

## The effects of grinding on the physicochemical characteristics of white sepiolite from Golesh

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The influence of grinding white sepiolites from the Magure-Golesh mine in Southern Serbia on their structure and on the rheological characteristics of their aqueous suspensions was investigated. Sepiolite samples of hard and soft consistency were ground in three different types of mills: a ball mill, air-stream mill, and a colloid mill. The effect of grinding on the sepiolite samples was investigated by SEM, XRD, IR, TG and BET methods and by chemical analysis. Grinding generally resulted in the separation of the sepiolite fibers, and partial amorphization. In addition, grinding produced substantial changes in the apparent viscosity and rheological characteristics of dilute aqueous suspensions of sepiolite. It is concluded that the viscosities are especially affected by the type of grinding employed.

*Keywords:* sepiolite, Golesh, grinding, structure, rheological characteristics.

### INTRODUCTION

The effects of grinding on the physicochemical properties of many clay minerals, for example kaolinite,<sup>1,2</sup> montmorillonite<sup>3</sup> and chrysotile,<sup>4</sup> have been investigated. The influence of grinding on the properties of sepiolite has only been partially investigated. Research on the structural alterations of sepiolite upon grinding involved only the process of dry grinding in a ball mill.<sup>5</sup>

Investigations of the structural changes occurring during the grinding of sepiolite from the Golesh mine, in Serbia,<sup>6</sup> have been commenced. Since sepiolite is a phyllosilicate of fibrous morphology<sup>7</sup> with an anisotropic structure, different methods of grinding may produce various structural changes (thinning of the fibers, distortion of the parallel units, or disruption of the structure). Also, since sepiolite is strongly hydrophilic, the grinding media may produce substantial differences in the quality of the products obtained by dry or wet grinding processes. For this reason, three mill types (ball mill, air-stream mill, and colloid mill) were employed. In the study of the structural

alterations of sepiolite, the effects of grinding were investigated in terms of its influence on the structure, morphology, and thermal characteristics, as well as on the changes of the specific surface area. The XRD, SEM, TG-DTA, and BET methods, as well as chemical analysis were used.

Since it is well known that phyllosilicates are used as thickeners and rheological additives,<sup>8</sup> the effects of grinding on the rheological properties of aqueous suspensions of sepiolite were also investigated. The changes in the apparent viscosity of dilute aqueous suspensions of sepiolite were measured using a structural-rotational viscosimeter.

### EXPERIMENTAL

Samples of sepiolite of soft and hard consistency originating from two levels of the Magura 14 vein (horizon 10 and horizon 14) were investigated. Their consistencies were as follows: samples 1H14 (soft consistency, horizon 14), 2H14 (hard consistency, horizon 14), 1H10-3H10 (soft consistencies, horizon 10), 4H10-6H10 (hard consistencies, horizon 10). All samples were prepared by drying at 105 °C and by manual crushing to pass through a 2-mm sieve. The chemical compositions of the samples are presented in Table I.

TABLE I. Chemical composition of the sepiolites from Golešh

% Oxide	1H10	2H10	3H10	4H10	5H10	6H10	1H14	2H14
SiO <sub>2</sub>	52.58	51.80	46.84	53.72	52.71	53.60	49.52	34.60
Fe <sub>2</sub> O <sub>3</sub>	0.06	0.05	0.05	0.05	0.05	0.21	0.08	0.08
CaO	0.18	0.18	0.20	0.13	0.37	0.14	0.23	0.48
MgO	25.53	26.29	27.99	25.26	24.59	23.96	27.31	31.80
NiO	0.03	0.04	0.04	0.03	0.04	0.05	0.03	0.05
Na <sub>2</sub> O	0.30	0.28	0.24	0.24	0.29	0.26	0.31	0.28
Loss on ignition	20.97	21.59	24.09	21.03	21.57	20.94	23.88	31.18
Total	99.65	100.23	99.45	100.46	99.62	99.16	101.36	98.47

Sepiolite was ground by dry and wet processes. Dry grinding was performed with two mills: a porcelain ball mill and a Bifar air-stream mill. The laboratory ball mill was a VEB Special Maschinenbau type 260-22, consisting of a 6.3-dm<sup>3</sup> porcelain container (working volume of 4.2 dm<sup>3</sup>) with porcelain spheres 25 mm in diameter, moving in planetary fashion at 68 rpm. All experiments were conducted with 1.5 kg batches of sepiolite. The soft-consistency sepiolites were ground for 24 h, 48 h, 72 h, 96 h, and 120 h, and the hard-consistency sepiolites for 72 h, 150 h, and 192 h.

The Bifar mill was an ACM 10 – Micro Pull type, in which grinding is performed in an air stream whereby the particles impact on a rotating disk with a peripheral velocity of 120 m/s. The maximal rotation speed of the rotor was 720 rpm, the air flow rates were 750–1500 m<sup>3</sup>/h, and the classifier speed was 700–4000 rpm. The grinding of the sepiolites was performed with a mill capacity of 4 kg/h, since at this capacity satisfactory particle size distributions were obtained.

The sepiolites were wet-ground in aqueous medium. Aqueous suspensions of the sepiolites were used ranging from 2.5 to 10 wt.%. Grinding was performed in a pilot mill of the Fin-o-Matic 30 type, with a rotor diameter of 15 cm. During grinding, the particle size distribution was controlled by a Hegmans grindometer with a range of 0–100 µm. In some cases, repeated runs were performed in order to achieve the required particle size distribution.

All samples were ground until a maximal diameter of about 20  $\mu\text{m}$  was achieved, which was controlled by determination of the particle size distribution. By applying all of the mentioned grinding processes to all sepiolite samples (soft- and hard-consistency sepiolite from horizons 10 and 14), the effects of the different grinding methods on the same sepiolite sample, as well as the performance of different sepiolite samples, after treating by a particular grinding method, were analyzed.

Scanning electron microscope investigations were carried out with a JEOL 4-JSM-840a instrument, using magnifications of 3000–5000.

XRD data were obtained using a Philips PW 1710 Automated Powder Diffractometer System equipped with a TTK Temperature Attachment (Anton Paar K.G.) mounted on a Philips PW1820 Vertical Goniometer using  $\text{CuK}\alpha$  radiation,  $V = 35$  kV, and  $I = 50$  mA, at a scanning rate of  $1.5^\circ 2\theta/\text{min}$ . Before compacting in the TTK sample holder, the samples were powdered manually in an agate mortar. Recording of the XRD spectra was conducted at room temperature. Simultaneous TG-DTA analyses were performed on a Stanton Redcroft Thermal Analyzer in the temperature range of 20–950  $^\circ\text{C}$  under  $\text{N}_2$  flow at a heating rate of 20  $^\circ\text{C}/\text{min}$ .

Chemical analyses of the samples were made as before<sup>9</sup> followed by gravimetric determination of  $\text{SiO}_2$ . From the remaining liquid phase, the determinations of the cations were carried out by atomic absorption spectrophotometry using a Varian 775 instrument.

The surface areas of the sepiolite samples were determined using an Autosorb 6-Quantachrome, with  $\text{N}_2$ , and calculated by the BET method. Prior to the measurements, the samples were outgassed in a vacuum at 110  $^\circ\text{C}$  and 200  $^\circ\text{C}$ .

The infrared spectra were recorded on a Perkin Elmer 9836 spectrometer in the range of 4000–400  $\text{cm}^{-1}$ , using KBr pellets.

Viscosity measurements were carried out with a structural-rotational viscosimeter of the Rheotest 2 type using coaxial cylinders in the range of 5/9–243 rpm.

## RESULTS AND DISCUSSION

### *Scanning electron microscopy (SEM)*

The fibrous nature of the original, untreated sepiolite is apparent from the scanning electron micrographs (Figs. 1a and 2a) for the samples 1H14 and H10, respectively. The fibers were arranged in bundles, with lengths of 2–10  $\mu\text{m}$  for sample 1H14 and up to 15  $\mu\text{m}$  for sample 4H10.

After grinding in the ball mill, the particles had the appearance of irregular spheres, with diameters of up to 20  $\mu\text{m}$  (Fig. 1b). This shape resulted from the impacts of the grinding balls, as well as interparticular collisions, and suggests that the particles were amorphized to some extent. At greater magnification (5000 $\times$ ) it can be seen that the spheroid particles were agglomerates of a large number of randomly oriented needle-like particles.

After grinding in the air-stream mill, the sepiolite kept its fibrous micro-morphology (Fig. 1c), but the bundles were partly separated into free fibers.

Wet grinding of sepiolite caused the appearance of flakes (Figs. 1d and 2b), with lengths between 5 and 15  $\mu\text{m}$ , which roughly correspond to the fiber lengths in raw sepiolite. At higher enlargements it can be seen that the flakes are agglomerates of fibers. When these samples were suspended in water with vigorous mixing, the fibrous micromorphology of sepiolite is apparent even at lower magnification (Fig. 2c).

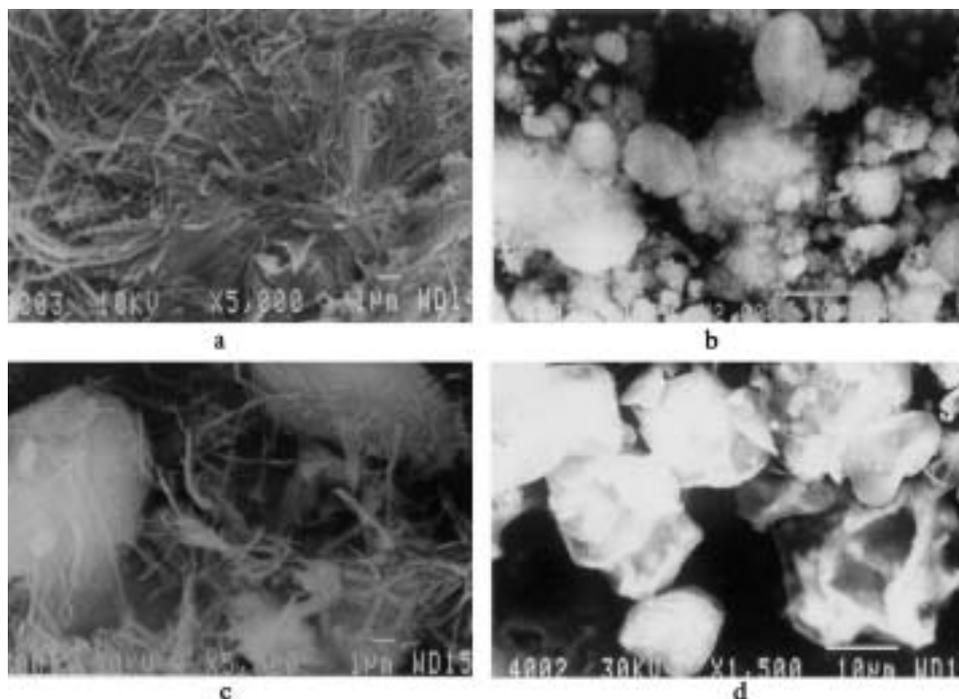


Fig. 1. Scanning electron photomicrographs of sample 1H14: a) original, b) ground in the ball mill, c) ground in the air-stream mill, and d) ground in the colloid mill.

#### *X-Ray diffraction (XRD)*

The diffractograms of the ground samples differ to a greater or lesser extent from those of the original sepiolite. Generally, when changes are noticeable, a reduction of the peak intensities occurs, with a broadening of the peaks and changes in the ratios of their intensities. By analysis of the diffractograms and by measuring the intensities of the 12 most intensive sepiolite peaks (and the magnesite peak at  $d = 0.274$  nm), it was found that the ratios of the intensities of different peaks change differently. The intensities of the peaks corresponding to the plane 080 ( $d = 0.337$  nm) for peak A and planes 112, 371 and 191 ( $d = 0.256$  nm) for peak B are of the same order of magnitude. Therefore, the ratio of the intensities of these peaks expressed in the form:  $R_I = I(A)/I(B)$  is suitable for analyzing the effects of grinding. Grinding has a clear influence on the change in the intensity of peak A and a negligible influence on the intensity of peak B. The problem here could be the heterogeneous nature of raw sepiolite (from the large number of experimental data, only those which indicate the general behavior of the investigated material are presented).

The diffractograms of sample 1H14 treated in a ball mill for different time periods (24–120 h) are presented in Fig. 3, and intensity ratios ( $R_I$ ) for samples 1H14 and 2H14 in Table II. From both Fig. 3 and Table II, it is clear that  $R_I$  decreases with increase duration of grinding.

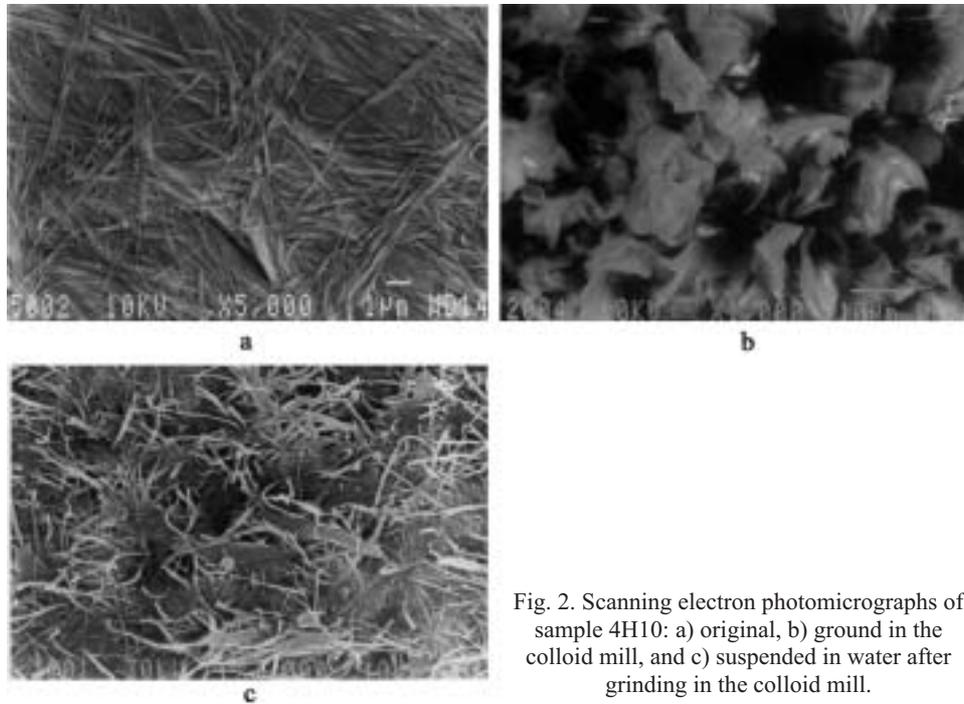


Fig. 2. Scanning electron photomicrographs of sample 4H10: a) original, b) ground in the colloid mill, and c) suspended in water after grinding in the colloid mill.

TABLE II. Relative ratios of the diffraction peak intensities in the positions  $d = 0.337$  nm (A) and  $d = 0.256$  nm (B) for samples 1H14 and 2H14 ground in the ball mill

Sample	$I(A)$	$I(B)$	$R_I = I(A)/I(B)$	$R_I$ (%)
1H14-0 h	10.95	6.60	1.66	100
1H14-24 h	8.90	7.70	1.16	70
1H14-48 h	10.70	6.80	1.57	95
1H14-72 h	9.25	6.75	1.37	83
1H14-96 h	9.10	6.95	1.31	79
1H14-120 h	7.50	6.90	1.09	66
2H14-0 h	8.00	4.80	1.67	100
2H14-24 h	7.10	5.30	1.34	80
2H14-72 h	5.95	4.70	1.27	76
2H14-150 h	4.35	3.65	1.19	71
2H14-190 h	4.85	4.90	1.02	61

There is no difference in the diffractograms of the raw 1H14 sample and of the same sample after grinding in the air-stream mill (Fig. 4), and since  $R_I$  of the treated sample is 98 % of  $R_I$  of the raw sample (Table III), it is clear that this type of grinding has a very small effect on the structural alterations.

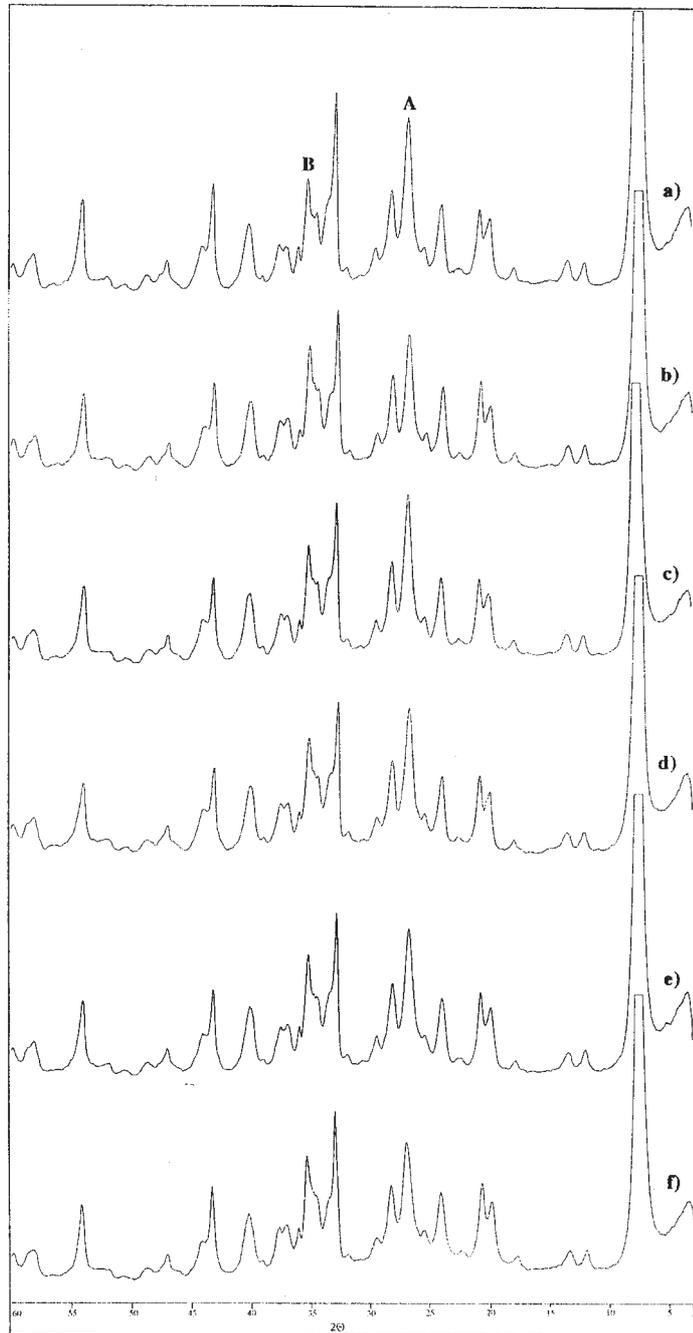


Fig. 3. X-Ray powder diffraction patterns: original sample 1H14 a), and the same sample ground in the ball mill for 24 h (b), 48 h (c), 72 h (d), 96 h (e) and 120 h (f).

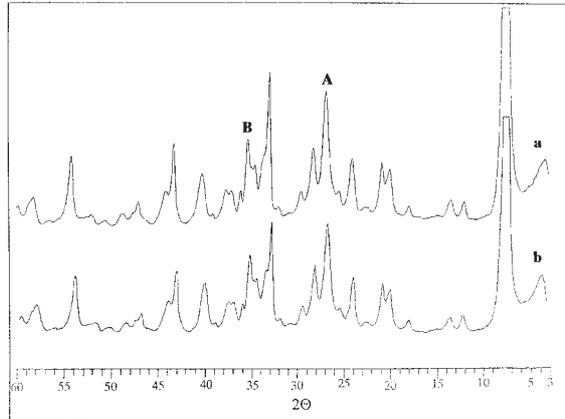


Fig. 4. X-Ray powder diffraction patterns of sample 1H14: a) original, b) ground in the air-stream mill.

TABLE III. Relative ratios of the diffraction peak intensities in the positions  $d = 0.337$  nm (A) and  $d = 0.256$  nm (B) for samples 1H14 ground in the air-stream mill

Sample	$I(A)$	$I(B)$	$R_1 = I(A)/I(B)$	$R_1$ (%)
1H14 original	10.95	6.60	1.66	100
1H14 ground	9.15	5.6	1.63	98

The X-ray patterns of raw and wet-ground sample 1H14 are presented in Fig. 5, and the corresponding ratio of peak intensities ( $R_1$ ) in Table IV.

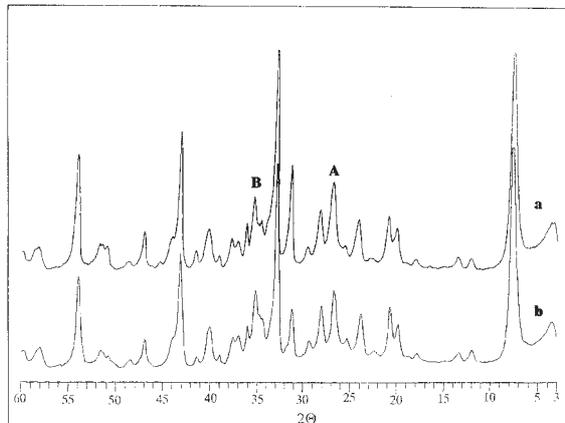


Fig. 5. X-Ray powder diffraction patterns of sample 1H14: a) original, b) ground in the colloid mill.

TABLE IV. Relative ratios of the diffraction peak intensities in the positions  $d = 0.337$  nm (A) and  $d = 0.256$  nm (B) for samples 1H14 ground in the colloid mill

Sample	$I(A)$	$I(B)$	$R_1 = I(A)/I(B)$	$R_1$ (%)
1H14 original	7.15	4.30	1.66	100
1H14 ground	5.85	5.30	1.10	66

The value of  $R_1$  for the treated sample is 66 % of  $R_1$  for the original sample, so it is clear that this type of grinding produces significant changes in the sepiolite structure.

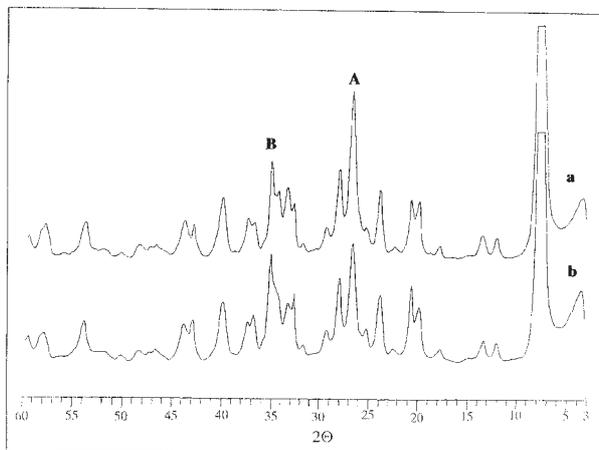


Fig. 6. X-Ray powder diffraction patterns of sample 2H10: a) original, b) ground in the colloid mill.

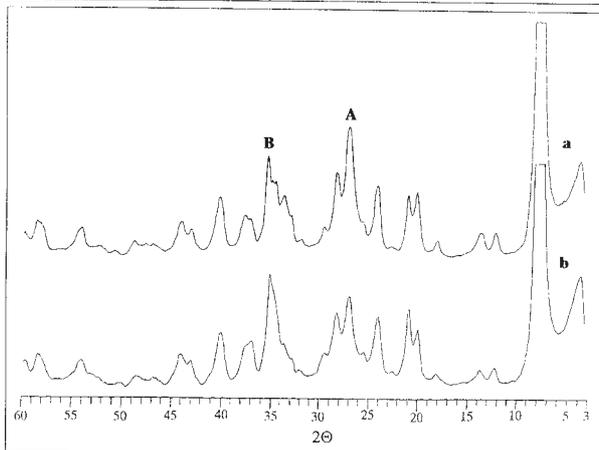


Fig. 7. X-Ray powder diffraction patterns of sample 4H10: a) original, b) ground in the colloid mill.

By comparing the influences of the different methods of grinding on the structural changes of sample 1H14, it is evident that only after 120 h of grinding in the ball mill does the value of  $R_I$  decrease to 66 % of the starting value, which corresponds to the value obtained by wet grinding, and that grinding in the air-stream mill has no influence on  $R_I$ .

The decrease of the  $R_I$  ratio for wet-ground sepiolite, as well as for sepiolite ground in the ball mill after very long treatment, can be attributed both to fiber separation and to partial amorphization, although this phenomenon is not yet completely clear.

The diffractograms of samples 2H10 and 4H10 before and after wet grinding are presented in Figs. 6 and 7.

The ratios of the peak intensities ( $R_I$ ) are generally higher for samples of a soft consistency than for samples of a hard one (Table V). For samples of a hard consistency, the  $R_I$  ratio can be as low as 36 %, although sample 5H10, where the  $R_I$  ratio is 111 %, is an exception.

In addition to the decrease in the peak intensities and their ratios, for samples with  $R_I < 0.60$ , the resolved peaks in the range of 34–36° ( $2\theta$ ) in many cases disappear and

their fusion into one peak occurs, with a maximum in the position of the diffraction planes 112, 371, 191 at  $35^\circ$  ( $2\Theta$ ).

TABLE V. Relative ratios of the diffraction peak intensities in the positions  $d = 0.337$  nm (A) and  $d = 0.256$  nm (B) for samples from horizon 10 ground in the colloid mill

	Sample	$I(A)$	$I(B)$	$R_I = I(A)/I(B)$	$R_I$ (%)
Original	1H10	17.30	7.60	2.28	100
	2H10	13.75	7.25	1.90	100
	3H10	13.20	7.95	1.66	100
	4H10	8.70	7.24	1.20	100
	5H10	3.85	7.20	0.53	100
	6H10	7.95	5.50	1.45	100
Ground	1H10	10.80	10.30	1.05	46
	2H10	9.30	8.30	1.12	59
	3H10	10.65	10.00	1.06	64
	4H10	5.050	8.80	0.57	48
	5H10	3.55	6.00	0.59	111
	6H10	4.05	7.80	0.52	36

Although treatment in the wet colloid mill has the same effects of changing the  $R_I$  ratios as prolonged treatment in the ball mill, for samples with a hard consistency from horizon 10, there is the additional effect of peak fusion in the  $2\Theta$  range of  $34\text{--}36^\circ$ .

#### *Infrared spectra*

The infrared spectra for the 1H14 sample before and after grinding in ball, air-stream, and colloid mills, show that the bands at  $3520\text{ cm}^{-1}$  and  $3400\text{ cm}^{-1}$ , corresponding to OH-stretching vibrations of bonded and zeolite water,<sup>10,11</sup> are not significantly changed. The same is valid for the OH-bending mode of vibrations for bonded and zeolite water in the range of  $1625\text{--}1640\text{ cm}^{-1}$ . Also, the Si–O combination bands at  $1210\text{ cm}^{-1}$ ,  $1100\text{ cm}^{-1}$ , and  $980\text{ cm}^{-1}$ , as well as peaks at  $1020\text{ cm}^{-1}$  and  $450\text{ cm}^{-1}$  (which correspond to Si–O–Si in plane vibrations), are not substantially changed (Fig. 8).

It can be concluded from this, that none of the employed grinding methods have any significant influence on the sepiolite structural changes in the 1H14 sample. The same was found for samples 2H10 and 4H10.

#### *BET Surface areas*

For all the samples, dry grinding has a very slight influence on the specific surface areas obtained by the BET method. Table VI presents the  $S_{\text{BET}}$  surfaces for sample 1H14 after grinding for various periods in the ball mill.

The specific surface areas at first slightly increase, but after 48 h of grinding start to decrease to some extent. This confirms our supposition that dry grinding in the ball or air-stream mill predominantly effects the separation of the sepiolite fibers.

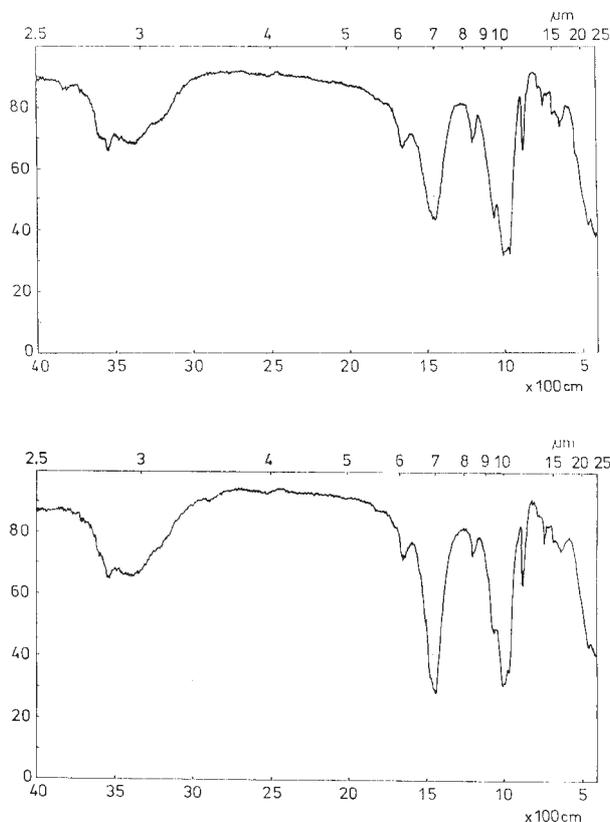


Fig. 8. IR spectra of sample 1H14: a) original and b) ground in the colloid mill.

TABLE VI. Variation of the specific surface area of sample 1H14 after grinding in the ball mill

Hours	0	24	48	72	96	120
$S_{\text{BET}}/(\text{m}^2/\text{g})$	243	253	248	244	242	236

In the case of wet grinding in the colloid mill, considerable changes in the specific surface areas were observed. For sample 1H14,  $S_{\text{BET}}$  decreases from the starting value of 243  $\text{m}^2/\text{g}$  to 140  $\text{m}^2/\text{g}$ , while for 4H10 this change is from 324  $\text{m}^2/\text{g}$  to 214  $\text{m}^2/\text{g}$ . These results show that the structure of the sepiolite fibers is changed by wet grinding. Probably the number of surface micro-pores decreases due to partial amorphization of sepiolite affecting the fibers surface.

#### *Thermogravimetric analysis*

Mass losses as a function of temperature for samples ground by the dry methods are essentially the same as for the raw samples. TG analysis reveals the presence of certain minor differences in the wet-ground samples where disruption of the sepiolite structure causes an increase in the number of free OH groups. This is presented for sample 4H1D in Fig. 9.

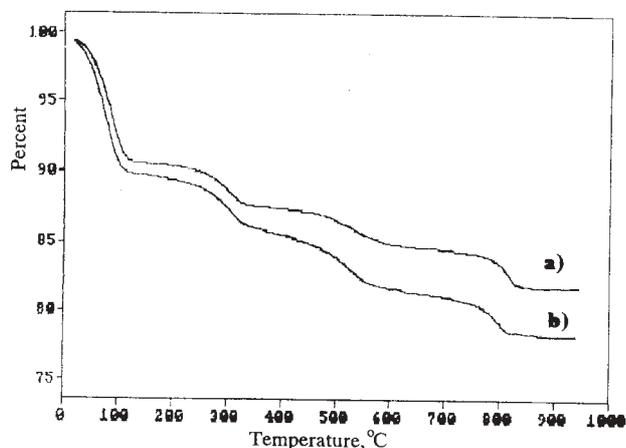


Fig. 9. TG plots of sample 4H10: a) original and b) ground in the colloid mill.

The weight loss for the treated sample is greater due to the loss of OH groups originating from the breakage of some Si–O–Si bonds. The final weight loss corresponding to structural OH groups and the accompanying endothermic peak on the DTA plot are observed at a lower temperature. The reason for this is that when the mass of the amorphous phase is increased, removal of the structural hydroxyls<sup>12</sup> and formation of the high-temperature phase are facilitated. The value of this weight loss remains unchanged during grinding, so it can be concluded that the ribbons within the fibers remain unaltered, although the fiber structure is changed, since the ribbons are either separated or broken lengthwise or crosswise.

#### *pH of sepiolite suspensions*

Dry grinding did not affect the pH values of 5 % aqueous suspensions of sepiolite sample 1H14, which remain at 6.7 even after 120 h of treatment in the ball mill. On the other hand, the pH values of the wet-ground samples 2H10 and 4H10 significantly increased, from 6.1 to 7.9 for sample 2H10 and from 6.9 to 8.2 for sample 4H10. The pH value increase is ascribed to partial amorphization, *i.e.*, to the formation of Mg(OH)<sub>2</sub>.

#### *Rheological characteristics of sepiolite suspensions*

Mineral thickeners and rheological additives based on sepiolite already have certain industrial uses. For this reason, the effect of the type of grinding on the rheological behavior of aqueous suspensions of sepiolite was examined. This involved the investigation of the changes in the particle surface and microfibrillar morphology of sepiolite upon grinding, as these properties are essential for determining the viscosities of sepiolite suspensions. The viscosity curves for aqueous suspensions of sepiolite 1H14 are presented in Fig. 10.

Curves (a) and (c) refer to a 4 % suspension of white sepiolite obtained by wet grinding in the colloid mill and dry grinding in the air-stream mill, respectively. The rheological characteristics of 16 % suspensions of dry-ground (air-stream mill) sepiolite are presented as curve (b). All the plots in Figs. 10 and 11 indicate pseudoplastic and thixotropic behavior of aqueous suspensions of sepiolite. These examples illustrate the predominant influence of the manner of grinding on the rheological characteristics of

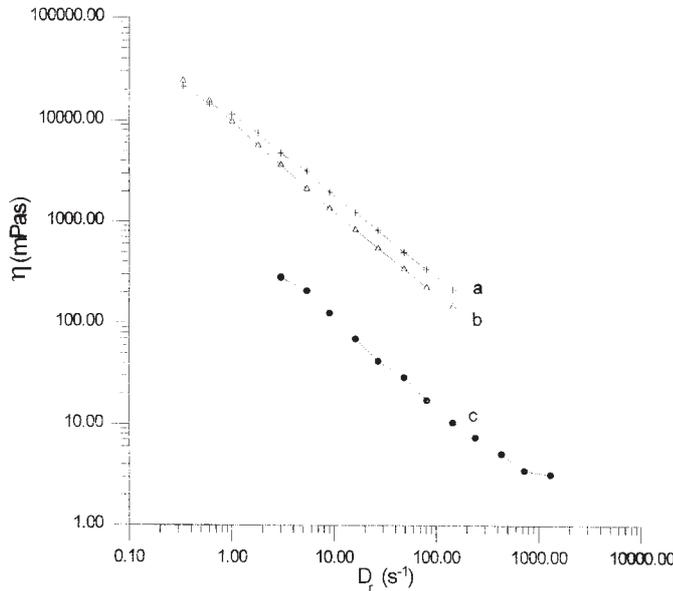


Fig. 10. Viscosity curves of aqueous suspensions of sepiolite 1H14: a) 4 % suspension of sepiolite ground in the colloid mill, b) 16 % suspension of sepiolite ground in the air-stream mill, and c) 4 % suspension of sepiolite ground in the air-stream mill.

aqueous suspensions of sepiolite, since the achieved mean particle size was approximately the same in all the employed grinding procedures.

The viscosity curves for the wet-ground samples from horizon 10 are presented in Fig. 11. All the curves refer to 4 % aqueous suspensions. The anomalous behaviour of 3H10, curve (e), ascribed to the large percentage of magnesite (about 40 %) in the sample, while in all other samples the content of magnesite as an associated mineral was 10–15 %.

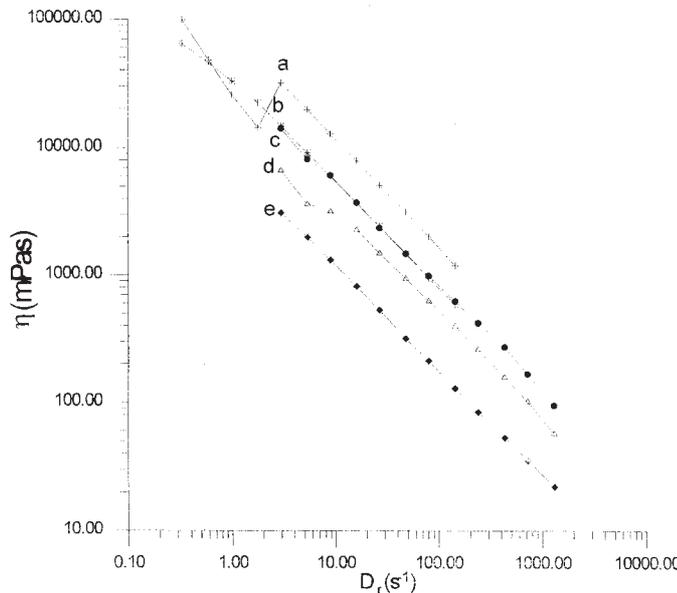


Fig. 11. Viscosity curves of 4 % aqueous suspensions of sepiolite samples from horizon 10: a) 4H10, b) 1H10, c) 6H10, d) 5H10, and e) 3H10.

The plots in Fig. 11 illustrate the fact that the starting consistency of the sepiolite has little influence on the rheological characteristics of its suspensions, but the content of the associated magnesite has a significant influence.

Our results show that the investigated sepiolites are much more stable to grinding than the Spanish ones.<sup>5</sup>

It is suggested that the changes in the diffractograms result from fiber separation inside the bundles or from a decrease in the crystallinity. We assign these changes in the diffractograms primarily to the separation of the fibers, which reduces the degree of crystal orientation along the *a* and *b* axes and especially lowers the intensities of the diffraction from the planes compared to the plane 080 (peak A).

Partial amorphisation of the samples during treatment also causes a decrease in the intensity and a broadening of the peaks in the diffractograms. This effect is added to that of fiber separation in the bundles. It is probable that the effect of fiber separation is so dominant that it masks the influence of amorphization on diffractograms, so it is impossible to determine the degree of fiber amorphization based only on the XRD analysis. The results of IR-analysis, measurements of SSA by BET, TG analysis, and pH values of suspensions confirm that amorphization was slight and that all the grinding methods produced samples with a dominantly preserved sepiolite structure, but SSA was affected by grinding.

It can be supposed that the degree of stability of the sepiolite structure to grinding is a function of its morphological characteristics. The fibers are united in primary bundles, which further form secondary bundles. Analysis of the results of all the employed methods confirms that amorphization is only partial and that the bulk of the mechanical energy of grinding is used for fibers separation. This suggests that the bundles are not simple mechanical aggregations of fibers but that rather strong forces hold the fibers together.

Fiber elasticity along the *c*-axes permits the wrapping of bundles into the flaked structures obtained as a result of wet grinding in the colloid mill. It is possible that this arrangement offers additional protection to the sepiolite structure against mechanical stresses during grinding, since the surface of the fibers inside the flakes are protected from direct erosion.

We assume that amorphization of sepiolite occurs mostly on the lateral sides of the bundles, where the ends of the fibers are destroyed, and by occasional breaking of the bundles along the *c*-axes.

A high degree of amorphization could occur only if the individual fibers which form the primary bundles were without the protection of higher order arrangements so that the grinding energy would predominantly effect amorphization.

The fibrous morphology of sepiolite is similar to that of chrysotile and other fiber-forming minerals, and its resistance to grinding treatment is therefore similar.<sup>4</sup>

#### CONCLUSIONS

Although the three applied types of sepiolite grinding produced particles of a roughly the same size (20  $\mu\text{m}$ ) their effects on the morphology of the sepiolite fiber bundles and on the surface of the fibers were completely different. Dry grinding in the air-stream mill resulted in separation of the bundles into sub-bundles and some break-

ing of the sub-bundles and fibers, without affecting the orientation of the fibers or their surfaces. Dry grinding in the ball mill, besides breaking the bundles, produced distortion of the fibers and their wrapping into balls, with randomly oriented fibers. Wet grinding in the colloid mill produced flakes, also with distorted and randomly oriented fibers, but the structure at the ends the fibers and, in the nano-scale on the surface of some fibers were affected.

Surface erosion, together with chemical actions of the aqueous medium, produce changes both in the “surface nano continuum” – thin layer of the fibers material where molecular forces exist, but, also, in the “two-dimensional nano-space” close to surface of the fibers.<sup>13</sup>

The BET surface decrease, since some surface nano-channels are destroyed, but the hydrophilicity of the surface of the fibers increases, producing significant changes in the rheological properties of aqueous suspensions of the ground sepiolite. Also, changes in the pH values of the suspensions indicate the formation of some  $Mg(OH)_2$  in the solution. The presence of  $Mg^{2+}$  ions in the solution and also the adsorption of  $Mg(OH)_2$  at the fibers surface could explain the changes in the rheological characteristics of the sepiolite suspensions.

The thermal behavior of wet grinded sepiolite changes, since, as the amorphous phase increases, the removal of structural hydroxyls and the formation of the high temperature phase are facilitated.

The results presented are valuable for future investigations and for the development of applications of Gollesh white sepiolite.

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#### ИЗВОД

#### ЕФЕКАТ МЛЕВЕЊА НА ФИЗИЧКО-ХЕМИЈСКЕ КАРАКТЕРИСТИКЕ БЕЛОГ СЕПОЛИТА СА ГОЛЕША

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Испитиван је утицај млевења на структурне и реолошке карактеристике водених суспензија белог сепиолита из рудника Магуре – Голеш (Јужна Србија). Узорци сепиолита чврсте и меке конзистенције млевени су у три типа млинова, куглични млин, млин са ваздушном струјом и колоидни млин. Ефекат млевења на узорке сепиолита испитиван је помоћу SEM, XRD, IR, TG и BET метода, а извршена је и хемијска анализа. Млевење генерално утиче на раздвајање сепиолитских влакана, а примећена је и парцијална аморфизација. Млевење битно мења привидну вискозност и реолошке карактеристике разблажених водених суспензија сепиолита, а на вискозност посебно утиче тип млевења. Површинска ерозија, заједно са хемијским деловањем водене средине, доводи до промена и у "површинском нано континууму" – танком слоју влакнастог

материјала, где постоје молекуларне силе а такође и у "двоструком нано простору" уз саму површину влакана. Термално понашање сепиолита подвргнутог мокром млевењу се мења, јер расте аморфна фаза, уклањају се структурне хидроксилне групе, па формирање високотемпературне фазе постаје лакше. Ови резултати су од значаја за будућа испитивања и примене белог сепиолита са Голеша.

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