Potassium Fixation in Soil Size Fractions of Arid Soils

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Abstract

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Amounts of potassium (K) fixed in soil fractions of 10 calcareous soils of southern Iran were measured to evaluate the contributions of different soil size fractions to K fixation. Soil particles were fractionated after dispersion of the soils with an ultrasonic probe. Potassium fixation analysis was done by addition of 1000 mg K/kg samples. Mineralogy of the size fractions was determined by X-ray diffraction. The clay fractions were dominated by smectite, chlorite, mica, and palygorskite. Potassium fixation capacities ranged from 104 to 148 mg/kg for clay, from 102 to 155 mg/kg for silt, and from 96 to 187 mg/kg for sand fractions. A positive and significant relationship ($P < 0.05$) was obtained between K fixation capacity and smectite content for the clay fractions. High amounts of K fixed in the sand fraction may be explained by a larger diffusion path of fixed K out of the frayed edges of micaceous and smectitic minerals into the extracting solution, low cation exchange capacity (CEC) of the coarse fraction and thereby the high concentration gradient along solution and interlayers, physical entrapment of K ions in coarse aggregates cemented by carbonates, and by the presence of clay particles in coarse fractions due to incomplete dispersion of coarse aggregates.

Keywords: calcareous soils; Iran; smectite; soil fractionation

Potassium (K) fixation is an important process in soils influencing K availability to plants. In general, K fixation is a chemical process that is governed by the equilibrium between K located in interlayer positions of K-bearing minerals and K held at planar sites and in the soil solution (Olk & Cassman 1995). The ability of soils for K fixation is controlled mainly by the nature of minerals (Sharma & Mishra 1991). Soils with a high content of expanding 2:1 type clay minerals such as illite, vermiculite and smectite have stronger K fixation (Sparks & Huang 1985).

Many studies have been conducted on K fixation in soils and pure minerals, but limited data are available on contributions of coarse fractions (sand and silt) to K fixation, particularly in calcareous soils. Calcareous soils may contain appreciable quantities of K fixing minerals such as mica in the sand and silt-sized fractions, and these components may be important in soil K fixation. Recently, Murashkina *et al.* (2007) reported for five Sierra Nevada granitic alluvium soils (California, USA) that the highest percentage of added K was fixed by silt fractions, dominated by vermiculite and hydrobiotite.

Many researchers (Munn *et al.* 1976; Leinweber *et al.* 1993; Caravaca *et al.* 1999) successfully used the ultrasonic dispersion of soils in water for size fractionation. This method is less destructive than chemical methods including pretreatment for $CaCO_3$ removal. In fact, $CaCO_3$ removal with acidic salt solutions (e.g. 1N sodium acetate buffered at pH 5) in highly calcareous soils requires a long time to perform and thus it is not suitable for K analysis, because salt solutions may extract K from soils and minerals (Lopez-Pineiro & Garcia-Navarro 1997; Jalali 2006; Najafi Ghiri *et al.* 2011), and subsequently affect their K fixation capacities.

Although soils in arid and semiarid regions of Iran contain large quantities of exchangeable and nonexchangeable K, their reserves in agricultural soils have been decreased since 20 years ago, because of the intensive crop production and little or no application of K fertilizers (Balali & Malakouti 1998), and thereby K fertilizer application is required for optimum plant production, but information about fate and behaviour of added K and also contributions of sand, silt, and clay fractions to K fixation is limited. The present study examines the mineralogy and characteristics of K fixation by sand, silt, and clay separates of 10 calcareous soils of divergent texture and mineralogy in southern Fars, Iran.

MATERIAL AND METHODS

Soils and physicochemical analysis. Ten soil series were selected from arid regions of southern Fars, Iran. Pits were dug, described (Soil Survey Staff 1993) and classified (FAO 2006; Soil Survey Staff 2010). Soils from different horizons were sampled, air-dried, and crushed to pass a 2 mm sieve for laboratory analysis. Then, surface soil samples (A or Ap horizons) were considered for particle separation and K fixation and mineralogical analysis.

Soils were fractionated by using a Virsonic Ultrasonic Cell Disrupter (Model 16-850, The Virtis Co., Gardiner, USA) for dispersion (Munn *et al.* 1976). The sand fraction was separated by wet sieving, and the clay was separated from the silt by repeated sedimentation and decantation.

Calcium carbonate equivalents of each soil sample and sand, silt, and clay fractions (Salinity Laboratory Staff 1954), organic carbon (Nelson & Sommers 1982), soil pH (Salinity Laboratory Staff 1954), electrical conductivity (Salinity Laboratory Staff 1954), cation exchange capacity (CEC) (Chapman 1965) were determined. Different forms of K were determined by methods of Helmeke and Sparks (1996): total K by digestion (383°K) of soil with HF and HCl, water soluble K in the saturated extract, exchangeable K by NH_4OAc (pH 7), and nitric acid-extractable K by extraction of soils with boiling 1M HNO_3 for 1 hour. Potassium was measured in all filtrated extracts using a Corning 405 flame photometer (Corning Incorporated, Corning, USA). Analyses were carried out in triplicate.

Potassium fixation experiments. For K fixation experiments, 5 g of each soil fraction sample was weighed into a 50 ml plastic bottle and was shaken on a rapid reciprocating shaker for 24 h at room temperature (20°C) after addition of 25 ml 5.13M KCl solution (equivalent to 1000 mg K/kg soil). Another set of soil samples was similarly shaken for 24 h after addition of 25 ml distilled water (control set). Samples were extracted for K three times with 1M NH_4OAc. The extractants were collected in a 100 ml volumetric flask, diluted to volume, and measured for K by flame photometry (model Corning 405). The K fixation capacity was calculated as follows:

$$\text{K fixed} = \text{added K} + NH_4OAc\text{-extractable K of control sample} - NH_4OAc\text{-extractable K.}$$

Mineralogical analysis. Before mineralogical analysis, chemical cementing agents including soluble salts, carbonates, organic matter, and free Fe oxides were removed using deionized water, 1N sodium acetate (pH 5), 30% H_2O_2, and citrate dithionate, respectively. The neutralization was performed in a water bath at 80°C. The sand fraction was separated by wet sieving. The clay was separated from the silt by repeated sedimentation and decantation. All size fractions were saturated with KCl and $MgCl_2$. The Mg-saturated samples were X-rayed (mica identification), glycolated (smectite identification), and X-rayed again with a Philips D500 diffractometer using Ni-filtered CuKa radiation (40 kV, 30 mA). The K-saturated samples were X-rayed after air-drying and again after heating to 550°C (chlorite identification). For identification of kaolinite in the presence of chlorite, samples were treated with 1N HCl at 80°C, overnight. The content of minerals in sand, silt, and clay fractions was determined semiquantitatively, using the ratio of X-ray peak areas (001) on the diffractograms of ethylene glycol solvated specimens according to Johns *et al.* (1954).

RESULTS AND DISCUSSION

Physicochemical properties of soils

Table 1 shows soil classification, parent material, land use, altitude, slope, physicochemical soil

Table 1. Some properties of the studied soils

Soil No.	Classification		Parent material	Land use	Altitude (m)	Slope (%)	Particle size distribution (g/kg)			pH	CCE[1]	OC[2]	EC[3]	CEC[4] (cmol(c)/kg)	K concentration (mg/kg)			
	USDA	WRB					sand	silt	clay	(H_2O)	(g/kg)		(dS/m)		WS[5]	NH_4OAc	HNO_3	total
1	Typic Xerofluvents	Haplic Regosols	Calcareous alluvium	wheat	1500	0–2	380	390	230	7.5	460	10	4.0	8	4.8	126	659	3622
2	Typic Haplocalcids	Haplic Calcisols	Calcareous alluvium	wheat-maize	1599	0–2	460	420	120	7.3	450	8	3.7	13	7.8	167	740	4972
3	Typic Haplocalcids	Haplic Calcisols	Calcareous alluvium	wheat-maize	1616	0–2	410	400	190	7.6	480	6	0.9	12	6.1	249	790	5398
4	Typic Ustorthents	Haplic Regosols	Calcareous alluvium	rice	719	0–2	240	510	250	7.8	530	19	6.1	11	8.2	132	676	6017
5	Calcic Petrogypsids	Petric, Hypogypsic, Calcic Gypsisols	Gyp. & cal. alluvium	wheat	2056	2–5	350	490	160	7.4	490	6	1.8	8	12.2	256	856	5485
6	Aridic Ustorthents	Haplic Regosols	Calcareous alluvium	wheat	837	0–2	210	440	350	7.6	560	16	0.9	12	15.4	370	1134	5660
7	Typic Torriorthents	Haplic Regosols	Calcareous alluvium	wheat	854	2–5	300	560	140	7.6	540	3	7.1	5	8.0	83	412	5748
8	Typic Torriorthents	Haplic Regosols	Calcareous alluvium	wheat	844	0–2	500	410	90	7.7	600	7	8.0	5	17.0	127	628	5927
9	Sodic Haplocambids	Hypersalic Solonchaks	Calcareous alluvium	range	839	0–2	50	530	420	7.5	510	6	13.3	7	46.3	297	1010	5660
10	Aridic Haploxererts	Haplic Vertisols	Calcareous alluvium	wheat-maize	1219	0–2	120	510	370	7.5	430	8	3.1	9	11.7	223	1098	7628
Mean							302	466	232	7.6	505	9	4.9	9	13.8	203	800	5611

1 – calcium carbonate equivalent; 2 – organic carbon; 3 – electrical conductivity; 4 – cation exchange capacity; 5 – water soluble K

properties, and quantities of soluble, NH_4OAc-extractable, HNO_3-extractable, and total K. All soils are calcareous and belong to Vertisols, Entisols, and Aridisols, the soils developed under arid climatic conditions. The studied soils had different relative proportions of sand, silt and clay size fractions. Sand content ranged from 5 to 50% (mean = 30%), silt content from 39 to 56% (mean = 47%), and clay content from 9 to 42% (mean = 23%). There were large differences in soil equivalent $CaCO_3$ (range 43–60%), soluble K (range 4.8–46.3 mg/kg), NH_4OAc-extractable K (range 83–370 mg/kg), HNO_3-extractable K (range 412–1134 mg/kg), and total K (range 0.36–0.76%) among the studied soils. Soluble K was significantly correlated with EC ($r = 0.744^*$). In fact, K with calcium, magnesium, and sodium are the major cations contributing to soil salinity in arid regions of southern Iran (Najafi Ghiri 2010). Generally, NH_4OAc-extractable K, as an index of K availability in calcareous soils, had significant and positive relationships with clay ($r = 0.634^*$) and HNO_3-extractable K ($r = 0.887^{**}$). Similar relationships were also found for HNO_3-extractable K. Nonexchangeable K is calculated from differences between HNO_3- and NH_4OAc-extractable K. Some salt precipitants, such as sodium tetraphenyl-boron, are also able to extract nonexchangeable K from micas, but are less able to extract K from the structures of feldspars. However, much information about nonexchangeable K has been gained using destructive HNO_3 methods, although they have been less favoured because of their severe extraction conditions (Song & Huang 1988; Carey & Metherell 2003). According to the threshold level of K for calcareous soils reported by Tafarroji and Haghparast Tanha (2005) as 148 mg/kg, nearly 60% of the surface soil samples had NH_4OAc-extractable K well above this limit.

Calcium carbonate contents in different soil fractions (Table 3) ranged from 16 to 31% (mean = 23%, SD = 5.9) for clay fractions, 46 to 59% (mean = 55%, SD = 4.3) for silt fractions, and 47 to 81% (mean = 69%, SD = 9.9) for sand fractions.

Clay mineralogy of soil fractions

Mineralogical analyses of the clay, silt, and sand fractions are shown in Table 2. Clay fractions were dominated by smectite, chlorite, mica, and palygorskite with a trace amount of quartz. Smectites in calcareous soils of southern Iran may be the product of palygorskite and illite weathering or transformation, but most are inherited from parent rocks (marl formations). Palygorskite was common in soils 11, 16, 20, and 21. The clay fraction of soil 13 was dominated by chlorite. Mica occurred in all soils, but it was not a dominant clay mineral.

Mineralogical analysis indicated that quartz, mica, chlorite, smectite, and feldspars (mainly orthoclase) were dominant minerals occurring in the sand and silt fractions. Generally, quartz was a dominant mineral component of the sand and silt fractions.

Table 2. Mineralogy of sand, silt, and clay fractions of the studied soils (in mm)

Soil No.	Classification	Clay (< 0.002)	Silt (0.002–0.05)	Sand (0.05–2.0)
1	Typic Xerofluvents	S = C = M = P	Q >> C = S = F = M	Q > > F > M
2	Typic Haplocalcids	S = M = C > P > Q	Q >> S = M = C > F	Q >> F > M
3	Typic Haplocalcids	S = M > C = P	Q >> S = M = F = C	Q >> F > M
4	Typic Ustorthents	S = C = P > M	Q >> S = C > M = F	ND
5	Calcic Petrogypsids	C > M = S = P	Q >> S = C = M > F	ND
6	Aridic Ustorthents	S = P = M > C	Q >> C > M = S = F	ND
7	Typic Torriorthents	S > C > M	Q >> M > C = S = F	ND
8	Typic Torriorthents	S = C > M > P	Q >> M = C = S = F	Q >> F > M
9	Sodic Haplocambids	C = S = P > M	Q >> C > F = S = M	ND
10	Aridic Haploxererts	C = S = M = P > Q	Q >> C >> F = S = M	ND

C – chlorite; F – feldspars; M – micas; P – palygorskite; Q – quartz; S – smectite; ND – no data

Potassium fixation by soil fractions

Amounts of K fixed by sand, silt, and clay fractions, by addition of 1000 mg K/kg, are shown in Table 3. Results indicated that, on average, the sand fraction fixed significantly higher amounts of K than the other fractions, but we found no significant difference between silt and clay fractions in K fixation capacity. For better understanding of the effect of mineralogy on K fixation capacities by soil fractions, we studied the correlation between K fixation capacity and the content of common minerals occurring in each soil fraction. Results indicated that for clay fractions, K fixation capacity was significantly correlated with the smectite content (Figure 1, $r^2 = 0.55$, $n = 10$, $P < 0.05$). Some researchers (SHAVIV *et al.* 1985; MURASHKINA *et al.* 2007) stated that soils containing micas, hydrous micas, or vermiculites have the highest fixation capacities, whereas smectitic and kaolinitic soils have low fixation capacities. Since vermiculites are relatively rare in calcareous soils of southern Iran (OWLIAIE *et al.* 2006), it seems that smectites and weathered micas are the main clay minerals responsible for K fixation in the studied soils. However, we found no relationship between K fixation capacity and mica content in the clay fraction. Potassium fixation capacities in the clay fractions ranged from 104 to 148 mg/kg (mean = 126 mg/kg). Generally, K fixation capacities in the clay fractions were lower than 150 mg/kg (lower than 15% of added K) for all soils. The lowest amount of K fixation was found in clay 5 that was chloritic.

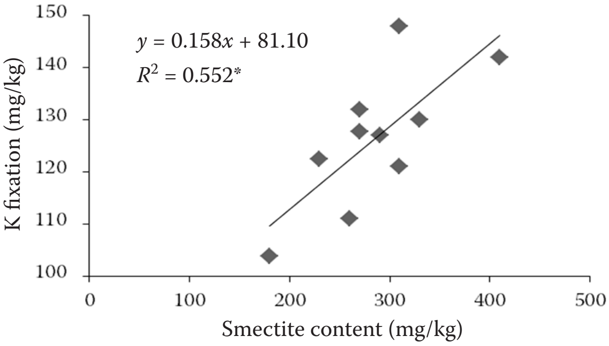

Figure 1. Relationship between K fixation capacity and smectite content in the clay fractions

K fixation in the silt and sand fractions ranged from 102 to 155 mg/kg (mean = 134 mg/kg) and from 96 to 187 mg/kg (mean = 163 mg/kg), respectively. By comparison, MURASHKINA *et al.* (2007) reported higher amounts of K fixation in the silt fractions (5.3–17.0 mmol K/kg silt) and also negative fixation in the clay fractions (release of 2.3–6.5 mmol K/kg clay). They stated that in these larger-size particles, K fixation may be affected by a longer diffusion path into the mineral, and/or by partial collapse of the fringe zones of vermiculitic and micaceous minerals. There was not a significant relationship between K fixation capacity and

Table 3. Contributions of the clay, silt, and sand fractions to soil K fixation

Soil No.	Calcium carbonate equivalent (%)			Smectite content (%)			K fixation (mg/kg)			Contribution to K fixation (%)		
	clay	silt	sand	clay	silt	sand	clay	silt	sand	clay	silt	sand
1	200	590	650	27	12	tr	128	153	96	23	48	29
2	160	540	750	31	13	tr	148	155	183	11	39	50
3	310	590	810	27	13	tr	132	139	157	17	39	44
4	230	580	660	29	14	–	127	120	173	24	45	31
5	300	510	470	18	15	–	104	131	185	11	45	44
6	190	460	680	31	9	–	121	102	187	33	36	31
7	160	540	750	41	11	–	142	142	156	14	55	31
8	310	590	810	33	13	tr	121	109	170	7	32	61
9	190	530	700	26	7	–	111	140	142	37	58	5
10	230	580	660	23	4	–	122	150	177	32	53	15
Mean	228^a	551^b	694^c	29	11	–	126^a	134^a	163^b	21^a	45^b	34^{ab}

Means followed by different letters are significantly different at $P < 0.01$ by *t* test; tr – trace mineralogy for sand and silt fractions. Although smectite was not a dominant mineral in the sand and silt fractions (particularly in sand fraction), weathered mica may be responsible for K fixation in these coarse fractions. High amounts of K fixed in the sand and silt fractions may be explained by a larger diffusion path of fixed K out of the frayed edges of micaceous and smectitic minerals into the extracting solution. On the other hand, the elevated soluble K concentration after addition of K to coarse fractions (as a result of low CEC) and thereby the high concentration gradient along solution and mica interlayers may aggravate K fixation in coarse fractions. Studies on the release of fixed K in micas by Scott (1968) showed that the initial release of K from surface sites was somewhat greater in smaller particles, presumably due to the larger surface area and more surface K on smaller particles. In fact, smaller particles resist the release of K fixed in the frayed edges. Therefore, NH_4OAc extraction of samples in fine particles is more effective than in the coarse ones during a short time of extraction (15 min in this study).

Results indicated that although calcium carbonate contents of the soil fractions varied widely, no significant relationship was obtained between the amounts of this compound and K fixation capacity in different soil fractions. However, $CaCO_3$ may be effective in physical entrapment of K ions in the coarse aggregates cemented by $CaCO_3$.

Table 3 also presents the relative contribution of each soil fraction to the total amount of K fixed. This is obtained by multiplying the K fixed in each soil fraction by the percentage of each soil fraction, and dividing this quantity by the total K fixed in all soil fractions. Results indicated that the silt fractions were responsible for 45% of the soil K fixation capacities (ranging from 32 to 58%). Although the role of silt in K fixation seems to be important (because of a high percentage of silt in the soils), this is not true of soils 2 and 8. In fact, only 39 and 32% of the total K fixation capacities were related to the silt fractions in soils 2 and 8, respectively. Contribution of the clay fraction to soil K fixation capacity ranged from 7 to 37% (mean = 21%). Contribution of sand fractions to soil K fixation was 34%, as average. Sandy soils such as soils 2 and 8 had high K fixation capacities in the sand fractions (higher than 50%). On the other hand, contribution of sand fractions to the soil K fixation in fine- and medium-textured soils (soils 9 and 10) was less than 20%. Generally, the larger contribution of silt fraction to soil K fixation is due to the high proportions of silt fraction in the calcareous soils. This is inconsistent with the finding of Murashkina *et al.* (2007), who concluded that the larger contribution of silt to K fixation was related to the predominance of vermiculite and hydrobiotite in this fraction.

CONCLUSIONS

It is concluded that K fixation may be an overriding process in strongly calcareous soils of southern Iran. Sand fractions fixed significantly more K than silt and clay fractions, but when particle size distribution is taken into account, the silt fractions seem to be responsible for nearly a half of the soil K fixation capacities. The dominant clay minerals responsible for K fixation in the studied soils were smectites. A positive and significant relationship was obtained between K fixation capacity and smectite content for the clay fractions. No such relationship was obtained for silt and sand fractions. Generally, higher K fixation in sand fractions may be due to the following reasons:

- The elevated soluble K concentration after addition of K to coarse fractions (as a result of low CEC) and thereby the high concentration gradient along solution and mica interlayers may aggravate K fixation in coarse fractions.
- A larger diffusion path of fixed K out of the frayed edges of micaceous and smectitic minerals into the extracting solution.
- Physical entrapment of K ions in coarse aggregates cemented by carbonates.
- Lower K release from coarse particles in comparison with fine fractions.
- Incomplete dispersion of fine particles in coarse fractions.

However, separation of soil particles without removal of soil cementing agents, particularly in highly calcareous soils, seems to arouse questions which must be taken into consideration. Further research is required to study the contribution of different soil fractions to K fixation after removal of soil cementing agents.

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