Potassium status of two clayey calcareous soils treated with zeolite under wetting-drying cycles

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ABSTRACT- Zeolite application to clayey soils may change potassium (K) pools distribution and its release rate. To test this hypothesis, two heavy-textured calcareous soils with different mineralogy (smectitic and mixed by XRD) from southern Iran were selected. Experiment was a completely randomized factorial arrangement. Treatments consisted of two soils and two zeolite levels (0 and 5%) and wetting-drying treatment. Soil samples were incubated in 50% of saturation percentage for 90 days. Then, three wetting-drying cycles were done on samples. Different forms of K and K release to 0.01 M $CaCl_2$, HCl and CH_3COOH by 12 successive 15 min extractions of soil samples were determined. Zeolite increased the content of different K forms in soil 1 (smectitic), while it increased soluble and exchangeable K and decreased non-exchangeable K in soil 2 (mixed mineralogy). This may be due to the high CEC (189 cmol(+)/kg) and K content (2.13% K_2O) of the used zeolite. Wetting-drying cycles increased non-exchangeable K in soil 1 and exchangeable K in zeolite treated soils. $CaCl_2$ extracted more K than HCl and CH_3COOH solutions (682, 281 and 292 mg kg^{-1}, respectively) because Ca ions are more efficient than H ions in replacing K from surface sites in the K-bearing minerals. Zeolite and wetting-drying had no effect on K release from soil 1 while they significantly decreased K release rate from soil 2.

INTRODUCTION

The ability of soils to release potassium (K) depends very largely on their physicochemical and mineralogical properties. Some researchers concluded that soil amendments such as zeolite had the ability to affect K release from soil minerals due to the change in the equilibrium that exists among different forms of soil K (Rezaei and Movahedi Naeini, 2009). Panuccio et al. (2008) and Barros et al. (2004) indicated that zeolite decreased K release from soils and stated that this may be due to the high affinity of zeolite toward K ions.

It is proved that alternate wetting-drying events affect K pools distribution, fixation and release (McLean and Watson, 1985; Najafi-Ghiri et al., 2011a; Najafi-Ghiri and Abtahi, 2013). Changes in labile K during wetting-drying cycles are thought to reflect the gradual equilibration among the various K pools (Olk et al. 1995).

Different extractants have been used by investigators for the study of release of K from soils. These extractants may have different reactions to K-bearing minerals. Srinivasarao et al. (2006) stated that citric acid extracted a larger amount of non-exchangeable K followed by $CaCl_2$ and HCl for six major benchmark soil series of India. Jalali (2005), for some calcareous soils of western Iran, indicated that cumulative K released to citric acid was considerably larger than that released to $CaCl_2$.

Although soils in arid and semiarid regions of Iran contain large quantities of available K in soluble and exchangeable forms, the content of these K forms was decreased in agricultural soils due to the intensive crop production and little K fertilizers application (Balali and Malakouti, 1998). The non-exchangeable K may be released to soluble and exchangeable K during plant growth and this process may be affected by wetting and drying cycles and zeolite application. Therefore, the main purposes of this investigation were to compare the ability of two high clay calcareous soils with different mineralogy to release K to $CaCl_2$, HCl and CH_3COOH, to compare the ability of these solutions to extract K, and to study the effect of zeolite application and wetting-drying cycles on soil K release. Results of this study may be useful for determining the fate, behavior and availability of K in heavy-textured calcareous soils treated with zeolite.

MATERIALS AND METHODS

Soils and Zeolite and Physicochemical Analyses

According to the previous studies (Najafi-Ghiri et al. 2011c), two high-clay soils from two different climatic zones of Fars province (Sepidan and Darab regions), Iran, were selected. Surface soil samples (0-20 cm) were collected, air-dried, and sieved (<2mm) for laboratory analyses. Particle size distribution was determined by method of Rowell (1994). Calcium carbonate equivalent was determined by acid neutralization (Salinity Laboratory Staff, 1954). Organic carbon was determined according to Nelson and Sommers (1982). The soil pH was determined in saturated paste (Salinity Laboratory Staff, 1954). Electrical conductivity was determined in the saturated extract (Salinity Laboratory Staff, 1954). Cation exchange capacity (CEC) was determined using sodium acetate at a pH of 8.2 (Chapman, 1965). Different forms of K including soluble, exchangeable and non-exchangeable were determined by methods of Helmke and Sparks (1996). Water soluble K was determined in 1:5 of soil to water extract. Exchangeable K was extracted with 1.0 M NH_4OAc (pH 7.0). Non-exchangeable K was determined by extraction of soil sample with boiling 1 M HNO_3. Potassium was measured on all filtrated extracts by Corning 405 flame photometer (ELE, UK). Mineralogical analysis of soils was done by X-ray diffraction after removal of chemical cementing agents and separation of the soil fractions by methods of Kittrick and Hope (1963) and Jackson (1975).

Zeolite used in this study was obtained from Semnan mines, Iran. The natural zeolite used in this study had a pH of 7.31 and CEC of 189 cmol(+)/kg. Elemental composition of zeolite is shown in Table 1.

Incubation Experiment and K Release Study

The experiment was a completely randomized $2\times2\times2\times3$ factorial arrangement. The experiment was done in polyethylene pots, each containing 200 g soil. Treatments consisted of two soils and two zeolite levels (0 and 5%) with three replicates. First, 200 g of each soil was treated with 10 g zeolite (5%). Soil with no zeolite was considered as control. The soil samples were added to plastic bottles. Then, the samples were incubated in room for 90 days at 22 ± 3ºC (Filcheva and Tsadilas, 2002). Enough distilled water was added to bring the soil moisture level to 50% of saturation percentage. At the end of the incubation time, samples were air dried and crushed to pass a 2 mm sieve.

In order to distinguish the effect of wetting-drying cycles on the K release, 50 g of each soil sample obtained from previous incubation experiment was added to plastic bottles. The first cycle began with addition of distilled water (50% of saturation percentage). Bottles were kept uncapped and allowed to dry in the oven with a temperature of 60°C according to Najafi-Ghiri and Abtahi (2012)'s method for rapid drying. When the soils reached dryness after 3 days, deionized water was added to the soil for the second and third wetting-drying cycles (sum of 9 days). Another set of the soil samples was similarly incubated for 9 days at 50% of saturation percentage. At the end of the incubation time, samples were air dried and crushed to pass a 2 mm sieve and analyzed to determine different forms of K (methods mentioned in the previous section) and K release to 0.01 M $CaCl_2$, 0.01 M HCl and 0.01 M CH_3COOH.

Cumulative K release analysis was carried out by successive extraction of two grams of soil samples with 20 ml of 0.01 M $CaCl_2$, 0.01 M HCl or 0.01M CH_3COOH and 15 minutes shaking (Hagin and Feigenbaum, 1962; Lopez-Pineiro and Navarro, 1997). Then, the samples were centrifuged, and the concentration of K was determined in the clear solution. The soil was mixed with a new portion of extractants, shaken and centrifuged. This procedure was repeated 12 times. Cumulative K release from soil samples during 12-time extractions was determined.

Statistical Analysis

Statistical analysis was performed by the software MSTAT-C. Comparison of means was performed using the Duncan's Multiple Range Test (P 0.05).

RESULTS AND DISCUSSION

Physicochemical and Mineralogical Properties of the Studies Soils and Zeolite

Soil 1 was developed in Sepidan with soil moisture and temperature regimes of xeric and mesic, respectively, while soil 2 was developed in Darab with soil moisture and temperature regimes of aridic and hyperthermic, respectively. According to Keys to Soil Taxonomy (Soil Survey Staff, 2014), soils 1 and 2 belong to Typic Haploxererts and Typic Haplocambids, respectively. Some physical and chemical properties of soils are shown in Table 2. Both soils are calcareous and their calcium carbonate equivalent were 27 and 46 % for soil 1 and 2, respectively. Both soils have clayey textural class. Potassium constituted 6.9 and 5.3% of soils 1 and 2, respectively. The content of soluble K in soil 1 was lower than that of soil 2. On the other hand, exchangeable, non-exchangeable and total K were higher in soil 1 than soil 2. Soil 1 was smectitic with minor contents of illite and chlorite, while clay fraction of soil 2 was dominated with palygorskite, illite, chlorite and smectite. The cation exchange activity (CEA) (CEA was defined as CEC/clay percentage.) of the clays in two soils was 0.56 and 0.35, respectively. This parameter helps with making interpretations of the nutrient holding capacity of soils (Soil Survey Staff, 2014).

Table 1. Elemental composition of the zeolite used in this study

Element	SiO$_2$	Al$_2$O$_3$	Fe$_2$O$_3$	Na$_2$O	K$_2$O	CaO	MgO	TiO$_2$	Cl
Content(%)	70.56	8.47	0.59	3.56	2.13	0.27	0.41	0.13	0.77

Potassium Forms and Zeolite and Wetting-Drying Treatments

Table 3 indicates the analysis of variance for the different factors. Zeolite had significant effects on the content of different K forms (Tables 3 and 4).

Zeolite significantly increased the content of all forms of K in soil 1. It also increased the content of soluble and exchangeable K in soil 2, but non-exchangeable K was not affected. Potassium pools distribution among soluble, exchangeable and non-exchangeable forms was affected by zeolite application (except for soluble K in soil 1). This was obtained by dividing each K form by the total K and multiplying it by 100. In both soils, zeolite concentrated K in the exchangeable form, and this effect was more pronounced for soil 2 (mixed mineralogy). Potassium content of the used zeolite was 1.77% (equivalent to 2.13% K$_2$O) and with zeolite application to soil, all forms of K may be affected due to the equilibration that exists among different forms of K. Non-exchangeable or fixed K in soil 2 was not affected and this may be due to the low fixation capacity of this mixed mineralogy soil. Najafi-Ghiri (2014) indicated that zeolite application to five calcareous soils with clay content of 9-51%

increased soluble and exchangeable K, but changes in non-exchangeable K was related to the soil type and zeolite application which decreased it in most soils. Rezaei and Movahedi Naeini (2009) indicated that zeolite application to soil reduced soluble K, but it had a positive effect on available K. Filcheva and Tsadilas (2002) also concluded that zeolite application to soil increased exchangeable K.

Cumulative K Released to the Extractants

The pattern of K release (as cumulative) from soil samples by 12 successive extractions with 0.01 M CaCl$_2$, 0.01 M HCl and 0.01 M CH$_3$COOH is shown in Fig. 1. The initial release of K to all extractants was rapid and reached a constant rate after 4 extraction periods. As shown in Fig. 1, the pattern depends on the extractant solutions. The release rate was similar for both acidic extractants but significantly less than that for CaCl$_2$. This trend was similar to that reported by Jalali (2006) and Najafi-Ghiri et al. (2011b) for calcareous soils of Iran.

Table 2. Some properties of the studied soils

Soils	%					pH	CEC	EC	K forms (mg kg^{-1})			
	Clay	Silt	Sand	CCE	OC		cmol(+)kg^{-1}	dSm^{-1}	Soluble	Exchangeable	NEK	Total
1	43	32	25	27	1.7	7.40	24	0.70	30	450	880	6946
2	52	34	14	46	0.9	8.07	18	1.89	50	333	498	5321

CCE: calcium carbonate equivalent; OC: organic carbon; NEK: non-exchangeable K

Table 3. Analysis of variance for soils (A), zeolite (B), wetting-drying cycle (C) and their interaction

Source of variation	df	K forms			K pools distribution			Cumulative K release			% of HNO$_3$-K that		
		Soluble	Exchangeable	NE	Soluble	Exchangeable	NE	CaCl$_2$	HCl	CH$_3$COOH	CaCl$_2$	HCl	CH$_3$COOH
Factor A	1	7884**	18150**	1725384**	118**	997**	1797**	3037**	1395**	3725**	3174**	495**	459**
Factor B	1	4154**	424536**	75600**	7.0**	613**	753**	3037**	651*	950**	2016**	360**	392**
A×B	1	1584**	21961**	156978**	6.0**	275**	362**	7704**	4620**	4134**	368**	100**	100**
Factor C	1	459**	8214**	273	3.7**	10.3**	1.8	4004**	5985**	1890**	60**	57**	35**
A×C	1	84**	793	2460	0.54**	6.9**	3.3*	337	950**	828*	17*	7.0**	1.0
B×C	1	84**	3037**	4134	0.48**	4.4*	2.0	2204*	1552**	3.4	11	5.0*	0.04
A×B×C	1	84**	1014*	4620	0.43**	0.7	2.4	4.2	135	0.04	0.2	0.38	1.0
Error	16	4.6	194	4678	0.03	0.6	0.5	223.5	98	100	4.58	0.75	0.83
Total	23												
C.V		4.25	2.62	9.30	4.04	1.90	1.34	2.19	3.52	3.42	3.89	3.81	3.88

df: degree of freedom; NE: non-exchangeable; CV: coefficient of variation

* and ** represent 0.05 and 0.01 levels of significance, respectively

Table 4. Potassium pools distribution in the studied soils as affected by zeolite and wetting-drying cycles

Soils	Zeolite (%)	W&D	K forms (mg kg^{-1})			K pools distribution (%)		
			Soluble	Exchangeable	NEK	Soluble	Exchangeable	NEK
1	0	Without	30fg	450c	880d	2.2ef	33.1f	64.7a
		With	25g	463c	953c	1.7g	32.1f	66.1a
	5	Without	40de	620b	1100b	2.3e	35.2e	62.5b
		With	35ef	704a	1181a	1.8fg	36.7de	61.5b
2	0	Without	50c	333d	498e	5.7c	37.8d	56.5c
		With	45cd	349d	486e	5.1d	39.6c	55.3c
	5	Without	100a	650b	450e	8.3a	54.2b	37.5d
		With	80b	685a	435e	6.7b	57.1a	36.3d

NEK: non-exchangeable K; W&D: wetting-drying cycles.
Means in the same columns followed by different letters are significantly different at *P*<0.05 by Duncan's test
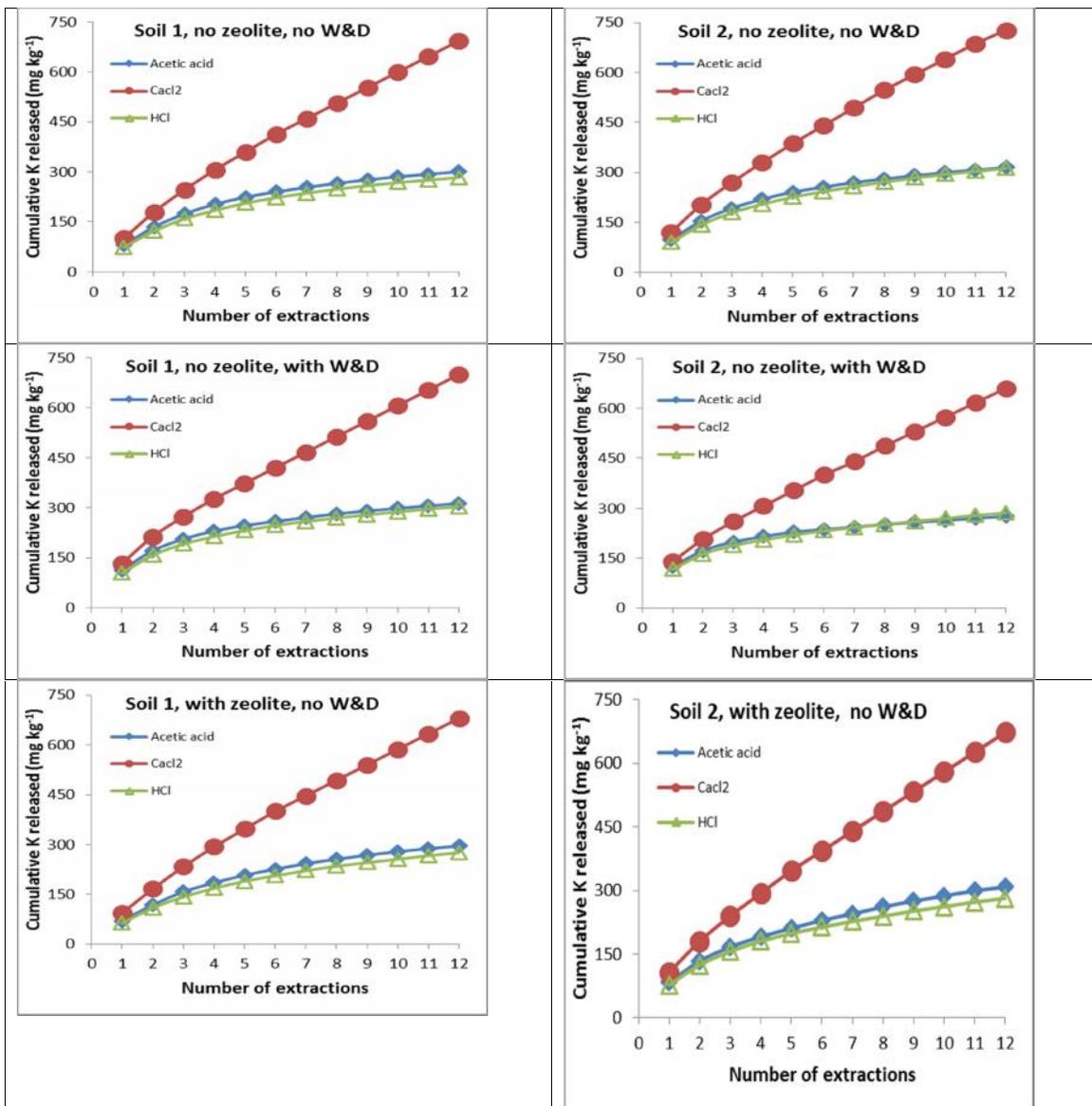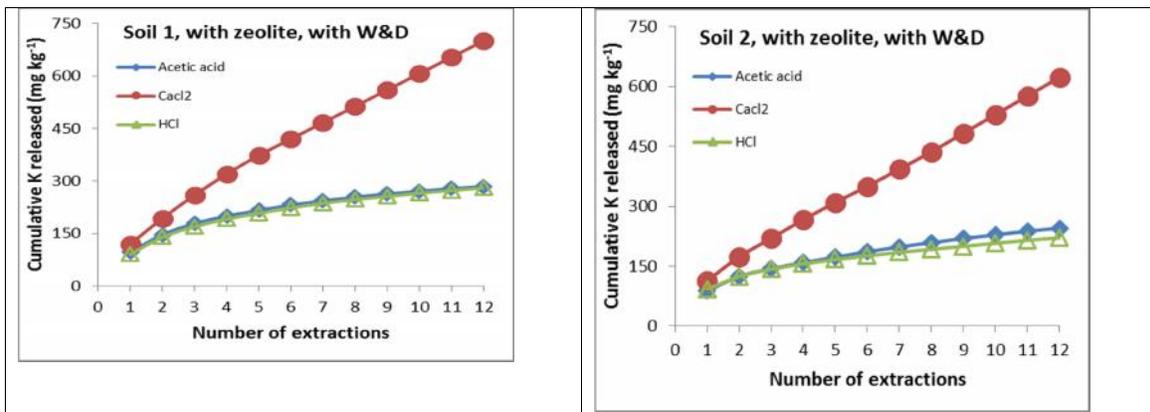
Fig. 1. Cumulative K released from different soil samples after 12-time extractions

It may be assumed that exchange reactions of K with Ca or H ions prevail in the first period, but Ca ions are more efficient than H ions in replacing K from surface sites in the K-bearing minerals. The soils continue to release K from internal positions, but because of larger size of Ca ions and hydration energy (Rich, 1968), it does not exchange easily with internal K (Srinivasarao et al., 2006). Some occluded K ions may be released due to the dissolution of carbonate particles by HCl and CH_3COOH. Thus, the initial phase of K release to acidic extractants may be due to the release of occluded K and exchange of H ions with K ions and also soluble K. The second slow release phase may be due to the exchange of H ions with K held on cation exchange sites.

Cumulative K released from soil samples by 12 successive extractions with 0.01 M $CaCl_2$, 0.01 M HCl and 0.01 M CH_3COOH is shown in Table 5. For all samples, $CaCl_2$ extracted more K than other acidic extractants, but there is not a significant difference between HCl and CH_3COOH in K extraction. Generally, $CaCl_2$ extracted K 2.43 and 2.33 times more than HCl and CH_3COOH, respectively. This is not consistent with Najafi-Ghiri and Jaberi's finding (2013) which showed no significant difference among $CaCl_2$, HCl and citric acid in K extraction from clay fractions of 10 calcareous soils. This difference may be due to the removal of soil carbonates and organic matter for clay dispersion and separation and thereby removal of soluble and exchangeable K.

Zeolite, wetting-drying cycles and zeolite*wetting-drying interaction had no effect on K release from soil 1. On the other hand, zeolite decreased K release from soil 2 significantly (*p*<0.05). This decrease was 6, 6 and 12 % for cumulative K released to $CaCl_2$, HCl and CH_3COOH solutions, respectively. This may be due to the affinity of zeolite for K adsorption in low CEA soils. Rezaei and Movahedi Naeini (2009) concluded that K desorption from a silty clay loam soil to Ca^{2+} and NH_4^+ solutions was decreased by zeolite application to soil.

Najafi-Ghiri (2014) also indicated that zeolite application to different calcareous soils may change K equilibration, but most soils released less K than the control soil. Decrease in K release with zeolite application may be explained by the fact that zeolite had a high affinity toward K ions (Barros & Arroyo 2004; Panuccio et al., 2008).

Wetting-drying cycles also significantly decreased K release from soil 2 to CH_3COOH (p<0.05); this decrease was 12%. Zeolite*wetting-drying interaction strongly decreased cumulative K released to all extractants (p<0.05) in soil 2. This was 11, 27 and 22 % for $CaCl_2$, HCl and CH_3COOH, respectively. Some K ions in the soil may be fixed by wetting and drying cycles and this may affect K release to extractant. In fact, as soil dries, soluble K becomes more concentrated in a smaller volume of soil solution, which increases the concentration gradient between solution and interlayers of K-fixing minerals and K fixation occurs (Nye and Tinker 1977; Sparks 1987). Increase in K fixation by wetting and drying cycles has been reported by Najafi-Ghiri and Abtahi (2012) for calcareous soils of southern Iran.

Percentage of HNO_3-extractable K that released to $CaCl_2$, HCl and CH_3COOH was 35-80, 15-35 and 16-36%, respectively. The highest percentage was observed in soil 2 without zeolite, while the lowest one was related to soil 1 treated with zeolite*wetting-drying cycles. Generally, zeolite and wetting-drying treatment decreased the percentage of HNO_3- extractable K released to all extractants. The effect of zeolite was more pronounced for soil 2. Generally, K fixation by wetting and drying cycles (Najafi-Ghiri and Abtahi, 2012) and high tendency of zeolite for K (Najafi-Ghiri, 2014) are the main reasons for increase in K fixation and decrease in K release from soils.

Table 5. Cumulative K released to 0.01 M CaCl$_2$, 0.01 M HCl and 0.01 M CH$_3$COOH during 180 min

Soils	Zeolite (%)	W&D	Cumulative K released (mg kg^{-1})			% of HNO$_3$-K that released		
			CaCl$_2$	HCl	CH$_3$COOH	CaCl$_2$	HCl	CH$_3$COOH
	0	Without	693abc	283bc	301abc	51bc	21d	22cd
1		With	680bc	276c	295abc	47c	19de	21de
	5	Without	727a	312a	315a	41d	18e	18e
		With	673bc	282bc	308ab	35e	15f	16f
	0	Without	700ab	304ab	314a	80a	35a	36a
2		With	700ab	280bc	285bc	80a	32b	32b
	5	Without	660c	286bc	276c	55b	24c	23c
		With	623d	221d	245d	52bc	18de	20d
Mean			682(a)	281(b)	292(b)	55(a)	23(b)	24(b)

NEK: non-exchangeable K; W&D: wetting-drying cycles.
Means in the same columns followed by different letters are significantly different at P<0.05 by Duncan's test

CONCLUSIONS

It was concluded that mineralogy, zeolite application to soils, wetting-drying cycles and kinds of extractant solutions were the effective factors on K release rate. The content of HNO$_3$-extractable K in soil 1 (with smectitic mineralogy) was significantly more than that of soil 2 (with mixed mineralogy). On the other hand, the percentage of HNO$_3$-extractable K that released to extractant solutions was lower for soil 1 than soil 2. Zeolite, wetting-drying and zeolite*wetting-drying interaction had no effect on K release from soil 1, while they decreased K release from soil 2 to all extractants. CaCl$_2$ solution extracts more K from both soils than HCl and CH$_3$COOH.

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وضعیت پتاسیم دو خاک آهکی رسی تیمار شده با زئولیت و چرخه‌های تری و خشکی

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چکیده- کاربرد زئولیت در خاکهای رسی می‌تواند توزیع شکل‌های پتاسیم و آهنگ آزادسازی آن را تغییر دهد. جهت بررسی این امر، دو خاک آهکی با بافت سنگین و کانی‌شناسی متفاوت (اسمکتیتی و مخلوط) از جنوب ایران انتخاب گردید. آزمایش به‌صورت فاکتوریل در قالب طرح کامل تصادفی بود. تیمارها شامل دو خاک و دو سطح زئولیت (۰ و ۵ درصد) و تیمار تری و خشکی بود. نمونه‌های خاک به مدت ۹۰ روز در حالت ۵۰ درصد رطوبت اشباع نگهداری گردیدند. سپس سه چرخه تری و خشکی روی نمونه‌ها انجام شد. شکل‌های مختلف پتاسیم و مقدار آزادسازی پتاسیم به‌وسیله عصاره‌گیرهای ۰/۰۱ مولار $CaCl_2$، HCl و CH_3COOH با ۱۲ عصاره‌گیری پیوسته ۱۵ دقیقه‌ای نمونه‌ها اندازه‌گیری گردید. زئولیت مقدار شکل‌های مختلف پتاسیم را در خاک ۱ (اسمکتیتی) افزایش داد و شکل‌های محلول و تبادلی را در خاک ۲ (کانی‌شناسی مخلوط) افزایش اما پتاسیم غیرتبادلی را کاهش داد. این در نتیجه ظرفیت تبادل کاتیونی بالا (۱۸۹ سانتی‌مول بر کیلوگرم) و مقدار پتاسیم بالای آن (۲/۱۳ درصد اکسید پتاسیم) می‌باشد. چرخه‌های تری و خشکی مقدار پتاسیم غیرتبادلی را در خاک ۱ و مقدار پتاسیم تبادلی را در خاکهای تیمار شده با زئولیت افزایش داد. عصاره‌گیر $CaCl_2$ پتاسیم بیشتری را نسبت به عصاره‌گیرهای HCl و CH_3COOH استخراج کرد (به‌ترتیب ۶۸۲، ۲۸۱ و ۲۹۲ میلی‌گرم بر کیلوگرم)؛ زیرا یون‌های کلسیم کاراتر از یون‌های هیدروژن در تبادل پتاسیم از سطوح کانی‌های پتاسیم‌دار هستند. زئولیت و چرخه‌های تری و خشکی تأثیری بر آزادسازی پتاسیم از خاک ۱ نداشت اما سرعت آزادسازی پتاسیم از خاک ۲ را به‌طور معنی‌داری کاهش داد.