ГЕОЛОШКИ АНАЛИ БАЛКАНСКОГА ПОЛУОСТРВА ANNALES GÉOLOGIQUES DE LA PÉNINSULE BALKANIQUE	67	109–116	БЕОГРАД, децембар 2006 BELGRADE, December 2006

Crystallite size distribution of clay minerals from selected Serbian clay deposits

VLADIMIR SIMIĆ[1] & PETER UHLÍK[2]

Abstract. The BWA (Bertaut-Warren-Averbach) technique for the measurement of the mean crystallite thickness and thickness distributions of phyllosilicates was applied to a set of kaolin and bentonite minerals. Six samples of kaolinitic clays, one sample of halloysite, and five bentonite samples from selected Serbian deposits were analyzed. These clays are of sedimentary, volcano-sedimentary (diagenetic), and hydrothermal origin. Two different types of shape of thickness distribution were found – lognormal, typical for bentonite and halloysite, and polymodal, typical for kaolinite. The mean crystallite thickness (T_{BWA}) seams to be influenced by the genetic type of the clay sample.

Key words: kaolinite, bentonite, halloysite, BWA technique, Serbia.

Апстракт. Мерење просечне дебљине кристалита филосиликата и њихове дистрибуције извршено је на узорцима каолинских и бентонитских минерала помоћу BWA (Bertaut-Warren-Averbach) метода. Проучено је шест узорака каолинитских глина, један узорак халојзита и пет узорака бентонита из изабраних лежишта у Србији. Те глине су седиментног, вулканогено-седиментног (дијагенетског) и хидротермалног порекла. Утврђена су два различита облика дистрибуције дебљине кристалита – логнормална типична за бентоните и халојзит, и полимодална типична за каолинитске глине. Просечна дебљина кристалита (T_{BWA}) изгледа да зависи од генетског типа узорака глине.

Кључне речи: каолинитске глине, бентонит, халојзит, BWA метод, Србија.

Introduction

The size distributions of crystallites can be measured by powder X-ray diffraction (XRD) because the widths of the XRD peaks broaden as the crystallite size decreases, if the influence of associated components on the degree of disorder of clay minerals (as presented for kaolinite by GALAN *et al*., 1994) is eliminated by adequate sample preparation. The interpretation of distribution and the shapes of crystallite thicknesses, measured by the Bertaut-Warren-Averbach (BWA) method, can then be related to crystal-growth mechanisms according to the theoretical approach of EBERL *et al*. (1998a).

The BWA technique has been applied to the measurement of illite particle thickness (EBERL *et al*., 1998b), to measure the crystallite size distribution of kaolin minerals (ŠUCHA *et al*, 1999), to explore crystal growth mechanisms for illite and smectite (ŚRODOŃ *et al*., 2000; MYSTKOWSKI & ŚRODOŃ, 2000), to study the diagenetic evolution of the crystallite thickness distribution of illitic material (KOTARBA & ŚRODOŃ, 2000), weathering processes which affected smectite and illite/smectite (ŠUCHA *et al*., 2001), and crystallite-size changes of pyrophyllite during grinding (UHLIK *et al*., 2000). EBERL *et al*. (1998a) studied the growth mechanism of minerals based on the shapes of the crystal size distribution.

Different clay deposits in Serbia have been explored and studied for many decades (SIMIĆ, 2001, 2004; SIMIĆ & JOVIĆ, 1997; RADOSAVLJEVIĆ *et al*., 1994; STANGAČILOVIĆ, 1970a, 1970b), but the crystallite size of the clay minerals has never been determined.

The main goal of this study was to measure the thickness and thickness distribution of kaolinite and smectite crystallites by the BWA technique and to compare the results with those obtained for similar clays from Slovakia and some other world deposits, and to

[1] Faculty of Mining and Geology, University of Belgrade, Đušina 7, 11000 Belgrade, Serbia. E-mail: simicv@rgf.bg.ac.yu

[2] Faculty of Natural Sciences, Comenius University, Mlynská dolina G, 84215 Bratislava, Slovak Republic. E-mail: uhlik@fns.uniba.sk

check if the mean crystallite size depends on the origin of the clay.

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Materials and methods

Twelve samples of kaolinites, halloysite, and smectites from selected deposits in Serbia were used for this study. The kaolinites were collected from the Vrbica (sample VRB), Ćirinac (CIR-Z), Lazine (L-1), and Košarno (KOS-5A) deposits (Aranđelovac basin), the Rudovci (RUD-3) deposit (Kolubara basin) and the Jasenovac coal mine, the halloysite was from the Novo Brdo deposit, and the smectites were from the bentonite deposits or occurrences Popovac (POP-1), Mečji Do (MD), Bogovina coal mine (BOG-I), Bivolica (BIV), and Drmno coal mine (D-3). These deposits were selected on the basis of their different clay minerals, genetic types and parent rocks.

Prior to analyses, < 2 mm fractions were separated from the bulk samples by sedimentation. Separated fine fractions were used for X-ray diffraction (XRD) analysis of oriented specimens. The oriented specimens were prepared by sedimentation of the clay suspension (10 mg/cm^2) onto glass slides. All specimens were analysed by XRD ussing a Philips PW 1710 diffractometer equipped with Cu radiation with a graphite monochromator. The step size was 0.02° 2Θ with a counting time of 5s for the oriented specimens.

The resulting basal reflections of the clay minerals were used for the determination of the mean crystallite thickness (crystallite = X-ray scattering domain) and thickness distribution by means of the BWA techniques (DRITS *et al.*, 1998) using the MudMaster program (EBERL *et al.*, 1996). The XRD method of crystallite size determination is based on the observation that XRD peaks broaden regularly as a function of decreasing crystallite size. The first basal reflection of all samples was subjected to BWA analysis in the recommended two theta intervals between 6 and 13° for the kaolinite (Fig. 1) and 2.5 to 7.5, for the smectites. All kaolinite samples, except the halloysite sample NB, contain illite in the clay fraction. Therefore, the illite peaks were chopped by the program PkChopr (Fig. 2). A longer XRD exposition time (5 s) was used to obtasin smooth XRD patterns for the analysis.

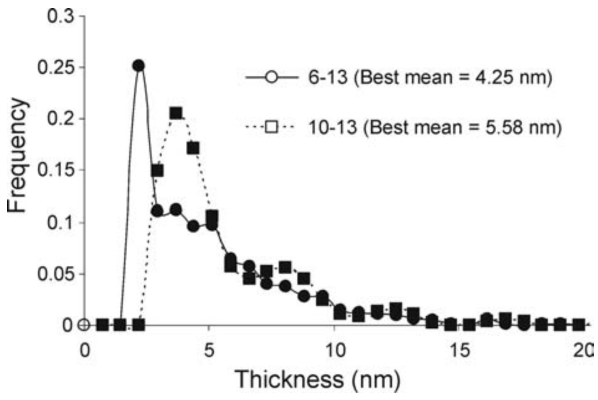

Fig. 1. Changes in the distribution and mean thickness of halloysite (sample NB) after using the incorrect analysed area (10–13° 2θ) in comparison with the recommended area (6–13° 2θ).

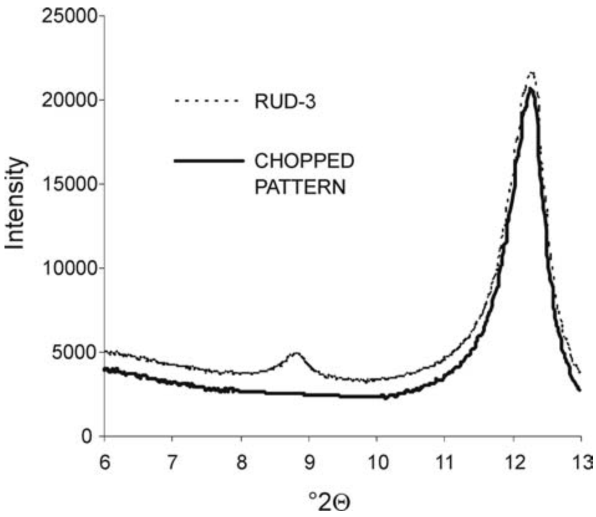

Fig. 2. Example of the modification of an XRD pattern before BWA-analysis the by PkChopr program.

Scanning electron images were taken from fresh rock chips coated with gold using a Jeol JXA 840 scanning electron microscope (SEM).

Geological features of the studied deposits

Samples of 6 kaolinites, 1 halloysite and 5 smectites from deposits in three different geological environments and origin were used for this study. Kaolinites and smectites from sedimentary (originating in a weathering crust and transported into sedimentary basins), volcano-sedimentary (formed by diagenesis of volcanic ash in a subaqual and subarial environment) and of hydrothermal origin were studied. The geological setting of each sample is indicated in Table 1. The analysed samples represent a selected collection of kaolinitic clays and bentonites from the economically most important deposits in Serbia, one sample of halloysite and one sample of Miocene tonstein, recently discovered in eastern Serbia.

Table 1. Basic geological features of the studied clays.

Sample	Genetic type	Age	Clay mineral	Basin	Deposit
Kaolinite					
VRB	Sedimentary	Miocene	Kaolinite	Aranđelovac	Vrbica
CIR-Z	Sedimentary		Kaolinite		Ćirinac
L-1	Sedimentary		Kaolinite		Lazine
KOS-5	Sedimentary		Kaolinite		Košarno
RUD-3	Sedimentary	Pontian	Kaolinite	Kolubara	Rudovci
JAS-5A	Volcano-sedimentary	Miocene	Kaolinite	Krepoljin	Jasenovac
Halloysite					
NB	Hydrothermal	Oligocene-Miocene	Halloysite (7Å)		Novo Brdo
Smectite					
POP-1	Volcano-sedimentary	Lower Miocene	Smectite	Paraćin	Popovac
MD	Volcano-sedimentary	Lower Miocene	Smectite	Zaplanje	Mečji Do
BOG-1	Sedimentary (?)	Miocene	Smectite	Bogovina	Bogovina
BIV	Sedimentary (?)	Lower Miocene	Smectite	Svrljig	Bivolica
D-3	Sedimentary	Pontian	Smectite	Kostolac	Drmno

The sedimentary kaolinitic clays were deposited in different basins, but in similar lacustrine settings. The kaolinitic clays from the Aranđelovac basin (Vrbica, Ćirinac, Lazine, and Košarno deposits) were formed by weathering and redeposition of materials from Bukulja granite (SIMIĆ, 2004). Kaolinite is the dominant clay mineral, with small amounts of illite. The clay from the Košarno deposit also has subordinate smectite. The parent rocks for the clays from the Rudovci deposit (Kolubara basin) are dacitic rocks and their pyroclastics. Kaolinite is also the most abundant mineral, accompanied by small amounts of smectite and traces of illite (SIMIĆ, 2004). The length of transport in both the Aranđelovac and Kolubara basins ranged from several hundred meters to 2–3 km, hence crystal disintegration during transport may have some influence on the crystallite size and thickness. Halloysite from the Novo Brdo deposit is of hydrothermal origin (MAKSIMOVIĆ & NIKOLIĆ, 1978). The kaolinitic clay from the Jasenovac coal mine is a typical tonstein, formed by the diagenetic alteration of volcanic tuff (ŽIVOTIĆ & SIMIĆ, 2003, unpublished report).

The smectite samples are also of different origin. Both Popovac bentonites, interbedded in marlstone in the quarry near the Paraćin Town and the Mečji Do deposit near the Vlasotince Town, are typical volcano-sedimentary rocks formed as a result of "in situ" subaqual alteration of the volcanic tuff. The bentonites from the Bogovina coal mine (East field) and the Bivolica deposit, near the Svrljig Town, are most probably the products of reworking of the weathering crust of andesitic rocks. The bentonite from the Drmno deposit (Kostolac coal basin) is of sedimentary origin (SIMIĆ *et al.*, 1997), but the primary source of clay minerals has not yet been established.

Results and discussion

Typical XRD patterns of each genetic type of clay are shown in Fig. 3, and the results of the BWA measurements of the kaolinite, halloysite and smectite samples in Table 2.

The T_{BWA} value of the sedimentary kaolinites studied varies between 5.55 and 7.91 nm, with an average value of 6.48 nm. The curves of all five samples are polymodal (Fig. 4), indicating that the samples consist of two or more generations of crystals with different thickness. The average T_{BWA} of the Serbian sedimentary kaolinites is slightly higher than the T_{BWA} of Slovakian sedimentary kaolinites, but, at the same time, significantly smaller than the T_{BWA} of selected world kaolinites (Table 3).

The BWA measurements confirmed the previously obtained geological, mineralogical and geochemical data that the weathering conditions during the Upper Oligocene–Lower Miocene did not lead to the origin of a well-developed kaolinitic weathering crust, neither in Serbia (MAKSIMOVIĆ & NIKOLIĆ, 1978; SIMIĆ, 2004), nor in Slovakia (KRAUS, 1989).

The halloysite sample of hydrothermal origin has a rather small mean crystallite thickness of 4.25 nm and a polymodal distribution pattern (Fig. 4). The T_{BWA} values of the Serbian and Slovakian halloysites are very similar, indicating a similar stage of hydrothermal alteration of the primary rocks. The distribution shapes of

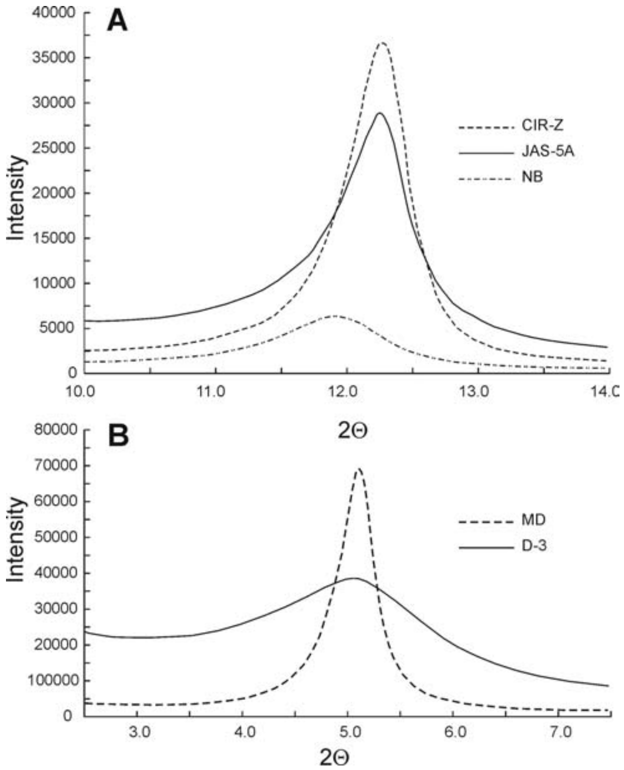

Fig. 3. Typical XRD patterns of the studied samples. A) Sedimentary kaolinite (CIR-Z), volcano-sedimentary kaolinite (JAS-5A), and hydrothermal halloysite (NB). B) Volcano-sedimentary smectite (MD), and sedimentary smectite (D-3).

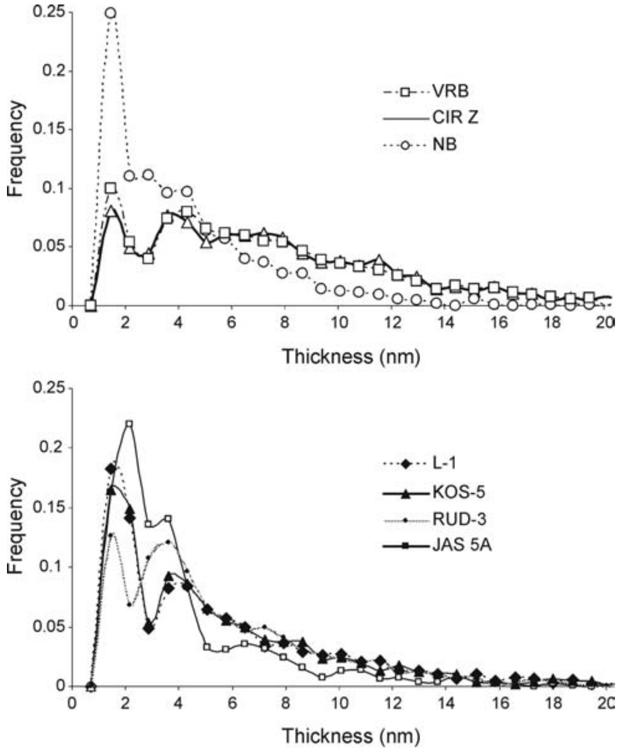

Fig. 4. Crystallite size distribution of kaolinites obtained by the BWA technique.

Table 2. List of clays used for BWA-analysis and the input and output data.

Sample	Genetic type	Position of peak maximum (θ)	d-spacing (Å)	Analysed area (θ)	Best mean (nm; extrapolated; area-weighted)
Kaolinite					
VRB	Sedimentary	12.27	7.21	6–13	7.53
CIR-Z	Sedimentary	12.29	7.20	6–13	7.91
L-1	Sedimentary	12.21	7.24	6–13	5.64
KOS-5	Sedimentary	12.30	7.19	6–13	5.55
RUD-3	Sedimentary	12.25	7.22	6–13	5.75
JAS-5A	Volcano-sedimentary	12.28	7.20	6–13	4.32
Halloysite					
NB	Hydrothermal	11.89	7.44	6–13	4.25
Smectite					
POP-1	Volcano-sedimentary	5.14	17.18	2.5–6	9.00
MD	Volcano-sedimentary	5.11	17.29	2.5–6	10.12
BOG-1	Sedimentary (?)	5.15	17.15	2.5–5.36	5.67
BIV	Sedimentary (?)	5.13	17.20	2.5–5.32	5.79
D-3	Sedimentary	5.07	17.41	2.5–4.88	5.21

Table 3. Average mean crystallite thickness (T_{BWA}) of Serbian, Slovakian and selected world kaolinites and halloysites. Values for the Slovakian and selected world kaolinites are from ŠUCHA *et al*. (1999).

Country	Genetic type	Average T_{BWA} (nm)
Kaolinites		
Serbia	Sedimantary	6.48
Slovakia	Sedimantary	4.81
World (selected)	Sedimantary	13.83
Serbia	Volcano-sedimantary	4.32
Halloysites		
Serbia	Hydrothermal	4.25
Slovakia	Hydrothermal	3.50

these two hydrothermal halloysites are different, as the Slovakian sample has an asymptotic shape and the Novo Brdo halloysite a polymodal one. The polymodal distribution of the Novo Brdo halloysite seems to be a combination of one lognormal and one asymptotic distribution. The asymptotic distribution is typical for samples with small T_{BWA} and could be characteristic for early stages of formation (EBERL *et al*., 1998a).

The tonstein sample of volcano-sedimentary origin also has a small mean crystallite thickness of 4.32 nm with a polymodal distribution pattern, but similar to lognormal (Fig. 4). The tonstein from the Jasenovac mine is generally weakly crystallized according to the XRD (Fig. 5) and at least two generations of kaolinitic minerals can be observed on the SEM image (Fig. 6), confirming the polymodal distribution shape.

The smectites from the volcano-sedimentary type have higher mean crystallite thickness with an average value of 9.56 nm than the smectites from the sedimentary bentonites with an average value of 5.56 nm. The crystallite size distributions for the volcano-sedimentary samples are lognormal (Fig. 7). Their shapes are quite different from the sedimentary types. The volcano-sedimentary smectites have identical distribution shapes with a theoretical lognormal distribution (Fig. 8A). The distribution of smectites from the sedimentary bentonites is different from the theoretical lognormal shape (Fig. 8B).

The mean thickness of smectites from selected world volcano-sedimentary bentonites varies from 6 to 9 nm (MYSTKOWSKI & ŚRODOŃ, 2000; MOL, 2001). The relatively wide range of T_{BWA} values of volcano-sedimentary smectites does not support the idea of using it to distinguish the origin in general. However, the measurement of T_{BWA} has sense for the differentiation of the origin of a bentonite in smaller regions, as was observed for the Serbian bentonites. A similar difference was found for in situ volcano-sedimentary and transposed bentonites from middle Slovakia (both types were characterized by ŠUCHA *et al*., 1996). Smectites originating from the in situ alteration of andesitic volcanoclastics have higher T_{BWA} values (up to 7 nm) in comparison with smectites originating by the redeposition of alteration products (5.5 nm).

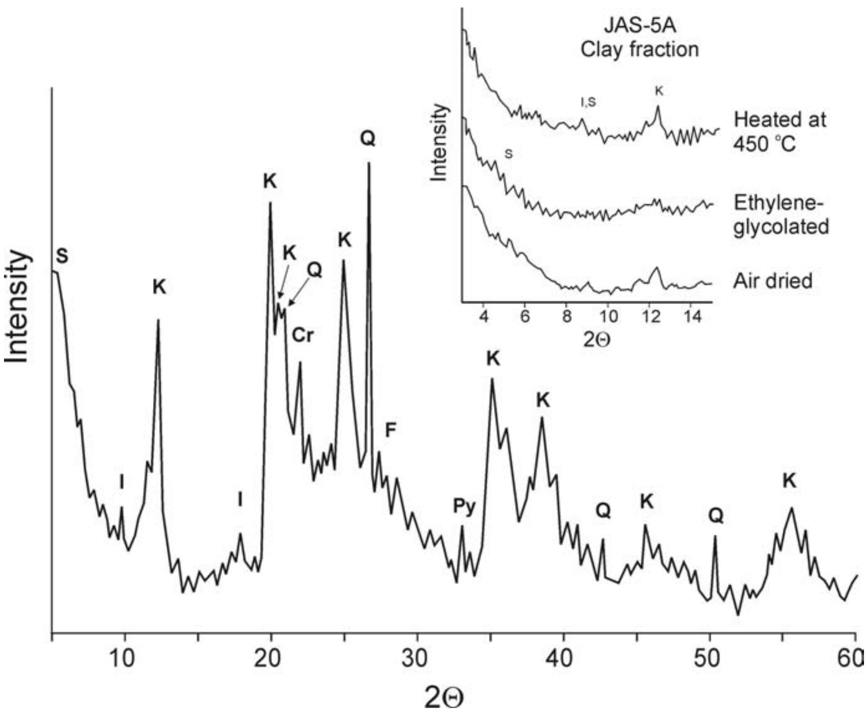

Fig. 5. XRD pattern of the kaolinite sample JAS-5A.
K – kaolinite,
S – smectite,
I – illite,
Q – quartz,
Cr – crystobalite,
F – feldspar,
Py – pyrite.

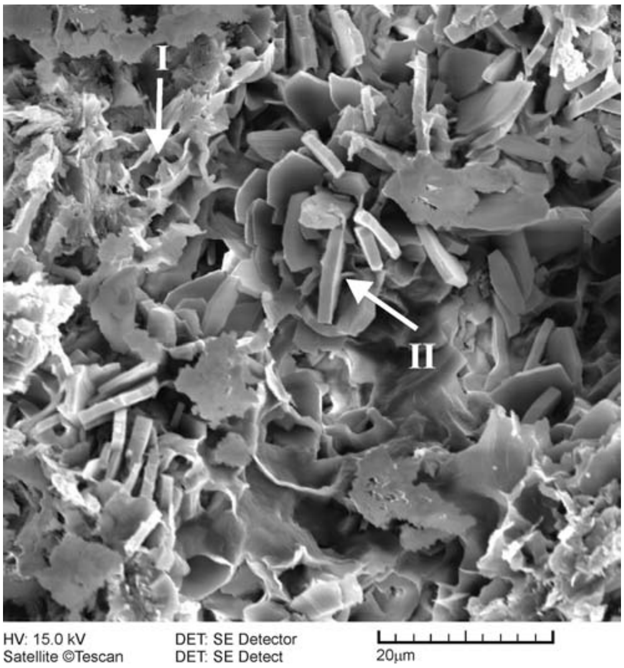

Fig. 6. SEM image of the kaolinite sample JAS-5A, showing two different particle generations (I, II).

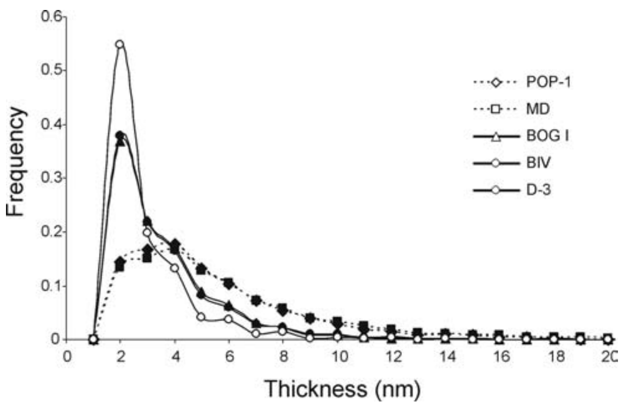

Fig. 7. Crystallite size distribution of the smectites obtained by the BWA technique.

Conclusions

Sedimentary kaolinites from five deposits in Serbia have a low mean crystallite thickness indicating a poorly developed kaolinitic weathering crust from which these clays were redeposited, a situation similar to Slovak kaolin deposits. The role of crystal disintegration during transport may also influence the crystallite size. The shape of the crystal size distribution is polymodal for all samples, most probably as a result of the presence of different kaolinite generations.

Volcano-sedimentary (diagenetic) tonstein from the Jasenovac coal mine has a very low mean crystallite thickness, typical for a weakly crystallized material, and a polymodal distribution shape, due to at least two generations of kaolinite particles.

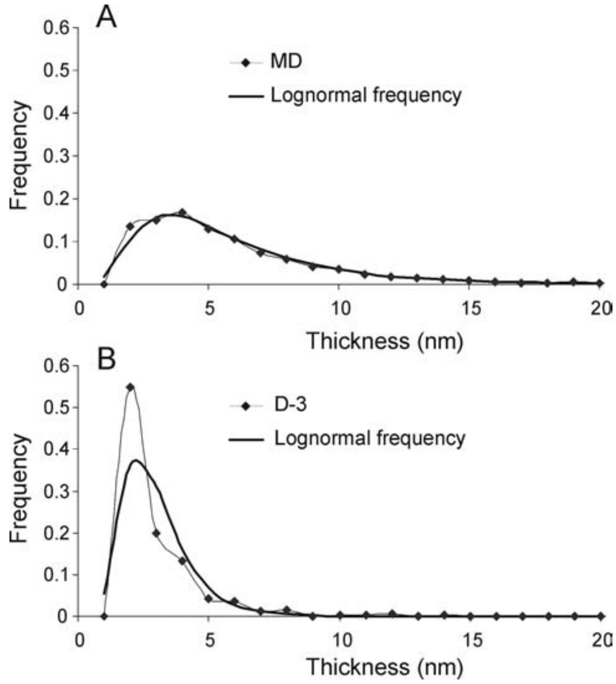

Fig. 8. Comparison of the measured (BWA) and the theoretical lognormal distributions of smectite particles. A) volcano-sedimentary type, B) sedimentary type.

Hydrothermal Novo Brdo halloysite also has a very low mean crystallite thickness and a polymodal distribution.

Two diverse shapes of the theoretically lognormal distributions were observed for the smectites. They correspond to different genetic types of bentonites – sedimentary and volcano-sedimentary. The mean crystallite thickness is also different in the sedimentary and volcano-sedimentary bentonites, with an average T_{BWA} of 5.56 and 9.56 nm, respectively. This means that "in situ" alteration of volcanic ash under subaqual conditions led to the formation of well-crystallised smectite with thicker crystallites.

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Резиме

Дистрибуција дебљине кристалита минерала глина из изабраних лежишта глина Србије

У овом раду измерене су дебљине кристалита каолинита и смектита и њихова дистрибуција методом BWA на узорцима из лежишта у Србији. Након извршених проучавања направљена је компарација са сличним глинама из Словачке и из неких светских лежишта.

Репрезентативни узорци 6 каолинитских глина, једног халојзита и 5 бентонита из лежишта ствараних у различитим геолошким условима: седиментним лежиштима глина (створених у корама распадања а затим транспортована у седиментне басене), вулканогено-седиментним лежиштима (насталим дијагенезом вулканског пепела у подводним или приповршинским условима) и хидротермалним лежиштима. Генетски тип лежишта и основне геолошке информације приказане су у табели 1.

Седиментни каолинити из пет проучаваних лежишта Србије одликују се малом дебљином кристалита, што указује на слабо развијен тип кора распадања из којих су ти минерали глина преталожени, као и на утицај уситњавања честица приликом транспорта. Таква ситуација је веома слична као код лежишта каолина у Словачкој. Облик криве расподеле дебљине кристалита је код свих узорака полимодални, највероватније као последица присуства различитих генерација каолинита.

Вулканогено-седиментни (дијагенетски) тонштајн из лежишта угља Јасеновац показује веома малу дебљину кристалита, типичну за слабо искристали-

сали материјал, и полимодални облик дистрибуције захваљујући присуству најмање две генерације каолинитских честица.

Хидротермални халојзит из лежишта Ново Брдо такође се одликује веома малом дебљином кристалита и полимодалним обликом расподеле.

Код испитиваних бентонита утврђено је присуство два различита типа теоретски логнормалне расподеле дебљине кристалита, које одговарају различитим генетским типовима бентонита – седиментном и вулканогено-седиментном. Средња дебљина кристалита је такође различита код седиментних и вулканогено-седиментних бентонита, са просечним T_{BWA} од 5,56 и 9,56 nm.