Purification of sepiolite and its dissolution in hydrochloric acid and sodium Acetate-Acetic acid buffer

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Article Info.	ABSTRACT
	The associated impurities with sepiolite, mainly palygorskite, quartz, and dolomite, reduce the industrial quality of this mineral. The objectives of this study were to i) investigate the efficiency of particle size separation for purifying sepiolite and ii) determine the kinetic dissolution of sepiolite in HCl and sodium acetate - acetic acid buffer (SAB). Bulk sepiolite was provided from a mine located around the city of Fairman, in northeastern Iran. Three particle size fractions of 20-50, <20. and <2 µm were separated from the bulk sepiolite. The XRD results showed that the particle size separation method has a great impact on reducing the amount of quartz as well as the complete elimination of dolomite in particles <2 µm and palygorskite in 20-50 µm fractions. The <20 µm fraction composed ~70% of the sample which it's impurities considerably decreased; Therefore, it is a cost-effective fraction for industrial use. Therefore, it is a simple and low-cost physical procedure, without applying chemical reagents to achieve suitable purified sepiolite. The results of dissolution kinetic showed that the data had the best fit with the rate equation of $Kt=[(1-(1-X)^{1/2})^2]$. Increasing acid concentration, temperature, and time had a direct relation, while the solid to liquid ratio had an inverse relation with the apparent rate constant. This trend was observed for SAB, too. Dissolution of sepiolite in HCl was higher than that of SAB. Acid concentration and temperature were the most effective factors on sepiolite dissolution.

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1. Introduction

Sepiolite with the largest abundant between latitudes of 30° to 40° in the northern and southern hemispheres is a common fibrous clay mineral in arid regions (Hojati and Khademi, 2011; Abbaslou et al., 2023; Daridia et al., 2016; Kadir et al., 2016). It is a 2:1 layered silicate mineral, which consists of two tetrahedral sheets that surround a Mg-rich octahedral sheet in the center. The ideal structural formula of this mineral is $Mg_8Si1_2O_{30}(OH)_4(H_2O)4.8H_2O$. High Porosity, and specific surface area, strong absorptivity and specific rheological characteristics are the main properties of sepiolite. The most important usages of this mineral are in drilling fluid, dyes, liquid detergents, glues, automotive paints, cosmetics and sanitary ware, and floor absorbents (Abbaslou et al., 2023, Murray, 2007).

Clay minerals are rarely found in high purity levels in nature and are commonly associated with other clay and non-clay minerals. Purification should be done in such a way that in addition to the efficient removal of impurities, the main mineral structure and characteristics do not change. Chemical, physical, or a combination of these methods are used to obtain acceptable purity. Soluble salts, carbonates, organic compounds as well as aluminum, titanium, aluminum, iron, and manganese compounds are dissolved by using different chemical treatments (Jacqueline et al., 2005; Ha Thuc, 2010). One of the main impurities associated with sepiolite is carbonates minerals, especially dolomite. Inukai et al. (1994) used HCl to remove dolomite from Yenidogan sepiolite. When the rate of acid addition is not controlled, due to the rapid decrease in pH, a competitive reaction of HCl with dolomite and sepiolite occurs, resulting in magnesium depletion and the decomposition of sepiolite and the formation of amorphous silica. However, with a controlled addition of HCl in a sample of 0.1 $MgCl_2$ solution and adjusting the pH of the suspension above 6, the possibility of successful purification of the sepiolite was provided. The content of the sepiolite increased from 47% to 92% and the content of dolomite decreased from 52 to 1 %.

Impurities usually accumulate in different particle size fractions. Jacqueline et al. (2005) have proposed a simple method for clay purifying using sedimentation and low-speed centrifugation. Ha Thuc et al. (2010) used sedimentation, centrifugation and ultrasonic waves and reported the efficiency the removing the quartz. Zhou et al. (2016) used the sedimentation method to remove quartz and HCl combined by microwave radiation to remove carbonates.

The largest reservoirs of sepiolite are located in Spain which is the most important exporter of this mineral in the world (Murry, 2007). There is limited information about the presence of sepiolite in Iran. Khademi and Mermut (1998) and Hojati et al., (2011) reported the presence a trace amount of sepiolite in Oligocene-Miocene rocks and Pliocene marls in central Iran, respectively. A sepiolite mine is located near the city of Fariman in northeastern Iran. For industrial applications, especially pharmaceutical, health and food applications, the purification of this mineral should be done as appropriately as possible. As stated, HCl is commonly used to remove the carbonates minerals. Furthermore, one of the steps in the clay mineralogy of soils and sediments is removing carbonates by acetic acid-sodium acetate buffer (SAB) (Haris and White, 2008). In some cases, hydrochloric acid is used (Kehl et al., 2005). In these cases, it is possible to dissolve fibrous minerals in acidic conditions. The objectives of this study were to i) identify the characteristics of bulk sepiolite and investigate the efficiency of particle size separation for sepiolite purification, and ii) determine the kinetic dissolution of sepiolite in HCl and sodium acetate - acetic acid buffer (SAB).

2. Materials and methods

2.1. Determining the characteristics of the sepiolite

The bulk sepiolite sample was prepared from Dorkav Company in Mashhad. The relatively

hard sample was ground and the resulting powder was used for the experiment. The mineral composition of the powdered sample was determined by X-ray diffractometer, model Explorer using monochromatic Cu-Kα. The elemental composition of the sample was determined using a PW1480-PHILIPS X-ray fluorescence spectrometer. The amount of calcium carbonate in the sample was measured by the neutralization method (Allison, 1960).

2.2. Purification
Purification consists of two general parts including purification and estimation of purification efficiency. Since minerals are concentrated in different particle sizes, the separation of different particle fractions is a common method of purification (Klute, 1986; Jacqueline *et al.*, 2005; Ha Thuc *et al.*, 2010; Liu *et al.*, 2102). For this purpose, 20 g of the powdered sample was passed through a 270 mesh (50 µm) sieve after wetting in a dispersing solution (0.1% sodium hexametaphosphate and sodium carbonate) and particles <50 µm transferred to a one-liter cylinder. Based on the particle settling velocity in suspension according to Stokes' law (Klute, 1986), the particles were separated into <2, <20 and 20-50 µm fractions.

The best chemical method to remove a variety of carbonates (such as calcite and dolomite) are to use sodium acetate buffer - acetic acid with pH = 5 or HCl (Ha Thuc *et al.*, 2010; Inukai *et al.*, 1994). Due to the low amount of dolomite in the sample and the sensitivity of the structure of the sepiolite to acid, no chemical methods were used in this study.

X-ray diffractograms were used to determine the mineralogical composition of bulk sepiolite and separated fractions and to evaluate the efficiency of the purity of the fractions.

2.3. Sepiolite dissolution kinetic
The <20 µm fraction was used to investigate the kinetics of dissolution. Three ratios of solid (sepiolite) to liquid ratios (1:10, 1:25 and 1:50) in three replications were examined. Dissolution of sepiolite in HCl was investigated in four acid concentrations (0.25, 0.5, 0.75 and 1 N), at two temperatures (25 and 50 ºC) and at seven times (10, 30, 60, 180, 360, 720 and 1440 minutes). The samples were prepared in 50 ml flasks and stirred in an incubator shaker during the experiment. The resulting suspension was centrifuged at 2400 rpm for ten minutes and the top solution was used for measuring Mg concentration. This procedure was done for sodium acetate- acetic acid buffer (pH =5). The release Mg concentration was measured by the titration method with EDTA (Klute, 1986).

3. Results and discussion
3.1. Mineralogy and elemental composition of bulk sepiolite
Based on the results of X-ray diffraction of the bulk sample (Fig.1), the sepiolite was the main phase and the quartz, dolomite and palygorskite minerals were the associated minerals.

Sepiolite has a high-intensity peak at 12 Å and low-intensity peaks at 4.47, 4.27, 3.74, 3.34, and 3.18 Å (Singer, 2002) which are observed in this sample, too (Fig. 1). The peaks at 4.26 and 3.34 Å indicate the presence of quartz, peak at 2.87 represents dolomite, and peak at 10.72 Å is the index peak of palygorskite. According to semi-quantitative analysis, sepiolite and quartz composed 80 and 10-20% of the sample, respectively. The amount of both dolomite and palygorskite are 5-10%. Dolomite, quartz, and palygorskite are the three most common minerals in the world's sepiolite reserves (Inukai *et al.*, 1994, Singer *et al.*, 1998; Hojati *et al.*, 2012). According to the results, the amount of sepiolite in the Fariman mine sample is relatively high, while in the sample of Yenidogan, Turkey, the sepiolite content is about 46% and its dolomite is more than 50% (Inukai *et al.*, 1994).

The elemental composition of the Fariman sepiolite (bulk sepiolite) and some sepiolite

reserves in the world are given in Table 1. In Fariman sepiolite, the amount of SiO_2 is 54.51%, which is almost equal to the values of SiO_2 in Vicalvaro, Spain (Viseras, 1998), Elbor, Somalia (Singer, 1998) and the measured value by Hojati and Khademi (2012) in the studied region. The small amount of SiO_2 (27.25%) in Yenidogan sepiolite is attributed to the high amount of dolomite impurities.

Fi. 1. X-ray diffractogram bulk sepiolite (S: Sepiolite, P: Palygorskite, Q: Quartz, D: Dolomite)

Table 1. Elemental composition (%) of Fariman sepiolite and in other countries

Sepiolite	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	MnO	P_2O_5	S	*LOI	Reference
Iran (Fariman)	54.51	0.63	0.81	1.70	22.29	0.03	0.02	0.02	0.009	0.005	0.005	19.68	Present study
Iran, Fariman	55.32	0.3	0.61	2.74	15.73	0.02	0.01	0.02	-	-	-	25.11	Hojati and Khademi, 2012
Spain, Vicalvaro	56.44	1.28	0.38	0.21	23.50	0.10	0.64	0.06	0.02	-	-	17.32	Viseras *et al.*, 1998
Somalia, El bor	56.70	1.00	0.50	1.30	20.80	0.00	0.47	0.12	-	-	-	0.19	Singer *et al.*, 1998
Turkey, Yenidogan	27.95	0.54	0.19	16.3	23.72	0.10	0.08	0.04	-	-	-	31.06	Inukai *et al.*, 1994

* LOI: loss on ignition

Magnesium is one of the main elements in the composition of sepiolite and MgO has the second amount in the sepiolite constituent. This mineral is mainly formed in sedimentary saline and alkaline environments with and high activity of silicon and magnesium and very low activity of aluminum (Singer, 2008). The amount of MgO in Fariman sepiolite is 22.29%, which is almost equal to the other samples (Table 1). The amount of CaO in Fariman sepiolite is approximately 1.7%, which is almost equal to the amount of CaO in Elbo, Somalia. The high amount of CaO (16.3%) in Yenidogan, Turkey is due to the high amount of dolomite. The LOI is 19.68%, which is almost equal to the amount of LOI in other countries, but this amount is equal to 31.06% for Yenidogan, which is due to the high amount of dolomite.

3.2. Efficiency of purification

The results of the particle size distribution of the powdered sample are presented in Table 2. Particles larger than 50 µm (sand size) make up about 20% and particles < 2 µm (clay size) make up about 37% of the particles. About 43% of the particles are silt-sized (2-50 µm), and about 32% are particles 2 to 20 µm in size (fine and medium silt).

Table 2. The amount of particles (percent) of different sizes (µm) Fariman sepiolite

>50	20-50	<20	2-20	<2
19.27	11.23	69.50	32.25	37.25

Mirhosseini *et al.*, (2014) showed that in Ghaen bentonite samples, impurities such as quartz accumulated in larger particles of powdered sample. For this reason, powdered sepiolite was divided into different fraction sizes to compare their mineralogical composition. On the other hand, the purification method should be economic. For this reason, the particles <20 µm (clay+ fine and medium silt size), which make up about 70% of the sample, were separated and examined, too.

As shown in Fig. 1, dolomite, quartz, and palygorskite are the main impurities in bulk sepiolite. Quartz and palygorskite cannot be removed by chemical methods, and physical methods such as sieving, settling the particles in suspension and the use of ultrasonic waves are commonly used to separate them (Jacqueline *et al.*, 2005; Ha Thuc *et al.*, 2010 Liu *et al.*, 2012). Mirhosseini *et al.* (2014), James *et al.* (2008) and Marroquin-Cardona *et al.* (2011) removed a significant portion of the impurities using the fraction <50 μm.

The mineralogy of the bulk sepiolite and separated fractions are presented in Fig. 2. In the fraction of 20-50 µm, in addition to the sepiolite, quartz and dolomite were also identified as the main minerals. At this fraction size, the peak of the palygorskite was not observed. It seems palygorskite has accumulated in finer particles. In addition to sepiolite, palygorskite, quartz, and dolomite minerals were identified in the <20 µm particle size. In fraction <2 µm, the palygorskite and quartz are present, whereas, dolomitic peak at this particle size was not observed.

The analysis of diffractograms (Fig. 2) indicated that palygorskite has been removed in the 20-50 µm and dolomite in the < 2 µm fractions. The change of intensity or ratio of the peaks is an index of the relative amount of minerals. Inukai *et al.* (10) and Zhou *et al.* (25) attributed the peak intensities to purification efficacy. To show the removal of impurities, the intensity ratio of sepiolite peak (12.3 angstroms) to the peaks of palygorskite (10.27), quartz (3.34) and dolomite (2.87) in all three particle size fractions and the bulk sample was calculated (Table 3). Accordingly, the intensity ratio of sepiolite peaks to quartz, dolomite and palygorskite, in the bulk sample is 2.82, 5.11 and 7.28, respectively. These ratios have increased in all particle sizes, which means significant amounts of impurities left in particles > 50 µm. Mirhosseini *et al.* (2014) showed that the quartz accumulated in silt size fraction of bentonite and was removed by particle size fractionation. The ratio of sepiolite to palygorskite in the fraction of 20-50 µm and the ratio of sepiolite to dolomite in fraction < 2 µm are very high which show considerable removal of the palygorskite from 20-50 µm and dolomite from fine <2 µm fractions.

Dolomite impurities are eliminated in < 2 µm fraction. Considering that that the amount of < 2 µm is 37.25% (Table 2), separating this fraction is both costly and time-consuming. However, this fraction is suitable for sanitary and pharmaceutical purposes. In addition, most

of the impuritiy in this fraction is palygorskite, which like sepiolite is a fibrous mineral and has relatively similar properties. Despite the small amount of 20-50 µm (11.23%) and the presence of quartz and dolomite impurities, it seems that this fraction can be ignored. However, this fraction is suitable if a palygorskite-free sample is required. Among the fractions, the <20 µm fraction is economically viable; because, the amount of this fraction is high (about 70%), and on the other hand, the amounts of impurities have been considerably decreased.
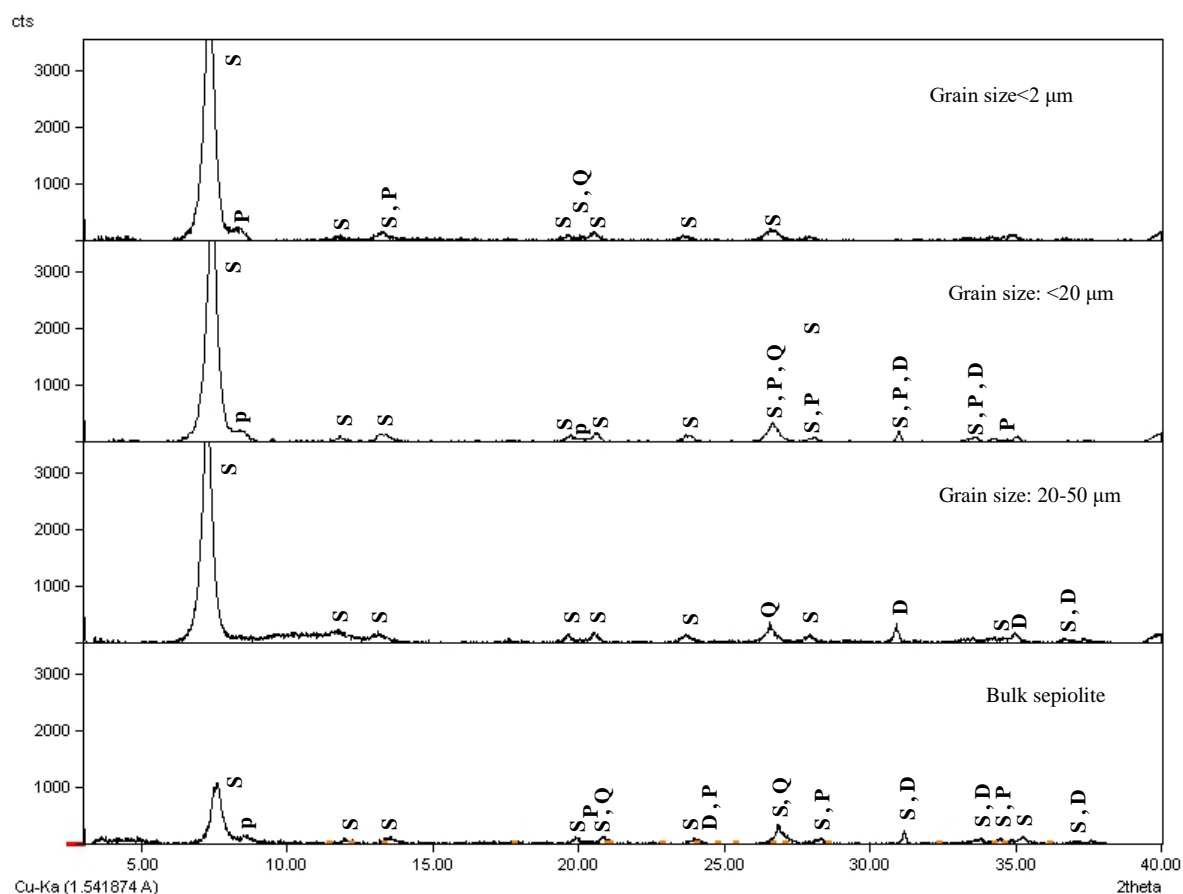
Fig. 2. X-ray diffractogram of bulck sample and partcles <2, 0-20 and 20-50 µm

Table 3. Ratio intensity of sepiolite indicator peak to indicator peaks of palygorskite, quartz and dolomite in the bulk and fractions of Fariman sepiolite

Mineral	Bulk sepiolite	20-50 µm	<20 µm	<2 µm
Palygorskite	7.28	High	20.28	17.60
Quartz	2.82	13.60	11.36	20.03
Dolomite	5.11	13.72	21.06	High

3.3. Dissolution of sepiolite

The fraction of <20 µm was used to investigate the dissolution of the sepiolite under the influence of HCl and SAB. The highest amount of released Mg was 100.5 mg g^{-1}, which occurred after 1440 minutes in HCl 1N, 1:50 solid to liquid ratio and 50 °C. The highest amount of Mg released in SAB solution, at the same conditions was 27.45 mg, which is much

lower than the HCl solution.

The trend of Mg release over time is similar at temperatures of 25 and 50 ºC (Fig. 3). At 10, 30, and 60 minutes, the slope of the released Mg curve is high, which decreases after 180 minutes, but does not reach a constant value after 24 hours. At all times of the experiment, the amount of released Mg at 50 ºC was higher than at 25 ºC, but over time, the difference in the amount of released Mg between temperatures increased, and this difference is greater for SAB. In HCl solution, the amount of released Mg clearly increases by increasing acid concentrations (Fig. 4).
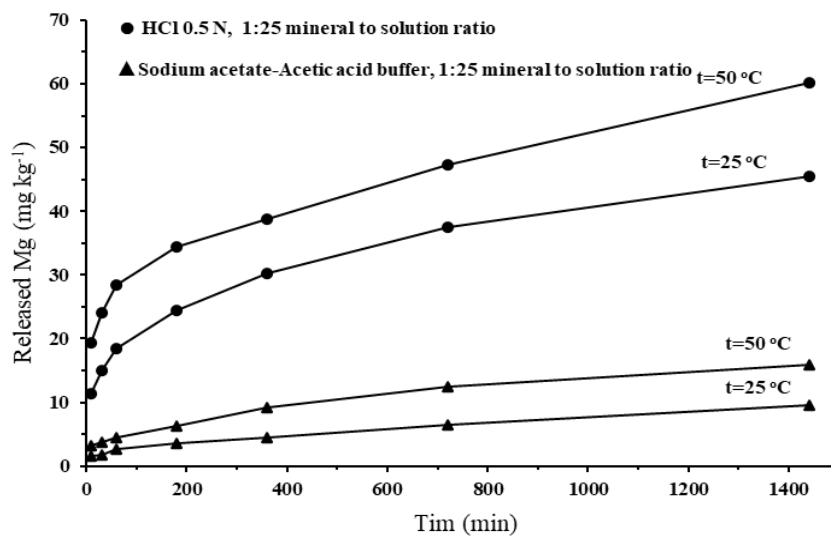
Fig. 3. Effect of temperature on released Mg from sepiolite under reaction with HCl and sodium acetate-acetic acid buffer
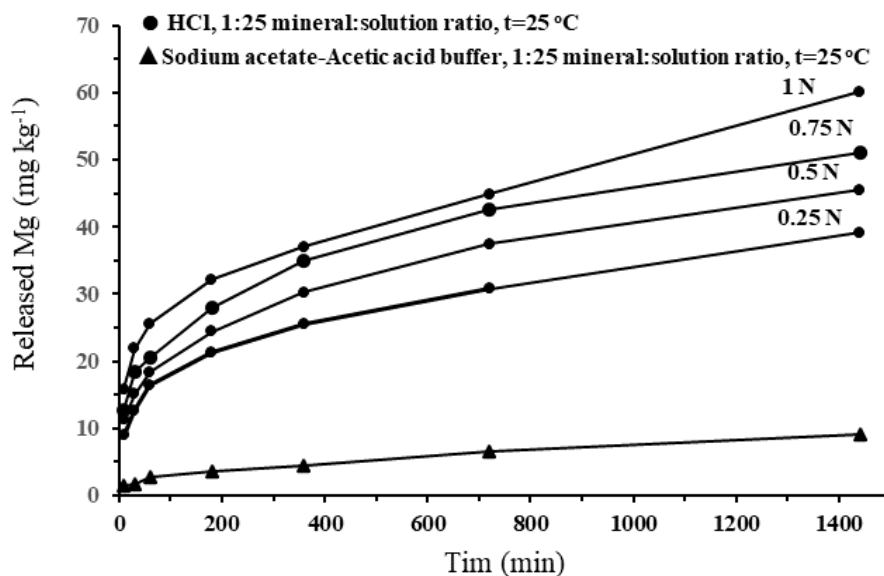
Fig. 4. Effect of HCl concentration and sodium acetate-acetic acid buffer on released Mg from sepiolite

In the early stages of the experiment, the Mg releases at the edges of the sepiolite layers, which is low but easily released, so the slope of the curve is high. However, over time, Mg must be released from the internal sepiolite structure, which is a slower process. This trend of releasing Mg is reported by Cetisli and Gedikbey (1990), and Ozdemir and Kipcak (2004).

As the soiled to liquid ratio increased, the amount of released Mg decreased (Fig. 5). It seems that with the increase of the solid to liquid ratio, sepiolite becomes a jelly-like state which the effective surface for the reaction decreases, and therefore, despite the higher amount of clay in higher ratios, the released Mg decreased.
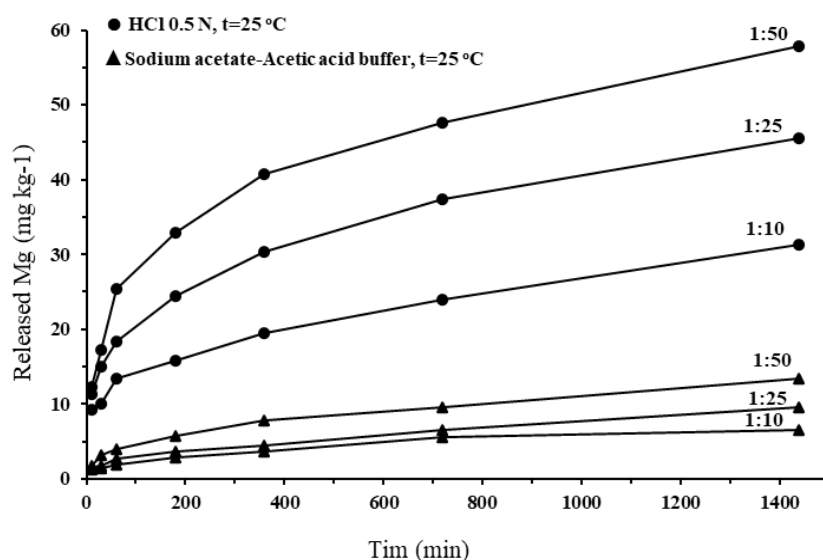

Fig. 5. Effect of solid (sepiolite) to liquid ratio on released Mg from sepiolite under reaction with HCl and sodium acetate-acetic acid buffer

3.4. Kinetic model

The data from the experimental treatments were fitted by kinetic equations to explore the mechanisms and influencing factors on the dissolution of the sepiolite. Based on the results, the data show the best fit with the kinetic rate equation of $kt = [(1-(1-X)^{1/2})^2]$. According to this equation, the step of controlling velocity reaction is the diffusion of reactants and products through the pores of solid phase (Ozdemir Kipcak, 2004).

The coefficient of explanation and the parameters of the equation are presented in Table 4. The results show that the apparent rate constant (k) is directly related to temperature, acid concentration and time and inversely related to the soli to liquid ratio. Ozdemir and Kipkak (2004) reported that this constant is a function of temperature, acid concentration, particle size and solid to acid ratio, the number of active sites on the solid and diffusion constant. They showed that with increasing temperature, concentration and time, the apparent rate increased and decreased with increasing the solid to liquid ratio.

In the experiment, increase of k was observed with increasing temperature from 25 to 50 °C both in HCl and SAB (Tables 4). In the HCl solution, by increasing the concentration of 0.25 to one M and 1:10 soiled to liquid ratio at 25 °C, the k value increased by 3.75 times. This increase was about 2.5 times in the 1:25 ratio and about twice in the 1:50 ratio (Tables 4). The same trend of k increase is observed at 50 °C. In other words, as the temperature increased, the rate of dissolution of the sepiolite and the release of Mg^{2+} ions increased. In the study of the effect of hydrochloric acid concentration on k increase, it was observed that with

increasing acid concentration from 0.25 to 1 M, the dissolution rate increased. It can be said that due to the increase in the rate of diffusion of hydronium ions from the solution to the active sites on the solid surface, which itself is due to the increase in acid concentration, the rate of Mg^{2+} diffusion from the solid phase to the solution increases. As the concentration of acid increases, the volume of pores and the total surface area increase, and eventually newly formed coarse and fine pores are formed that are more resistant to experiment conditions. Yebra-Rodriguez *et al.* (2003) stated that after acid treatment (0.5 N in 24 hours), the dissolution of tetrahedral and octahedral sheets starts from the center of the chain and creates tunnels of 6.29×5.20 angstrom along the a-axis. According to the data in Table 4, the reaction temperature was the most important factor in the dissolution of the sepiolite, followed by the acid concentration as the second influencing factor.

Table 4. The apparent rate constants (k) and coefficient of explanation (R2) of diffusion controlled rate equation for Sodium acetate-Acetic acid buffer and different concentration of HCl in different solid: solution ratio and temperature

Mineral: Solution ratio (w%)	Temperature (°C)	HCl concentration (N)								Sodium acetate-Acetic acid buffer	
		0.25		0.50		0.75		1			
		K (min)$^{-1}$	R^2	K (min)$^{-1}$	R^2	K (min)$^{-1}$	R^2	K (min)$^{-1}$	R^2	K (min)$^{-1}$	R^2
1:10	25°C	8×10^{-6}	0.90	1×10^{-5}	0.92	2×10^{-5}	0.90	5×10^{-7}	0.90	5×10^{-7}	0.95
	50°C	2×10^{-5}	0.73	3×10^{-5}	0.86	4×10^{-5}	0.85	7×10^{-5}	0.86	6×10^{-7}	0.92
1:25	25°C	2×10^{-5}	0.90	3×10^{-5}	0.91	4×10^{-5}	0.88	5×10^{-7}	0.93	9×10^{-7}	0.99
	50°C	4×10^{-5}	0.90	5×10^{-5}	0.87	7×10^{-5}	0.92	1×10^{-4}	0.86	3×10^{-6}	0.98
1:50	25°C	4×10^{-5}	0.98	5×10^{-5}	0.87	8×10^{-5}	0.92	8×10^{-5}	0.82	2×10^{-6}	0.98
	50°C	4×10^{-5}	0.92	9×10^{-5}	0.94	1×10^{-4}	0.92	2×10^{-4}	0.94	8×10^{-6}	0.99

3.5. Revision in clay mineralogy and carbonates measurements

Acceding to the results of this study, two revisions is suggested clay mineralogy and carbonate measurements of the soils, which may contain sepiolite in clay fraction.

Carbonates are one of the most common impurities associated with clay minerals. In clay mineralogical identification, the first step for clay fraction separation is removing the carbonates by dissolution in SAB, which has a slow reaction rate. In some cases, dilute HCl is used for this purpose (Inuki *et al.*, 1994; Hojati, 2011, Ha Thuc *et al.*, 2010). However, the high sensitivity of the sepiolite to the acidic environment and its dissolution, may remove the sepiolite from the sample, especially, in the samples with trace amount of sepiolite.

The carbonates of soil and sediments are usually measured by neutralization by HCl (Allison, 1960). However, in soils that contains sepiolite, the dissolution of this mineral may cause over-measuring the carbonates. According to the dissolution of sepiolite in HCl, acid titration does not appear to be a precise method to measure carbonates in samples containing sepiolite. During carbonate measurement, after titration and changing color to pink, the suspension become colorless after a few moments, and this process continued continuously after titration, indicating that the sepiolite was continuously dissolved.

To overcome the dissolution of sepiolite both in clay mineralogy study and carbonates measurement, increasing the Mg concertation in the solution proposed to inhibit the sepiolite dissolution as confirmed by Inuki *et al.* (1994).

4. Conclusion

Palygorskite, quartz, and dolomite are the associated minerals of Fariman sepiolite in northeastern Iran. The results of this study showed that separating bulk sepiolite in different fraction sizes is a time-consuming and cost-effective purification method. A portion of impurities were removed by initial sieving in particles > 50 µm. In particles < 2 µm, dolomite was removed, but palygorskite was present in small amounts. In general, <20 µm fraction which composed the major part of the sample, was appropriately purified and economically accepted. The results of sepiolite dissolution in an acidic environment showed that its solubility in HCl was much higher than that of SAB. The acid concentration had a positive and the solid to liquid ratio had a negative effect on magnesium release. Therefore, in soils with high sepiolite content, it's dissolving may cause the over-measuring of the carbonates by acid neutralization method. In addition, in soils with low sepiolite, it may dissolve during the removal of the carbonates by SAB and may not be detected in the soil.

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