INVESTIGATION OF DISSOLUTION OF ALUMINOSILICATES IN AQUEOUS ALKALINE SOLUTION UNDER LABORATORY CONDITIONS

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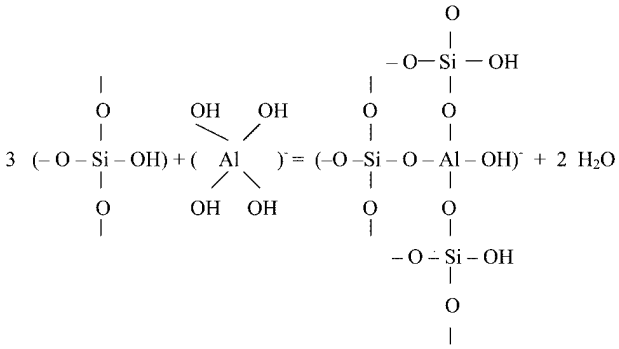
The investigation was aimed to the elucidation of the properties of colloidal mixtures of aluminosilicates in an aqueous alkaline solution and to the verification of the possibilities of sol-gel reactions at room temperature and atmospheric pressure. Basic factors influencing the reaction course are the dehydroxylation of the starting materials, their particle size distribution, the pH of alkaline aqueous solution and the intensity of agitation.

INTRODUCTION

The paper intends to explain the possibility of converting kaolin-like aluminosilicates to solutions of sol and the conditions of its transformation to gel. (The transfer of silicates to the solution means a real dissolution without previous melting with soda or borax). The dissolution of the aluminosilicates in aqueous alkaline solution by a chemical process without thermal influence needs a detailed knowledge of their behaviour from the point of view of chemical reactivity.

The dissolution reaction of SiO_2, especially the questions of hydration of SiO_2, leading to the formation of soluble silicates of alkali metals, is explained in many papers summed up in [1,2]. The properties of SiO_2 differ from the behaviour of aluminosilicates. The tendency of SiO_2 to form polymer chains changes if hydrated forms of Al_2O_3 are present. The anions of the [O-$Al(OH)_3$]- affect the polymerisation as follow:

1. The course of the dissolution reactions through the formation of [O-$Al(OH)_3$]- and a suggested complex:



2. The physicochemical behaviour of the formed systems sol-gel.

Thanks to the bond properties of aluminium and silicon atoms the transformation of sol to gel, the gelatination of the system and its conversion to long chain polymers takes place. These processes are described in papers [3,4]. There remain, however, a lot of unanswered questions of their reaction mechanism.

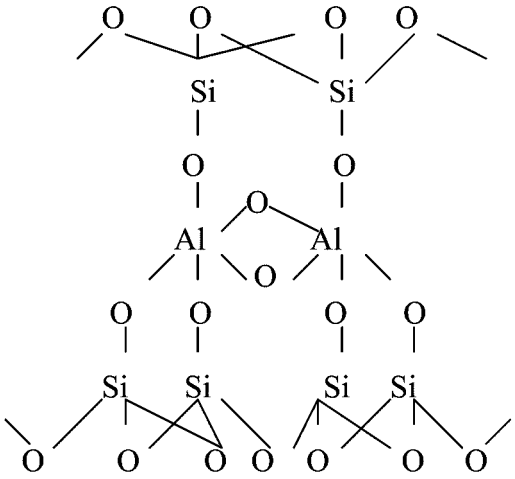
Basic factors that influence the following chemical reactions in the low temperature syntheses are the interlinking of octahedrons of $AlO_2(OH)_4$ and tetrahedrons of SiO_4 of kaolinitic clay and changes in its structure in the course of the dehydroxylation. The complete dehydroxylation and disintegration of the original kaolinitic structure means the formation of an unstable metakaolinitic form and the change of the Al atom coordination number from 6 to 4, or to 5. Both forms with the coordination number 5 and 4 are reactive, it means ready for hydration. The possible change in Al coordination could be described as:

$$AlO_2(OH)_4 = AlO_4 + 2H_2O \qquad (2)$$

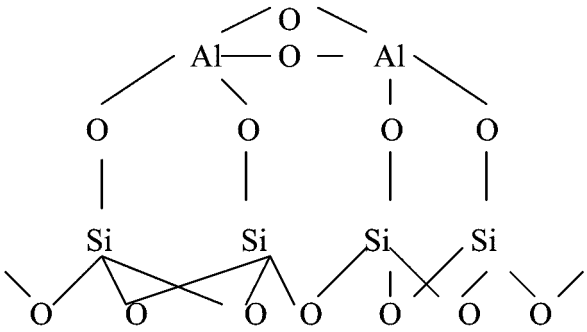
The change in the coordination number of Al ions during dehydroxylation of kaolin was studied in solid state by the NMR analyses [5]. The NMR spectra show mainly large peak signal at 0 ppm in the case of raw kaolin. Above the temperature of 400°C the signals at 30 and 60 ppm rise; this change could be explained by the reduction of anions bond to the Al ion. The line at 30 ppm is rather rare and was assigned to the andalusite (Al_2SiO_5). The Al atom is in the hybrid state of sp^3d with the coordination number 5 (penta-coordination

Al/5/). In the case of andalusite each Al ion shares two atoms of oxygen with another Al ion and three atoms of oxygen with silicon tetrahedrons formation. In the case of metakaolinite two Al ions share two oxygen ions, which belonged to the previous OH^- groups before the dehydroxylation. Each Al shares three oxygens with silicon in the tetrahedron network. Each of the two-shared oxygen atoms is from the original bond Si-O-Al and one oxygen atom is from the upper layer of SiO_4 tetrahedrons.

The possible penta-coordination of Al ions in metakaolinite is as follows [5]:



The offered [5] tetra-coordination (Al/4/) of Al ions in metakaolinite is as follows:



Brindly and Nakahira [6] suggested a metakaolinitic structure in which each Al ion is coordinated to one oxygen in the interlayer plane and three oxygen ions of SiO_4 tetrahedrons layer. Presented structures are considered as metastable formations and therefore highly reactive ones [5]. In aqueous alkaline solutions their dissolution takes place passing from the monomer formation on the surfaces of solid aluminosilicate particles, to form $[Al(OH)_4]^-$ and $[OSi(OH)_3]^-$ as precursors of the gel. Its formation continuously fills the space between the solid particles, giving rise to polymer gel structures where electronegative charges are equilibrated by the present cations. The equations expressing the dissolution process were proposed as follows (M^+ means Na^+ or K^+ ions):

$$(Al\text{-}Si)_{solid} + (OH^-)_{liquid} = \underset{monomer}{[Al(OH)_4]^-} + \underset{monomer}{[OSi(OH)_3]^-} \quad (3)$$

$$[OSi(OH)_3]^- + OH^- = {}^-OSi(OH)_2O^- + H_2O \quad (4)$$

$$^-OSi(OH)_2O^- + OH^- = {}^-OSi(OH)O^- + H_2O \quad (5)$$

$$\underset{monomer}{M^+ + {}^-OSi(OH)_3} = \underset{monomer}{M^+{}^-OSi(OH)_3} \quad (6)$$

$$\underset{monomer}{2M^+ + {}^-OSi(OH)_2O^-} = \underset{monomer}{M^+{}^-OSi(OH)_2O^-{}^+M} \quad (7)$$

$$3\,M^+ + {}^-OSi(OH)O^- = \underset{monomer}{M^+{}^-OSi(OH)O^-{}^+M} \quad (8)$$

$$M^+ + [Al(OH)_4]^- + OH^- = \underset{monomer}{M^+{}^-OAl(OH)_3} + H_2O \quad (9)$$

$$\underset{monomer}{OSi(OH)_3} + \underset{monomer}{M^+{}^-OSi(OH)_3} + M^+ = \underset{dimmer}{M^+{}^-OSi(OH)_2\text{–}O\text{–}Si(OH)_3} + MOH \quad (10)$$

$$\underset{monomer}{{}^-OSi(OH)_2O^-} + \underset{monomer}{M^+{}^-OSi(OH)_3} + M^+ = \underset{dimmer}{M^+{}^-OSi(OH)_2\text{–}O\text{–}Si(OH)_2O^-} + MOH \quad (11)$$

Previous knowledge and studies of dissolution of aluminosilicates in aqueous alkaline solutions [7,8] postulate the fundamental factors that mostly influence the formation and stability of the gel as a precursor of new materials as well as the preparation of such materials:

- The dehydroxylation. The realization of the sol-gel reaction of aluminosilicates is conditioned by the total dehydroxylation of the kaolin group raw material.
- Size of particles. The formation of dispersion environment of the sol is conditioned by the presence of particles of the order of micrometers.
- Value of *pH*. Dissolution is conditioned by the *pH* of the alkaline aqueous solution.
- Mixing. The intensity and time of mass transportation by convection are also conditions for the formation of the sol-gel reaction, similar role plays water content.

Theoretical studies [5,9] start from kaolinitic group, structurally double-layer types of aluminosilicates, which have the defined molar ratio Al_2O_3/SiO_2. Well defined water content in raw kaolinite $Si_2O_3.Al_2(OH)_4$ gives possibility to explain shifts of Al ions from the stable coordination number of Al/6/ to the metastable Al/5/ and Al/4/ ones.

The presented paper deals with kaolinitic waste materials from various industrial branches in the Czech Republic and finds out the next possible use of them. The main aim of this work is to recognize and define conditions of dissolution in alkaline aqueous solutions and also to open a discussion about the physicochemical processes in sol-gel systems.

EXPERIMENTAL

The used samples were recollected from mining company Keramost a.s. Most. One sample was from the producer of burnt shale clay ČLUZ a.s. Nové Strašecí. The task was to study the behaviour of contaminated (iron oxides and organic matters) kaolinitic materials and compare the dissolution conditions with dissolution of standard kaolin Sedlec Ia. Clay stones from the different part of mining deposit of Keramost a.s. Most are generally kaolinic materials with high content of Al_2O_3 (up to 42 wt.%) and Fe_2O_3 from 2 - 3 wt.%. The samples of clay stone W-extra-J-tm and W-special-J-tm have dark colour due to the content of organic matters up to 3 wt.%. The sample of burnt shale clay, also kaolinitic type, obtained from textile filter of milling industrial process was contaminated by iron oxides and partially preheated.

The method of solubility estimation was based on mass losses of prepared samples. The treatment in alkaline aqueous solution was studied as a function of the concentration of alkali and exposure time in the agitated solution. The degree of dissolution of the samples was pursued also by checking the distribution of clay particles. The samples were prepared by milling up to a defined size composition, with the maximum size of particles up to 100 micrometers. The solubility of raw-material samples was compared with the solubility of dehydroxylated ones, prepared by heating them up to 750°C for 6 hours. The temperature and time of dehydroxylation was defined according to the behaviour of standard kaolin Sedlec Ia.

The samples weight in all experiments was 5.0000 ± 0.0001 g. Low-temperature syntheses were performed under usual laboratory conditions of temperature and atmospheric pressure. Samples were treated in 40 ml of 5 M and 10 M NaOH aqueous solutions and constantly agitated. The time of agitation varied from 1.5, 2, 3, and 4 up to 6 hours of dissolution. The residues of the samples were filtered and washed by distilled water. Later, they were dried to constant weight, subsequently heated at two different temperatures, 220°C and 500°C. In all three temperature steps their mass losses, the influence of agitation time and NaOH concentration in the aqueous solution were checked. The industrial samples of clay stones and burnt shale clay were not heated after treatment in 5 M NaOH at 200°C and 500°C because of very low differences in mass losses of dehydroxylated samples.

For the identification of the particle size distribution the Laser Diffractometer CILAS 920L series 166 was used. The solid-state NMR spectra were obtained by the spectrometer Bruker DSX 200 NMR. The curves of XRD diffraction were done by diffractograph Philips PW 1840, radiation Cu 1.540562 A, angle range 2.800 - 9.740 deg.

RESULTS AND DISCUSSION

Some obtained knowledge shows the necessity to describe the behaviour of aluminosilicates by the solid-state NMR analyses better then study through DTA curves [5]. It is necessary to explain the origin of inorganic sol-gel systems on the basis of the formation of siloxo-sial chains. First of all the behaviour of Al ions which in the original materials form one of the main construction element and at the same time the key to their reactivity.

The investigation of dehydroxylation of kaolinic structure was checked at three different temperatures, starting with 600°C by the X-ray diffraction (XRD) method. It was found, that the complete disintegration of well-organized kaolinitic structure is attained above 600°C and depends also on the thermal exposition time. Our analyses of kaolin Sedlec Ia. show the typical kaolinitic increase, even at the temperatures of 600 or 700°C, on the XRD curves close to 20 deg (figure 1). The diminution of it could be seen on the line of 850°C. In agreement with [10] it means that the disintegration of the crystallographic network of well-organized kaolinite is not completed as yet. Also observation by the solid-state NMR confirms the possible change [5] in coordination number of Al ions of investigated kaolin Sedlec Ia. dehydroxylated at various temperatures (figure 2). The presented spectra (^{27}Al CP/MAS NMR) could give only a qualitative indication due to the low differentiation, given by the used magnetic strength. However, according to [5] it is possible to assume that the geometry of atoms coordinated around the Al atom has changed. The state of disequilibrium is caused by the temperature rise. A widening of the NMR signal confirms supposed change in coordination number of Al ions. The curve at a higher dehydroxylation temperature shows that the intensity of the unsymmetric broad signal rises up to the 50 ppm, which could be explained as a change in the coordination number of Al. Both experiments with dehydroxylated standard kaolin Sedlec Ia. - NMR and XRD - fixed the temperature and time of dehydroxylation of all samples, means total destruction of original structural red.

Our results confirm changes in coordination number of Al ions through the dissolution of raw kaolin Sedlec Ia. Raw kaolin presents low mass losses even if the molarity of alkaline aqueous solution rises from 5 M to 10 M. On the contrary, in case of dehydroxylated kaolin the more concentrated solution of NaOH exhibits the stronger attack and the solubility rises (table 1).

Table 1. The solubility of kaolin samples indicated by the losses of mass (wt.%).

Sample	5 M NaOH	10 M NaOH
raw kaolin	2.07	2.07
dehydroxylated kaolin (750°C)	12.57	24.09

Table 2. The solubility of raw and dehydroxylated kaolin indicated by loss of mass (wt.%) and the influence of temperature. Time of agitation was 3 hours.

Sample in solution of NaOH	The loss in mass by dissolution	Difference in mass loss by 220°C	Difference in mass loss by 500°C
raw kaolin, 5 M NaOH	2.07	0.28	6.66
raw kaolin, 10 M NaOH	2.07	0.34	8.46*)
dehydroxylated kaolin at 750 °C, 5 M NaOH	12.57	0.58	0.53
dehydroxylated kaolin at 750 °C, 10 M NaOH	24.09	0.29	0.76

*) The possible interpretation of higher loss could be explained by the loss of more OH^- groups

Table 3. The loss of mass of dehydroxylated kaolin as a function of time of agitation (wt.%).

Sample	Agitation time				
	1.5 h	2 h	3 h	4 h	6h
kaolin, 750°C, 5 M NaOH	7.2	11.4	12.6	11.6	10.1

Table 4. The loss of mass of dehydroxylated burnt shale clay as a function of time of agitation (wt.%).

Sample	Agitation time		
	1.5 h	3 h	4.5 h
burnt shale clay, 750°C, 5 M NaOH	4.86	13.34	13.52

The samples of kaolin were, after the treatment in NaOH solution, dried to constant weight at 110°C and later heated at 220°C and 500°C with the goal to check a potential loss of mass. The results are shown in table 2.

From the data in table 2 results that the hydration does not dissolve raw kaolin and, on the contrary, dehydroxylated kaolin is dissolved. This confirms the supposed hydration reactions in alkaline aqueous solutions [11]. The loss of mass of the raw samples at 500°C is due to the loss of crystalline water. The course of dissolution of dehydroxylated kaolin as a function of on time of constant agitation is presented in table 3.

The course of dissolution could be, on the basis of presented suggestions [12,13], explained as follows: dissolution of dehydroxylated kaolin rises up to certain limit, showed in table 3 with a maximum of the loss of mass after 3 hours of agitation. Later on, according to [12,13], a next stage comes - the formation of longer chains of silicates and the polymerization of aluminosilicate. This is a reason why the loss of weight, after 4 and 6 hours of agitation, is lower than at a shorter time. From the behaviour of dehydroxylated kaolin it could be assumed that the prolongation of agitation time leads to the rise of polymer chains. Estimated dissolution and in the mean time formation of new solids (residues on the filter) is functions of time and velocity of stirring. The presence of Na^+ ions brings to the formation of dimmers and longer chains [7,8,14,15]. According to [14] the new solids are generally amorphous. The similar course of dissolution of the sample of burnt shale clay is presented in table 4.

The application of following investigation was orientated to the waste and secondary materials of the kaolinitic type materials with double-layer network, which are contaminated and than rarely used in classic ceramic technology. The dissolving conditions were the same as in case of kaolin. On the beginning of dissolution estimation the distribution of the particle sizes were checked comparing them with the distribution of kaolin Sedlec Ia. ones. The used materials and their particle size distribution are presented in table 5.

From the table 6 is evident that the dissolution of raw aluminosilicates in alkaline aqueous solutions is rather low. Solubility rises considerably when the condition of dehydroxylation is fulfilled. Very close values of dissolution (range from 10.97 to 13.34 % of weight) in 5 M solution of NaOH could be explained through the determinate quantity of Na^+ ions. The example of dissolution of kaolin in 10 M solutions of NaOH (table 1) leads to the conclusion that the more Na^+ ions cause the higher solubility. This conclusion supports presented equations (3-11) where a part of the above-mentioned dependencies of the estimated dissolution is also a function of molar ratios SiO_2/Na_2O and Al_2O_3/Na_2O.

Table 5. The particle size analysis of the investigated dehydroxylated materials (wt. %).

Sample	Up to 2 μm	Up to 4 μm	Up to10 μm	Up to 20 μm	Up to 40 μm	Up to 100 mm
kaolin Sedlec Ia.	24.90	42.54	73.27	94.71	100.00	100.00
burnt shale clay	18.07	36.67	77.65	98.22	100.00	100.00
clay stone W-super	6.08	13.19	25.74	36.04	48.05	76.80
clay stoneW-special	6.80	14.09	27.11	38.75	53.00	81.00
clay stone W-extra	7.41	15.68	29.89	41.99	55.15	78.00

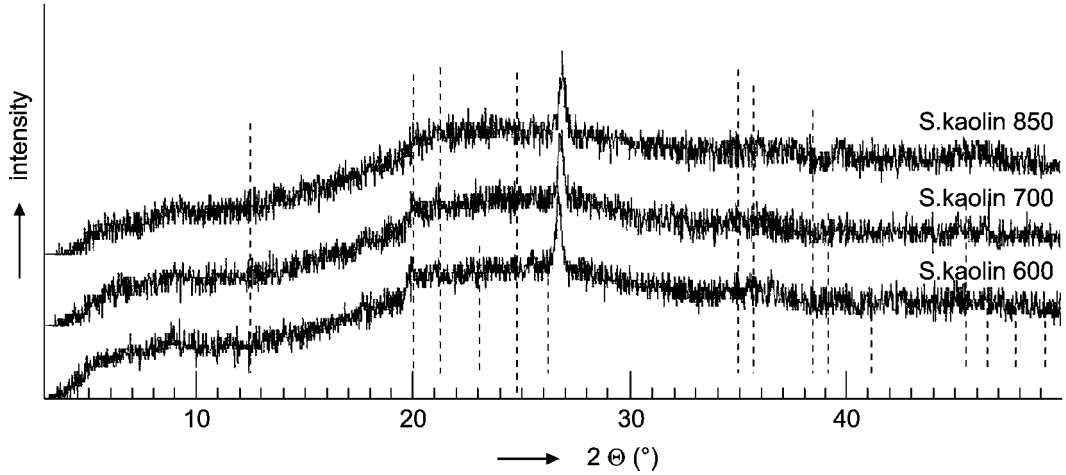


Figure 1. The XRD pattern of the Sedlec I.a kaolin treated at various dehydroxylation temperatures.

Table 6. The loss of mass of raw and dehydroxylated materials (wt.%) - 5 M NaOH, 3 hours of agitation.

Sample	Raw	Dehydroxylated
kaolin Sedlec Ia.	2.07	12.57
burnt shale clay	8.26	13.34
clay stone W-super	1.24	10.97
clay stone W-special	2.59	11.91
clay stone W-extra	6.90 *)	12.38

*) The increase in the quantity of dissolved particles part in this case is probably due to the dissolution of organic matters in clay stone. (The filtrate of the solution was dark).

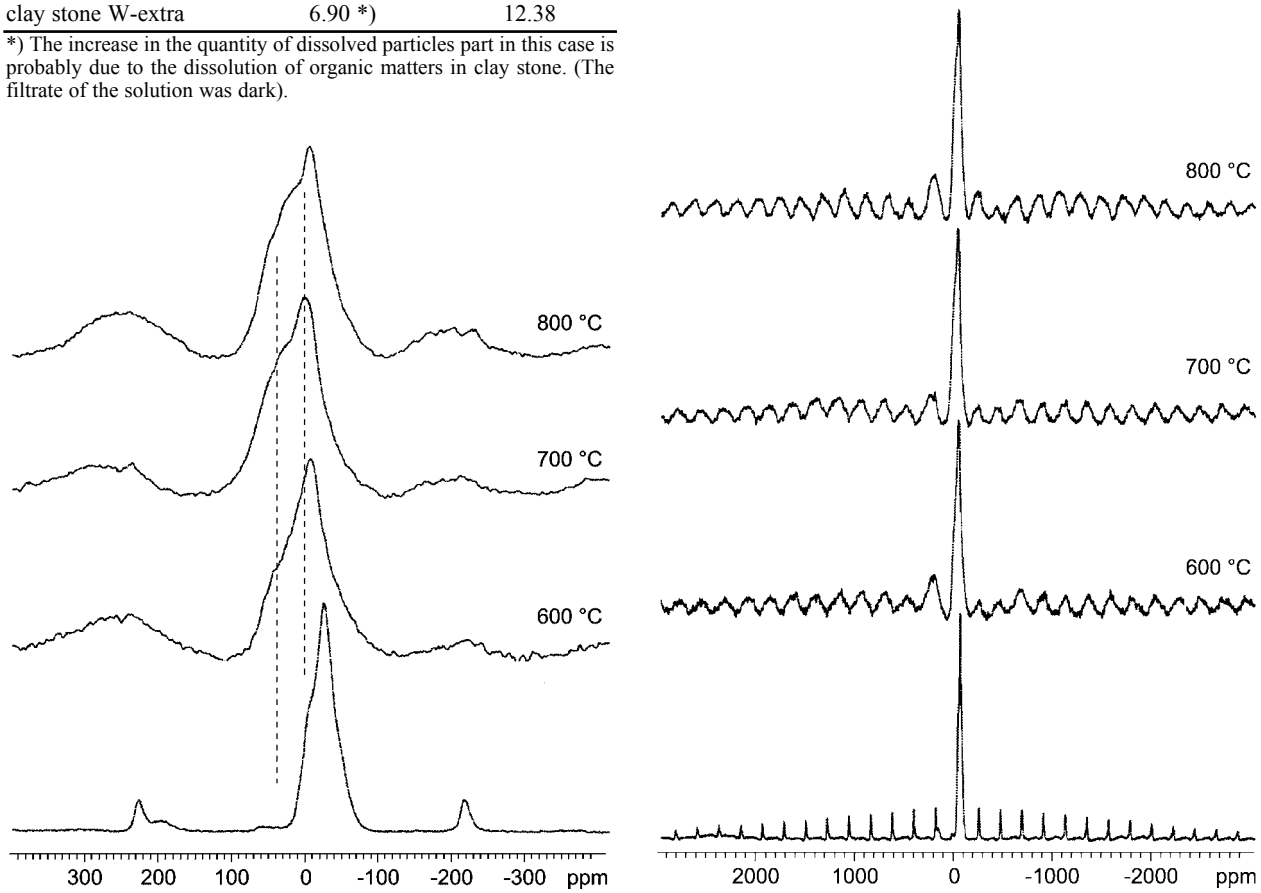


Figure 2. The ^{27}Al CP/MAS NMR spectra of the Sedlec I.a kaolin treated at various dehydroxylation temperatures (upper part) and ^{29}Si CP/MAS NMR spectra of kaolin Sedlec I.a.

Table 7. The particle size distribution of clay stone W-super (wt. %).

Particles	Up to 2 μm	Up to 4 μm	Up to 10 μm	Up to 20 μm	Up to 45 μm
laboratory milling	6.08	13.19	25.74	36.04	49.99
powder from the textile filter	6.53	15.56	34.26	51.47	73.57

Table 8. The particle size distribution of clay stone W-special (wt. %).

Particles	Up to 2 μm	Up to 4 μm	Up to 10 μm	Up to 20 μm	Up to 45 μm
laboratory milling	6.8	14.09	27.11	38.75	55.52
powder from the textile filter	6.17	15.24	34.91	52.28	77.67

It was found that the dissolution of tested raw materials is similar as in the case of kaolin. The only exception is the case of powdered burnt shale clay, which was partially preheated by the previous industrial process. The quantity of aqueous alkaline solution between the treated particles has the main influence on the gelatination of such a system. In the cases of completely dehydroxylated kaolinic structures it is considered that the smaller are the particles the greater part of them could be transferred to the sol-gel system. The quantity of dissolving water must permit the hydration and transportation of alkali ions or alkaline earth compounds which, in consequence, balance the negative charge of the groups $[Al(OH)_4]^-$ and their complexes with $Si(OH)_4$ or monomers $[OSi(OH)_3]^-$ etc. The water content also influence on the formation of the sol-gel system. The distance between the dissolving particles could be smaller or larger due to the water content. The formation of monomers on the surfaces of solid particles than fills larger or smaller distances among the particles.

If the samples of clay stone W-super are micromilled directly by the technological line or are gathered from the textile filters from the industrial production and compared with the distribution of particles attained by the capacities of laboratory milling we observe similarity in particle sizes distribution (tables 7 and 8).

The quantity of dissolved particles is proportional to the level of dehydroxylation when the part of small fractions (i.e. sum up to 4 micrometers) is similar or very close in both types of milling. It was found that during dissolution of the completely dehydroxylated sample of clay stone heated at 750°C the loss of mass were 11.09 % but only 8.01 % in case when the temperature of dehydration attained only 600°C. This difference supports previous opinion of the importance of dehydroxylation process. Lower temperature means less transformation in the original network. The reactivity of the samples with the same content of the smallest (reactive) particles rises proportionally to the dehydroxylation of the samples and the conversion of the system sol-gel proceeds more easily. The influence of particles larger than 20 micrometers is not remarkable in sol-gel reactions but their presence and ability of the hydration of their surface layer could serve as a nucleus of gelatination. The gel around the solid particles encapsulates undissolved parts in case when the quantity of aqueous solution allows minimal distances between them.

The powder of partially burnt shale clay recollected from the textile filter of the industrial milling production has a similar behaviour as kaolin. Complete dehydroxylation of such a powder leads to dissolution of 13.52 % of original weight during 4.5 hours of agitation. Dehydroxylated powder from industrial production of burnt shale clays could be transferred similarly as kaolin to the sol-gel system, where the ability of aluminosilicates chains formation begins. The course of the system sol-gel formation is conditioned by the same factor [16] as in the case of kaolin. The iron and titanium oxides content in samples of industrial waste materials (i.e. powders of burnt shale clay and clay stone W-special) does not change the behaviour of dissolution and change only the colour of filter residues.

CONCLUSION

The double-layers aluminosilicate materials could be transferred to the sol-gel system only after complete dehydroxylation and with the corresponding particle size. By the quantity of water the distance of the particles and the transfer of obtained sol-gel could be regulated.

A larger distance of particles means a lower possibility of gelatination. The quantity and concentration of OH^- ions in an aqueous solution has crucial influence on the level of hydration and solubility, similarly as intensity and time of agitation. It was found that the transportation of mass by convection ensures together with the quantity of water the hydration reaction and transfer of system sol-gel to the formation of polymer chains.

The finding that used waste kaolinic materials, very often contaminated by organic matters or/and by ferrous and titanium oxides, exhibit the same behaviour as kaolin, could have a consequence for their future industrial use.

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STUDIE ROZPOUŠTĚNÍ ALUMINIUM-SILIKÁTŮ VE VODNÉM PROSTŘEDÍ ZA LABORATORNÍCH PODMÍNEK

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Pro objasnění dosud málo známých dějů v koloidní soustavě sól-gel v případě aluminium-silikátů typu kaolinitu byly ověřeny možnosti převedení těchto silikátů do roztoku sólu a přechodu sólu v gel za laboratorní teploty a atmosférického tlaku. Dosavadní práce vycházejí z kaolinitických, strukturně dvojvrstvých typů, které mají definovaný molární poměr Al_2O_3/SiO_2 a současně vykazují přechod iontu Al s koordinačním číslem 6 (stabilní forma) na iont s koordinačním číslem 4 a 5 (metastabilní formy). Tato práce vychází z kaolinitických materiálů získaných z různých oblastí průmyslové činnosti v ČR. U těchto materiálů byly určeny podmínky rozpouštění a tvorby sol-gel soustav ve vodném prostřední za laboratorních podmínek. Základními faktory tvorby gelových prekurzorů nových látek jsou dehydroxylace výchozího materiálu, velikost částic, hodnota *pH* vodného prostředí a míchání.