotlina basin Kalinovo-Hrabovo halloysite $2H_2O$ fraction less than 1 μm	0,6	0,052	0,15	0,037	0,44 (0,225) (0,076)	0,10 (0,051) (0,145)	4,024
7	Lučenecká dolina basin Kalinovo-Hrabovo halloysite $2H_2O$ fraction 1–2 μm	0,85	0,143	0,17	0,036	0,21 (0,47)	0,066 (0,103) (0,152)	4,056
8	Lučenecká kotlina basin Kalinovo-Hrabovo clayey cement	2,75	0,10	0,25	0,084	0,4	0,20 (0,145)	5,175
9	Bátovská kotlina basin Tekovská Nová Ves	1,52	0,14	0,165	0,049	0,625	0,112 (0,101) (0,161)	6,445
10	East Slovakian Neogene basin Michalovce-	1,31	0,11	0,23	0,041	0,37 (0,485) (0,264)	0,081 (0,100) (0,116)	4,9
	Biela Hora suspension extraction replica	1,28	0,105	0,168	0,043	0,275 (0,37) (0,48)	0,078	5,024
11	Karlove Vary area Jimlikov	4,05	0,35	0,17	0,078	0,82 (0,545) (1,16)	0,097 (0,165)	11,35

We have come to the conclusion that no sample contains only one statistically important width or length. In most cases there are up to three maxima.

We have also found out that particles with different parameters of shape as well as total distribution of individual widths and lengths may occur from the same area as it is in the case of the Slatinská kotlina basin (Fig. 14 and 15). Connected with different conditions of origin.

The measurements of tube widths of the same sample (halloysite from Michalovce) once prepared by the method of extraction replica (Fig. 11) and another time by suspension method (Fig. 10) have shown that the widths in suspensions are of greater value (Fig. 21, 22 and Tab. 1). We explain it so that the widths of tubes may enlarge during the process of sample preparation, probably by splitting, expanding or unfolding of some tubes.

From the statistical viewpoint to the results obtained appears as noteworthy regular relation between parameters "L" and "B", regarding to the character of halloysite formation (in situ, redeposited), and to quantitative representation of H_2O (halloysite $4H_2O$ – halloysite $2H_2O$).

Evaluation of results

In spite of that the last years brought an uncommon amount of knowledge for clay minerals from the kaolinite group, mutual relation and position between two most important minerals of this group – kaolinite and halloysite remain still open. Following this important problem by means of up to present methods, we often meet contradictory results. We know cases when kaolinite crystals of perfect morphological delimitation display typical features of disordered structure with X-ray diffraction and on the other hand, tubular particles show all features of well-ordered structure with X-ray diffraction. In solution of these questions in kaolinite group clay minerals two contradictory conceptions arose: G. W. Brindley and P. S. Santos (1966) expressed the opinion that a gradual order of minerals with one structure layer in the fundamental cell exists from kaolinite with high structure order, through mineral with disordered structure in direction of "b" axis (so called fireclay with one-dimensional disorder), to halloysite with high disorder in "a" and "b" axes directions – so called two-dimensional disorder. F. V. Chukhrov and B. B. Zvyagin (1966) consider halloysite as own mineral, characterized by monoclinic two-layer structure with parameters of elementary cell different from kaolinite. They prove that the degree of structure order changes in halloysite to a similar extent as in kaolinite and therefore they exclude the possibility that halloysite is the terminal member of the order with lowest structure order degree. According to these authors the degree of structure order in halloysite decreases with increasing content of water in interlayer space. The so called "tubular kaolinite", with triclinic twolayer structure according to G. Honjo et al. (1954) and P. S. Santos et al. (1965), is a typical halloysite with monoclinic two-layer structure after B. B. Zvyagin et al. (1966), which is an analogue of highly ordered kaolinite in this regard.

The presented study of morphological extent of halloysite at various localities also points to considerable variability in their structure order. This moment is distinctly shown in variations of shapes and sizes and in characteristic parameter of shape "λ".

I. Kraus et al. (1972) have found out that halloysite from the Lučenecká kotlina basin (samples No. 3, 4, 5, 6, 7, 8.) is characterized by a higher degree of structure order when compared with halloysite from the Slatinská kotlina basin (samples No. 1, 2,). In agreement with this statement it has been confirmed now that halloysite from the Lučenecká kotlina basin display a higher value of parameter of shape "λ", compared with halloysite from the Slatinská kotlina basin. Sample No. 3 (halloysite from the Lučenecká kotlina basin, Halič, THV-18) has the highest parameter of shape "λ" and fully supports the assumption that "λ" is a function of structure order degree.

Samples No. 11, 12 (Karlove Vary area – Jimlikov and Douaria-Tunisia) we similarly may range among halloysites with a higher degree of structure order, characteristic of which are also high values of the parameter of shape "λ". Some disproportion in this regard we recorded in halloysite from Michalovce-Biela Hora (sample No. 10), which is extremely finely dispersed, marked by a low degree of structure order and, however, with medium-high "λ" value.

Conclusions

1. The detailed study of shapes of tubular halloysite forms, which originated from rocks of various petrographic composition (granites, andesites, dacites, rhyolites) and age (Late Paleozoic, Miocene, Pliocene) has confirmed the probability of mutual relation between the degree of structure order and parameter of shape "λ". At the same time it has been confirmed that parameter of shape "λ" is reflection of conditions, under which halloysite formed.
2. The knowledge obtained supports the views of authors assuming that halloysite is an own mineral in which the degree of structure order changes to a similar extent as in kaolinite. The conception of halloysite as the end, disordered member of kaolinite order we further consider as little probable.
3. It has been shown that the detailed study of halloysite morphology is inevitable for establishing its structure order by the method of electron diffraction that will be a subject of further research.

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