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Obtaining High Temperature Stable Sepiolite via Optimization of Acid Treatment Conditions

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Abstract

Acid treatment of sepiolite under different molarity of HCl at various times was studied to find the best acid treatment conditions for obtaining anhydrous sepiolite without structural folding and with fully open and empty channels. It was found that high molarity (e.g., 3 M) acid treatment causes severe acid attack and heterogeneous magnesium dissolution from sepiolite particles. In contrast, the low molarity (e.g., 0.5 M) acid treatment dissolves magnesium homogeneously through the particles. This, in turn, affects the behavior of sepiolite upon calcination at 450ºC in that homogeneous magnesium dissolution gives better structural stability and consequently higher amount of open channels with the least amount of magnesium removal (25%). This is verified by rehydration behavior and specific surface area measurements after the calcination, where over 90% of the surface area could be preserved after the calcination of the low molarity acid-treated samples. No change in the morphology of sepiolite fibers was observed after acid treatment.

Keywords: Sepiolite, acid treatment, structural stability, rehydration, open channel

1. INTRODUCTION

Sepiolite is a fibrous hydrated magnesium silicate with a formula of $Si_{12}Mg_4O_{30}(OH)_4(H_2O)_4 \cdot 8H_2O$. Within the class of 2:1 type clay minerals, a magnesium octahedral layer is located between two tetrahedral silica layers. This structural unit expands continuously along the z direction forming a ribbon-like structure. The ribbon-like structures are linked by the inversion of the tetrahedra through Si–O–Si bonds; thus, the octahedral layer becomes discontinuous. Such structural arrangements cause microporous channels along the axis with a size of about 3.6 x10.6 Å and fibrous morphology [1]. These channels are filled by zeolitic water, which is bonded to the oxygen ions on the tetrahedral sheet or to other water molecules via hydrogen bonds. The water molecules completing the coordination of magnesium ions at the edges of the octahedral sheet are named coordination water [2]. When sepiolite is heated above 100ºC, the zeolitic water is eliminated, and all the zeolitic water and approximately half of the coordination water are lost by 300ºC. The remaining amount of the coordination water is lost between 300-500ºC without any other significant structural changes.

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During the last stage of dehydration, the structural water molecules are removed at temperatures $>700^{\circ}C$ [3]. Due to its structure, sepiolite has a relatively high specific surface area; values between 95 m^2/g and 400 m^2/g are reported depending on deposits [4–6]. However, the specific surface area is reduced dramatically when heated over $300^{\circ}C$, which was attributed to the folding of the crystal structure of sepiolite, eliminating the microporous channels [3, 7–9]. Acid treatment was effective in increasing the specific surface area and keeping it stable up to high temperatures [8, 10-11]. This was attributed to the dissolution of octahedrally coordinated magnesium ions, which creates new surfaces and prevents structural folding.

Several studies have been carried out to utilize sepiolite as an adsorbent [12-13], catalyst [14], catalyst support [9, 15-16], filtration membranes [17], and molecular sieves [18]. In catalyst and catalyst support applications of sepiolite, the relevant processes may require high temperatures of more than $300^{\circ}C$. In such cases, it is crucial that the sepiolite structure and specific surface area should be stable. Marosz et al. [14] observed a significantly increased catalytic efficiency of sepiolite for methanol and ethanol dehydration after acid treatment, compared to untreated sepiolite, and this was explained to be due to a significant increase in the BET-specific surface area, pore volume, and surface concentration of acid sites for the sepiolite after acid treatment.

It is also likely that the unfolded structure of sepiolite also plays an essential role in the improved efficiency due to the availability of the open channels for the catalytic reactions.

Sepiolite has also been used as a template to produce nanosized carbon/graphite fibers [19-20] and organic/inorganic hybrid materials [21]. In these cases, organic molecules were introduced into the channels of sepiolite by removing zeolitic water in the channels via heat treatment at around $150^{\circ}C$.

However, Kuang et al. [21] observed that 50% of the organic molecules were lost together with the removal of the coordination water when temperature is increased, reducing the capacity of the sepiolite channels. Valentín et al. [22] suggested that if the coordination water is removed without the structural folding, the capacity of sepiolite for these applications could be improved. In order to demonstrate this, they acid-treated sepiolite with 3 M HCl at $60^{\circ}C$ for different times between 5 to 70 minutes to find suitable conditions to dissolve just enough magnesium ions in order to keep the structure stable without folding at high temperatures, claiming that an excessive magnesium removal would degrade the sepiolite structure.

They observed that 40 min. treatment time removed all of the magnesium ions from the sepiolite structure. While 5 min. treatment time was not enough to prevent folding, 20 min. treatment time was found to be sufficient to keep the structure stable, even after calcination at $550^{\circ}C$. Valentín et al. [22] attributed this anomalous thermal behavior to the coexistence of the channel structure coming from sepiolite crystal and silica generated by partial destruction of the sepiolite structure in a singular fiber, in which silica is responsible for the thermal stabilization of the structure.

Although Valentín et al. [22] aimed to develop anhydrous sepiolite with open nanometric channels with minimal changes in its structure and chemistry, the high molarity (3 M) acid treatment they used may not be the ideal condition for this since molarity affects the kinetics of dissolution significantly [4, 23]. Therefore, this study aims to find the best acid treatment condition concerning molarity to get anhydrous sepiolite with a maximum amount of open channels to take full advantage of this mineral in prospected applications such as catalyst, catalyst support, hybrid materials, nano clay composites, etc. As far as the author knows, there is no such study in the literature on this line.

2. EXPERIMENTAL

Oncu Madencilik supplied sepiolite minerals from the Eskisehir region in Turkey as 3-5 cm fragments. The fragments were ground into a powder by using a laboratory mortar grinder (Fritsch Pulverisette 2, Idar-Oberstein/Germany). The particle size of the crushed powder was measured by light scattering (Mastersizer 2000, Malvern, UK). This powder was acid treated in a hydrochloric acid (HCl) solution at different molarities at 60°C under reflux conditions for various times by using 5 g sepiolite powder dispersed in a 30 ml solution.

The acid treatment conditions are given in Table 1. Although HCl is used in this study, it should be noted that types of acid does not affect the dissolution kinetics significantly [4]. At a constant temperature, since the acid concentration was reported to be the most effective parameter for the dissolution, the treatment time was reduced with increasing molarity in a manner to keep the MgO dissolution at acceptable levels [23].

Table 1 Experimental parameters for acid treatment.

HCl Concentration (M)	Time (min)
0.5	20, 30, 40, 50, 70
1	20, 30, 35, 40
2	5, 10, 15, 20, 25, 30
3	2, 5, 10

The acid-treated samples were designated by considering molarity and treatment time in that ASEP-0.5M/70 indicates acid treatment in 0.5 M HCl solution for 70 minutes. Then, the samples were washed several times with deionized water until free from chlorine ions. Parts of the samples were calcined at 450°C for 1h in a laboratory muffle furnace under ambient conditions. Chemical and mineralogical analysis before and after acid treatment were carried out by X-ray fluorescence (XRF, Rigaku ZSX Primus, Tokyo/Japan) and by X-ray diffraction (XRD,

Rigaku Miniflex 600, Tokyo/Japan), respectively. Samples for XRF analysis were prepared by melting them using LiB_4 at 10:1 LiB_4:powder mass ratio, followed by casting into glass tablets.

The XRF results were reported as oxides present in the samples without considering loss-on-ignition. XRD analysis was made by using Cu Kα radiation at a scanning speed of 1o 2θ/min between 4°- 35° 2θ. BET-specific surface areas were measured by N_2 gas adsorption (Quantachrome-NOVA touch LX, FL, USA) after degassing the samples at 200°C for 10h before the measurement. Thermogravimetry (TG, STA409 Netzsch, Selb/Germany) measurements were taken with a heating rate of 10°C/min under flowing air. Microstructural examinations were carried out by scanning electron microscopy (SEM, Zeiss Supra VP, Germany) using secondary electron imaging mode. Before examinations, samples were coated with a thin layer of gold to prevent charging.

3. RESULT AND DISCUSSION

The average particle size of the ground sepiolite powder was 10 μm with 23 μm D_{90} and 3 μm D_{10} values. Considering the nanometric size of sepiolite fibers, the particle size data represents the agglomerate size of the sepiolite fibers. The chemical analysis of the sepiolite powder is given in Table 2.

The powder mainly consists of SiO_2 and MgO, but considerable amounts of Al_2O_3 and Fe_2O_3 are also present with some impurities, including CaO, K_2O, and Cr_2O_3. Fig. 1 shows the amount of MgO remaining in the sepiolite powder with the acid treatment time at different molarities between 0.5-3 M, as measured by XRF. Based on a previous study, the maximum acid concentration was kept at 3 M [22]. As seen in Fig. 1, increasing acid concentration increases the dissolution rate of magnesium ions significantly.

Therefore, the acid treatment time was significantly reduced as the molarity

increases to avoid excessive magnesium dissolution. The dissolution kinetics of sepiolite was studied by several authors [4, 23-24] who observed that the dissolution process was controlled by product layer diffusion with first-order reaction and reaction temperature, acid concentration, solid-to-liquid ratio and particle size were rate-controlling parameters. It is not the intention of this paper to study the dissolution kinetics of sepiolite but, keeping the other parameters the same, significant differences in the dissolution rate with the acid concentration (molarity), as seen in Fig. 1, should also have an impact on the homogeneity of the magnesium removal process from the sepiolite particles and associated structural stability as a result of the dissolution.

Then, the question arises as to what the optimum dissolution conditions would be to have a minimum structural disorder and chemical modification to sepiolite (i.e., minimum magnesium removal) and maximum stability upon calcination.
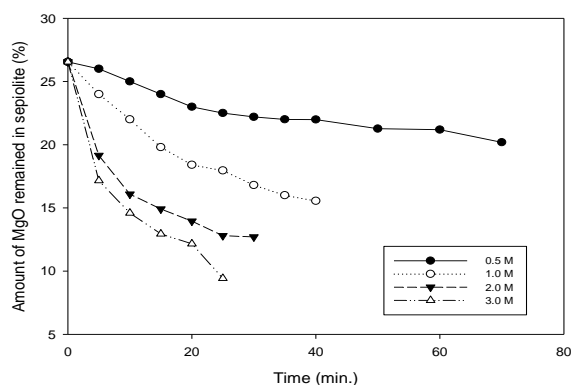

Figure 1 Dissolution behavior of MgO from sepiolite with time under different acid concentration

XRD spectra of untreated and the acid-treated sepiolite at different molarities for different times are given in Fig. 2a-g, while those after calcination at 450°C are shown in Fig. 3a-g.
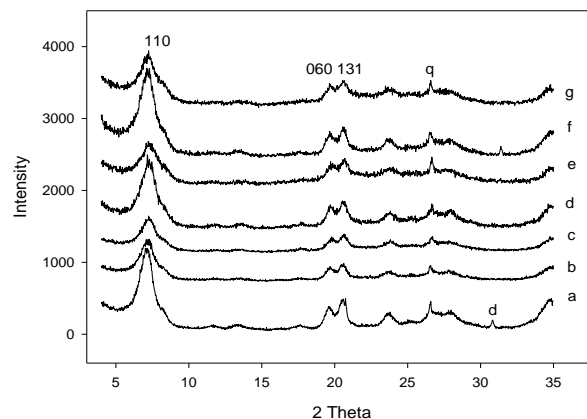

Figure 2 XRD spectra of seiolite: (a) as-received, (b) ASEP 0.5M/70, (c) ASEP 1M/30, (d) ASEP 2M/5, (e) ASEP 2M/10, (e) ASEP 3M/5 and (g) ASEP 3M/10. (q=quartz; d= dolomite. Only 110, 060 and 131 peaks of sepiolite are indicated

For clarity, not all the acid-treated samples' XRD spectra studied are shown in Fig. 2 & Fig. 3. However, the selected ones are based on the increased molarity-reduced treatment time criteria to avoid excessive magnesium dissolution from sepiolite.
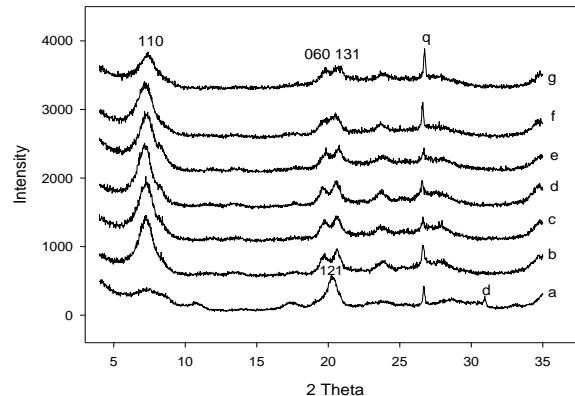

Figure 3 XRD spectra of sepiolite after calcination at 450°C: (a) as-received, (b) ASEP 0.5M/70, (c) ASEP 1M/30, (d) ASEP 2M/5, (e) ASEP 2M/10, (e) ASEP 3M/5 and (g) ASEP 3M/10 q=quartz, d= dolomite. Only 110, 060 and 131 peaks of sepiolite and 121 peak of anhydrous sepiolite are indicated

As seen in Fig.2a, the main phase in the untreated sepiolite is sepiolite with a trace amount of quartz (26.6° 2θ) and dolomite (about 31° 2θ). Knowing that 1% CaO present in the chemical analysis is due to dolomite, which equates to about 3% dolomite, we can

estimate that the as-received sepiolite contains at least 85% sepiolite phase based on the amount of MgO present (Table 2).

Calcination at 450°C causes collapsing of the sepiolite crystal structure forming anhydrous sepiolite (Fig. 3a). Structural collapsing (folding) of sepiolite upon calcination at temperatures over 300°C has been addressed by various authors, and detailed discussion about the reasons for it could be found elsewhere [7-8, 22]. It causes the channels in sepiolite to be lost with a consequent reduction in specific surface area, and this precludes the usefulness of sepiolite in applications that have been addressed in Section 1 of this paper.

Table 2 Chemical analyses of as-received sepiolite on the basis of oxides. Note that loss-on-ignition is not taken into consideration

	SiO_2	MgO	Al_2O_3	Fe_2O_3	CaO	K_2O	Cr_2O_3
wt.%	63.9	26.6	4.5	3.1	1.0	0.6	0.3

XRD spectra of the acid-treated sepiolites given in Fig. 2b-2g show that under the given conditions of time and molarity of the acid treatment, the samples contain sepiolite as the main phase with a small amount of quartz as an impurity mineral. Compared to the untreated sample, a noticeable effect of the acid treatment is that sepiolite peaks are getting somewhat less sharp, which may be attributed to structural disordering and the reduced scattering due to removing magnesium ions from the structure.

In addition, an increased background, particularly between 20-30° 2θ, is evident for 2M and 3M acid-treated samples (Fig. 2e-g), and this may be due to the formation of a silica-rich amorphous phase [1, 22]. XRD spectra of the acid-treated sepiolites after calcination at 450°C (Fig. 3b-g) resemble those of the uncalcined samples (Fig. 2b-g), indicating that acid treatment stabilized sepiolite structure upon calcination, preventing folding of the structure.
However, a detailed examination of the calcined sample's spectra indicates a noticeable difference in the spectra of the samples treated at 3 M. Namely, two well-defined peaks of 060 and 131 planes of sepiolite are not well-defined in 3 M acid-treated samples. This appears to be due to the presence of anhydrous sepiolite (folded structure) because the peak of 121 plane of anhydrous sepiolite (Fig. 3a) is located between 060 and 131 planes of sepiolite, making all three peaks less defined. Although not mentioned by them, a similar overlap is also evident in the study carried out by Valentín et al. [22], who used a 3 M acid treatment concentration. These observations suggest that 3 M acid treatment keeps the structure of a certain amount of sepiolite stable, but some are folded.

In order to get a further insight into the stability of the channel structure of sepiolite upon calcination, changes in specific surface areas of the selected acid-treated samples before and after calcination were compared with the untreated sepiolite (Table 3). The selected samples were ASEP 1M/30 and ASEP 3M/5 as they had almost the same amount of MgO removal, 9.4 and 9.3%, respectively. The untreated sepiolite had 478 m^2/g specific surface area, close to the values reported for sepiolites from Eskisehir region [4, 6]. The specific surface area of the untreated sepiolite was reduced to 286 m^2/g after the calcination, representing 40% reduction in its specific surface area.

It is known that untreated sepiolites experience a rather significant reduction in their specific surface areas due to the destruction of the channel structure as a result of folding of the structure, and a similar rate of reduction in the specific surface area upon calcination at 500°C was also reported by

Balci [3]. ASEP 1M/30 and ASEP 3M/5 samples did not show a significant change in the specific surface area, being 455 and 480 m²/g, respectively.

Esteban-Cubillo et al. [1] reported that the specific surface area does not change due to acid treatment (1 M) until up to about a third of the magnesium ions are dissolved while an increase in the specific surface area is noted upon further dissolution. 9.4 and 9.3% MgO removal from sepiolite correspond to about the dissolution of a third of the magnesium ions, considering the presence of 26.6% MgO in the as-received state (Table 1) and confirm the finding of Esteban-Cubillo et al. [1].

When the specific surface areas upon calcination at 450ºC are compared, ASEP 3M/5 sample lost 25% of its specific surface area while ASEP 1M/30 sample lost only 7% (Table 3).

Table 3 Changes in specific surface area of sepiolite after acid treatment and calcination at 450ºC

Samples	Uncalcined	450ºC calcined	Reduction in surface area due to calcination (%)
Untreated Sepiolite	478	286	60
ASEP 3M/5	480	360	25
ASEP 1M/30	455	426	7

Even though the amounts of magnesium dissolution were almost the same for both of the samples, the differences in the reduction of the specific surface area give a clear indication that the acid treatment under 3 M is not as good as that under 1 M as far as the structural stability at higher temperatures is concerned. This observation is also supported by the XRD spectra of 3 M samples, as discussed above.

Considering the similar XRD spectra of the calcined samples acid treated between 0.5 and 2 M, they may be regarded as similar with respect to structural stability. Nevertheless, rehydration of the calcined samples was carried out in order to find out whether there were any differences between the stabilized sepiolite samples treated at different molarities concerning the number of open channels, which may not be resolvable by XRD.

Valentin et al. (2007) suggested that rehydration behavior would give us information about the amount of stable open channels of sepiolite since water molecules would refill the channels under humid conditions as zeolitic water. The calcined samples were kept in a closed humid environment for 1 week for the rehydration experiments. They were then kept in an oven at 70ºC for 3 days to remove physical water before TG analysis because the rehydrated samples contained a large amount of physical water (> 20 wt.%) due to their high specific surface areas. TG analysis of the rehydrated samples is given in Fig. 4.
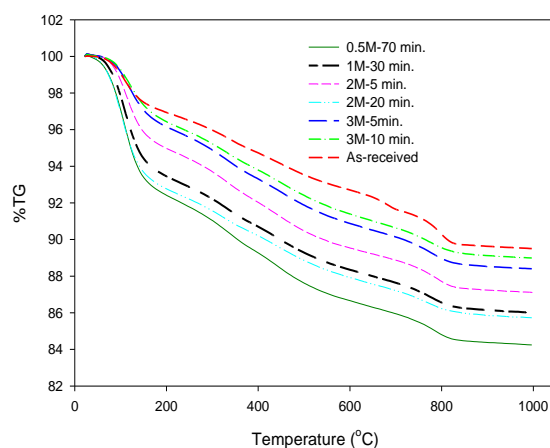

Figure 4 Thermogravimetric analysis of acid treated sepiolite after calcination and rehydration

Since the samples were dried at 70ºC for 3 days before TG analysis, most of the mass losses seen in the TG graphs up to 250ºC could mainly be correlated to zeolitic water in the channels. However, it should also be stressed that some amount of this water

should be related to fine pores in the sepiolite mineral particles [3]. Nevertheless, it is not expected that the acid treatment at different molarities carried out in this study and the subsequent heat treatment condition should not affect the structure of the fine pores since no significant change in the specific surface area was observed under given acid treatment conditions, as seen in Table 3.

Therefore, the differences in mass losses between the samples up to 250ºC in the TG graphs in Fig. 4 reflect the differences in the mass gain due to the rehydration and could well be compared and correlated to the number of open channels. They are given in Table 4 with the amounts of MgO remaining in the sepiolite after the acid treatment and of MgO dissolved. The amounts of MgO dissolved are simply derived by subtracting the amount of MgO remaining in the acid-treated sepiolite from that in the as-received sepiolite. As seen in Table 4, the untreated sepiolite is the least rehydrated one, only 3.5%. This is because the sepiolite structure collapses at 450ºC, as discussed above (compare Fig. 2a & Fig. 3a), and consequently, the channels are lost. Assuming that no open channels remained in the calcined untreated sepiolite, we can mainly assign its 3.5% mass gain due to the rehydration to water adsorption in porosities in the sepiolite mineral particles.

Therefore, we can gain information about the extent of open channels remaining after calcination by subtracting 3.5% from the mass gain data of the acid-treated samples in Table 4. The higher the mass gain, the higher the number of open channels. With this respect, ASEP 0.5M/70 samples could have the maximum open channels, closely followed by ASEP 2M/20 and ASEP 1M/30 samples. ASEP 3M/5 and ASEP 3M/10 samples had the least open channels.

One of the main observations drawn from Table 4 is that there is no correlation between the amount of MgO removed from sepiolite and the amount of mass gain achieved, i.e., of

open channels. For example, 0.5M-70 min. and 2M-20 min. acid treatments remove 6.6% and 12.6% MgO but achieve similar mass gain as 8.1% and 7.8%, respectively. In contrast, 1M-30 min. and 3M-5 min. acid treatment removes a similar amount of MgO (9.4% and 9.3%, respectively), but their mass gains are very different, 7% and 4.3%, respectively.

These observations indicate that although MgO removal is necessary for the structural stabilization of sepiolite, there is no relation between the amount of MgO removed and the extent of structural stability, at least under the experimental conditions of this study.

From this perspective, it can also be stated that where and how MgO is removed from sepiolite is important for structural stability or crystal folding upon calcination. The sepiolite crystal is known to be folded by rotation of the fibers on axes through the Si–O–Si edge bonds that join the fiber units along the length of their edges.

Table 4 Weight losses observed due to zeolitic water as a result of rehydration in the acid treated samples and corresponding amount of MgO remained and of MgO removed after the acid treatment

Samples	Weight loss up to 250ºC after rehydration (wt.%)	Amount of MgO left in sepiolite after acid treatment (wt.%)	Amount of MgO removed from sepiolite (wt.%)
Untreated	3.5	26.6	0
ASEP 0.5M/70	8.1	20.0	6.6
ASEP 1M/30	7.0	17.2	9.4
ASEP 2M/5	5.5	19.2	7.4
ASEP 2M/20	7.8	14.0	12.6
ASEP 3M/5	4.3	17.2	9.3
ASEP 3M/10	4.0	14.5	12.1

Valentín et al. (2007) stressed that acid treatment (3M-20 min. HCl) creates silica sheets by removing MgO from octahedral layers totally and that a channel structure coming from the sepiolite crystal and a sheet structure corresponding to silica coexist in a singular fiber. When enough silica is formed (or MgO is removed), the ribbons conforming to the structure become not interconnected due to the loss of some of their octahedral sheet forming a layer structure, which diminishes the torque necessary to fold the structure. However, they also stated that the mechanism could be more complex such that magnesium ions located close to the channels are preferentially extracted compared to those located in the center of the block, in which case silica sheets do not form.

This mechanism has also been put forward by Esteban-Cubillo [1], who claimed that acid treatment (1 M concentration) dissolves mainly the magnesium cations located at edges of the octahedral layers up to the magnesium leaching fraction of 0.33 (corresponding to 5 wt.% Mg or 8.3% MgO dissolution).

Based on the observations in this study and on the literature studies given above, the unexpected result of having no relation between the amount of MgO removed and the extent of structural stability of sepiolite could be explained by MgO dissolution kinetics from the sepiolite structure, which was reported to be controlled by diffusion through the reaction layer [4, 23-24]. At a constant temperature, acid concentration was reported to be the most influential parameter for the dissolution [23], as also noticeable in Fig. 1. It appears that short treatment times at a high acid concentration may lead to heterogeneous magnesium dissolution such that severe acid attack dissolves more magnesium from the external surfaces of sepiolite particles where the acid meets first than from the inner regions.

On the other hand, a mild acid attack at a low acid concentration enables the acid to reach through the sepiolite particles without excessive magnesium dissolution at the external surfaces and remove magnesium homogeneously through the particles. This is schematically illustrated in Fig. 5 for the cases of ASEP 0.5M/70 and ASEP 3M/5 samples. Under such circumstances, sepiolite treated at a high acid concentration would experience more structural collapse in the inner regions after calcination due to less magnesium dissolution, which is supported by the appearance of the peak of 121 plane of anhydrous sepiolite, located between the peaks of 060 and 131 planes of sepiolite in 3 M acid treated samples' XRD spectra (Fig. 3f-g). On the other hand, outer regions would be more structurally disordered due to more magnesium dissolution, which is supported by the occurrence of amorphous silica in their XRD spectra (Fig. 2f-g). In contrast, sepiolite treated at a low acid concentration would remain stable thoroughly after the calcination because of homogeneous magnesium ion dissolution through the particles. Such differences in the dissolution behavior and related structural behavior upon calcination can then explain the differences in the mass gain attained due to zeolitic water during the rehydration, seen in Fig. 4.
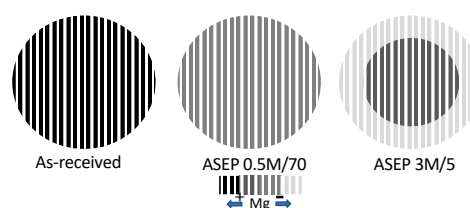

Figure 5 Schematic drawing of sepiolite particles showing the effect of acid concentration on the distribution of magnesium thorough the particle

Based on the above observations and explanations, one can now make an assumption as to where magnesium ions are mainly dissolved from the structure and its relevance to the structural stability of sepiolite. If ASEP 0.5M/70 samples are taken as an example, 6.6% MgO is removed from the structure, corresponding to about 25% of MgO in the octahedral layer. Considering the presence of 8 magnesium atoms in the

octahedral layer across the ribbon-like structure, we can postulate that 6.6% MgO removal corresponds mainly to the dissolution of magnesium ions from edges of the octahedral layer near the channel walls, as also proposed by Esteban-Cubillo [1].
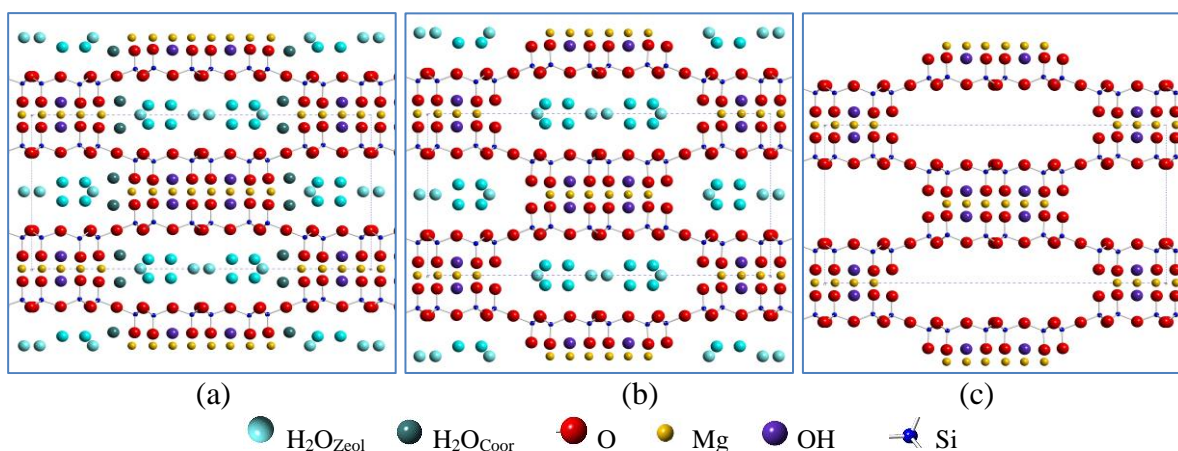

Figure 6 Crystal structure view of sepiolite (a) before acid treatment, (b) after acid treatment and (c) after acid treatment and calcination (For coloured image, please refer to web version)

This is schematically illustrated in Fig. 6 which shows the structure of untreated sepiolite, ASEP 0.5M/70 before and after calcination at 450°C. Likewise, 12.6% MgO removed from ASEP 2M/20 sample corresponds to about half of MgO present in the as-received sepiolite and thus the dissolution of magnesium ions from the edge of the octahedral layer near the channel walls as well as the next layer to the edge. Both of these samples have similar amounts of rehydration despite different MgO content. This indicates that as long as magnesium ions are removed categorically in an order from the edges of the octahedral positions to the inner locations of the octahedral layer across the ribbon-like structure, then the channel structure could be kept stable at high temperatures, even up to the removal of half of the magnesium ions. These observations lead us to conclude that if the sepiolite structure is to be kept stable without folding upon calcination and at the same time with the least structural modification (e.g., the least amount of magnesium removal), a rather mild acid treatment conditions (e.g., 0.5 M) should be used to dissolve magnesium.

Having seen that ASEP 0.5M/70 sample seems to be the best stabilized one according to the phase analysis and rehydration behavior, this sample was further examined by comparing TG analysis after the acid treatment and after the acid treatment followed by the calcination and rehydration (Fig. 7).
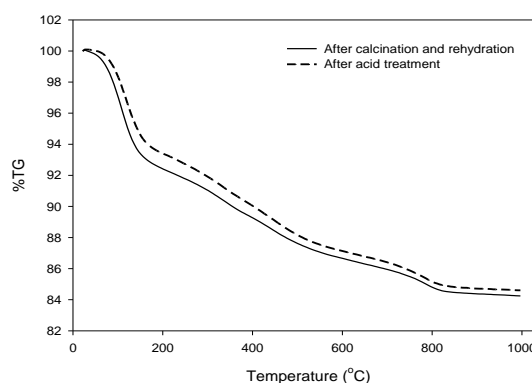

Figure 7 Comparison of thermogravimetric analysis of ASEP 0.5M/70 acid treated sample right after acid treatment and after calcination and rehydration

As seen in Fig. 7, TG graphs are almost identical indicating that ASEP 0.5M/70 sample remains stable with almost no change in its structure upon calcination at 450°C and

the channels could be refilled totally by water molecules.

Fig. 8 shows secondary electron image SEM microstructures of the as-received and 0.5 M acid-treated sepiolite for 70 min. In general, the fibrous morphology of sepiolite changes into a coarse particle structure due to the aggregation of silica units forming when the majority of magnesium ions are removed from the structure by acid treatment [1, 10-11].
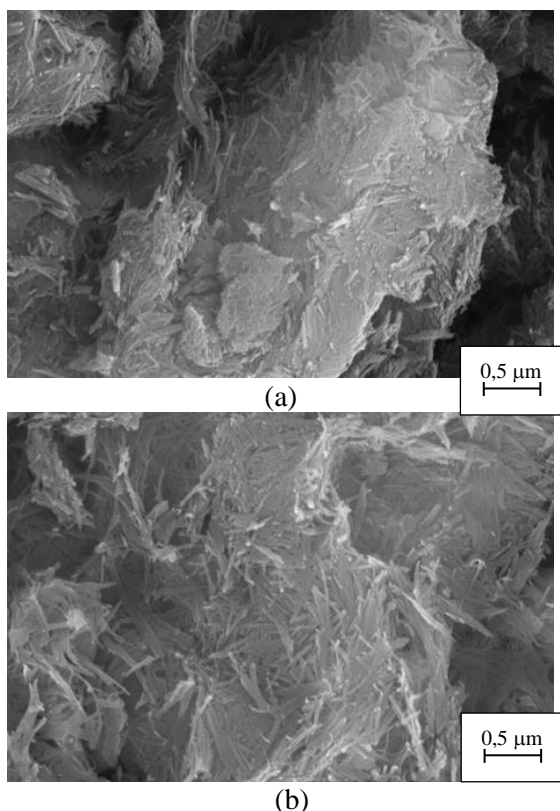

Figure 8 Scanning electron microscopy images of (a) as-received sepiolite and (b) ASEP 0.5M/70

However, it is seen that there is no change in the morphology of sepiolite fibers after the acid treatment. This is probably due to the lower amount of magnesium dissolution, which does not cause the formation of silica units.

4. CONCLUSIONS

This study investigated the best acid treatment condition to produce sepiolite with a minimal structural disorder that is stable at temperatures up to 450°C. Excellent stability upon calcination was achieved when sepiolite was treated under mild acid treatment conditions (e.g., 0.5 M). Removal of about 25% of magnesium ions from the sepiolite structure, which roughly corresponds to magnesium ions located at the octahedral positions close to the channel surface, was found to be sufficient for high temperature stability as long as the mild acid treatment conditions prevail.

It has been shown for the first time that anhydrous sepiolite with total high temperature structural stability and open channels could be produced. Such a sepiolite structure may find potential applications in high-temperature catalysis and catalyst support applications, as well as in hybrid materials and nanoclay composites development.

The Declaration of Conflict of Interest/ Common Interest
No conflict of interest or common interest has been declared by the authors.

The Declaration of Ethics Committee Approval
This study does not require ethics committee permission or any special permission.

The Declaration of Research and Publication Ethics
The authors of the paper declare that they comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification on the data collected. In addition, they declare that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic

publication environment other than Sakarya University ournal of Science.

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