

Wet Fixation of Potassium in an Iraqi Soil, Ameriya Silty Clay Loam¹

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1. Introduction

Potassium fixation is observed in many soils, whereby added potassium is rendered unextractable by ordinary laboratory cation-exchange reagents. Wide interest in the subject was shown in the past on account of its relation to the availability of potassium to plants as well as to the behaviour of the 1:2 expanding lattice-type of the alumino-silicate clay minerals. Due to its suitable size the dehydrated potassium ion is conceived to be the best fitting among cations in the hexagonal voids created by the basal tetrahedral oxygens of adjacent structural units of these minerals (*Page and Bawver*, 1940). The polarizability of potassium and other ways were thought to be operative also in both wet and dry potassium fixation (*Schuffelen and Marel*, 1955).

Research works concerning potassium fixation and its relation to soil and conditioning factors were discussed and reviewed by *Reitemeier* (1951), *Schuffelen and Marel* (1955), *Agarwal* (1960), and *Black* (1968).

Arid and semi-arid soils of Iraq are generally well-supplied with available potassium. Due to increasing cropping intensity in many projects, this investigation, which is the first about the subject, seems feasible.

2. Material and methods

The Soil

Ameriya silty clay is a calcareous alluvial soil formed under arid climatic conditions in the middle Mesopotamian plain from mixed sediments mostly from Euphrates river. Some of the physical and chemical soil characteristics are shown in table 1.

Table 1. Some physical and chemical properties of Ameriya silty clay loam¹

pH of saturated soil paste	8.09	Organic matter (%)	1.85
Specific conductance (mmhos/cm, 25 °C)	1.40	Lime equivalent	20.00
Soluble ions (me/100 g):		Gypsum (%)	0.77
Ca ⁺⁺	0.69	C. E. C. (me/100 g)	28.24
Mg ⁺⁺	0.53	Moisture at saturation (%)	55.00
K ⁺	0.039	Particle size distribution (%):	
Na ⁺	0.586	sand	13.0
CO ₃ ⁻⁻	0	silt	57.0
HCO ₃ ⁻	0.44	clay	30.0
Cl ⁻	1.37		

¹ Values from the M. sc. thesis by *F. A. A. Latif*, College of Agriculture, Abu Graib/Iraq, 1975.

¹ Contribution from the University of Baghdad (Iraq).

All laboratory work was done at Martin Luther University, Halle (GDR).

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Methods

Sorbed potassium was determined in soil suspensions at various time intervals in order to determine the time lapse at which approximate equilibrium is established in potassium-treated soil suspensions. In this experiment, 100 g soil samples, on an oven-dry basis, were placed in polyethylene bags, with 100 ml of potassium chloride solution of increasing concentrations from 50 ppm to 1000 ppm K added. The suspensions were incubated at 27–30 °C, and supernatants were sampled for potassium at 4, 7, 15, 30, and 64 days after mixing. The potassium concentration was determined by EEL flame photometer and sorbed K was then calculated.

The procedure used in the potassium fixation experiment was as follows:

Two grammes of air-dried soil samples were placed in 10 ml centrifuge tubes. Volumes of 1.5 ml, 2.0 ml, and 3.0 ml, each having potassium concentrations of 0 ppm, 500 ppm, 1000 ppm, and 2000 ppm, were added to the soil samples in the centrifuge tubes. Moisture was thus varied as well as potassium concentration. Potassium chloride, potassium nitrate, and potassium sulfate were used as sources of potassium. In the case of potassium sulfate, an additional volume of 1.0 ml was used. The tubes were stoppered and shaken overnight and then placed in a closed desiccator containing some water to provide a humid atmosphere inside. The desiccator was placed for a week in the oven either at 60 °C or at 30 °C. After the lapse of the incubation period, the tubes were centrifuged. The supernatant solutions were filtered, their volumes measured, and the solutions analyzed for potassium. The remaining soil materials were then extracted three times by 6.5 ml of 1 N ammonium acetate, pH 7.0. After each extraction, the soil material was resuspended by properly mixing the reagent and then shaken for ten minutes before recentrifuging. The three extracts were collected together and were made up to 20 ml volume in graduated tubes. Potassium in solutions and extracts was determined by flame photometer, type AAS 1 by Carl Zeiss Jena (GDR).

Exchangeable potassium was calculated from extracted potassium after subtracting from the latter the potassium contained in the equilibrium solution remaining inside with the extracted soil material. Fixed potassium (K_F) was calculated according to the following formula:

$$K_F = K_A + K_{XC} - K_{XT}$$

where K_A = added potassium

K_{XC} = sum of soluble (K_S) and exchangeable (K_E) potassium of the control soil sample

K_{XT} = sum of K_S and K_E of the potassium-treated soil sample.

Added and exchangeable K were expressed on an oven-dry soil basis. Soluble K mentioned or tabulated in this work, however, was always expressed on the solution basis.

To test for the possible effect of microorganisms, one part of potassium nitrate-treated soil samples was sterilized by steam at a pressure of about two atmospheres. Otherwise, all laboratory operations used are the same as previously stated. Soils were found to have normal microflora before sterilization and practically none after it, using the serial dilution method.

3. Results and discussion

The sorption of potassium shown in table 2 was initially fast and then changed very little after 7 days of contact. In other soils, important changes in fixed potassium were observed with time (*Anne and Marocke, 1964*); such observations, perhaps, are due to differences in the nature of the soils used. The period of 7 days was thus chosen as incubation time.

Table 2. Sorbed potassium (me/100 g) in relation to time of equilibration at different levels of potassium added in 1:1 soil: water suspensions of Ameriya silty clay loam

Added potassium (me/100 g)	Days of contact				
	4	7	15	30	64
0.128	0.041	0.046	—	0.043	0.050
0.256	0.151	0.155	0.251	0.144	0.158
0.512	0.362	0.354	0.358	0.352	0.348
0.768	0.576	0.572	0.572	0.572	0.568
1.280	0.876	0.906	0.916	0.896	0.946
2.560	1.878	1.898	1.908	1.898	1.888

Table 3. Effect of moisture, temperature, amount of added potassium, and kind of associated anion on the soluble, exchangeable, and fixed potassium in Ameriya silty clay (closed system)

Added potassium (ppm)	Soluble potassium (ppm)		Exchangeable potassium (ppm)		Fixed potassium (ppm)	
	KCl	KNO ₃	KCl	KNO ₃	KCl	KNO ₃
<i>At 150% moisture</i>						
0	19.2	21.3	341.6	360.6	347.4	381.8
764	95.2	99.4	820.4	895.6	907.5	870.2
1527	242.6	242.1	1230.9	1177.1	1275.9	1419.5
3055	645.4	689.5	1970.8	1853.6	1832.3	2242.3
<i>At 100% moisture</i>						
0	24.6	24.4	345.7	360.1	369.3	369.7
509	80.7	87.6	699.5	730.5	752.4	713.4
1018	174.3	179.7	986.2	977.9	1019.4	1113.0
2037	508.6	515.5	1588.2	1505.2	1458.1	1701.9
<i>At 75% moisture</i>						
0	29.4	25.2	349.4	366.8	316.8	354.6
382	80.7	76.5	612.1	673.2	665.0	616.3
764	162.3	147.2	808.3	847.7	854.6	833.5
1527	380.6	396.0	1346.8	1278.4	1414.4	1442.5
<i>At 50% moisture</i>						
0	—	—	—	—	—	—
255	—	—	—	—	—	—
509	—	—	—	—	—	—
1018	—	—	—	—	—	—

All values except those of soluble potassium, which were expressed in ppm on solution basis, are expressed in ppm on the oven-dry soil basis. S denotes the use of sterilized soil samples. NS denotes the use of unsterilized soil samples.

The potassium fixation data obtained in this work are shown in table 3. The net ranges and averages as per cent of K_A , which were higher than their respective controls and for all treatments at each temperature used, are shown in table 4 for the various potassium forms.

Table 4. Net ranges and averages as per cent of K_A for all treatments

K form	30 °C		60 °C	
	Range	Average	Range	Average
K_S	9.95-23.76	16.49	9.74-29.19	15.90
K_E	59.92-74.01	66.01	48.61-84.16	64.97
K_F	9.34-23.70	16.41	8.77-28.93	18.13

The average amounts fixed at 60 °C were not very much higher than those at 30 °C, but when the K_F sets of KCl-treated suspensions at 30 °C and 60 °C were compared by the "t" test, differences between values of the two sets were found to be highly significant. The same was found for their corresponding K_E , but their K_S values had significant differences only at the 5 % probability level. As reflected by these averages, the increase in temperature resulted in an increase in K_F accompanied by a decrease in K_E and to a lesser degree in

Table 5. Properties of lines and curves of relations for various forms of potassium

Treatment and variables	Type of equation ¹	a	b	c	r
<i>KCl, 30 °C</i>					
$y = K_F, x = K_S$	1	0.358	101.19	-	0.8003
	2	18.415	0.0056	-18.777	0.9346
$x = K_E$	1	0.140	29.17	-	0.8084
	2	19.236	0.0092	-20.362	0.8838
$x = K_A$	1	0.082	86.87	-	0.8107
	2	17.592	0.0066	-18.221	0.9287
$y = K_E, x = K_S$	1	2.516	519.85	-	0.9778
<i>KCl, 60 °C</i>					
$y = K_F, x = K_S$	1	0.500	113.37	-	0.9155
	2	33.648	0.0042	-34.166	0.9592
$x = K_E$	1	0.233	-4.83	-	0.9434
	2	11.874	0.0196	-13.346	0.9579
$x = K_A$	1	0.123	88.22	-	0.9418
	2	167	0.2164	-510	0.9730
$y = K_E, x = K_S$	2	61.316	0.529	55.756	0.99073
<i>KNO₃, 60 °C</i>					
$y = K_F, x = K_S$	1	0.679	41.37	-	0.9226
$x = K_E$	1	0.394	-210.56	-	0.9492
$x = K_A$	1	0.179	-1.53	-	0.9337
<i>K₂SO₄, 60 °C</i>					
$y = K_F, x = K_S$	1	0.909	22.95	-	0.9248
$x = K_E$	1	0.254	-93.33	-	0.9556
$x = K_A$	1	0.160	11.20	-	0.9728
<i>All salts at 60 °C</i>					
$y = K_F, x = K_S$	1	0.658	59.62	-	0.9044
$x = K_E$	1	0.296	-107.66	-	0.9084
$x = K_A$	1	0.162	21.78	-	0.9471

¹ 1: $y = ax + b$

2: $y = ax^b + c$

All calculations were done without the $K_S, K_E,$ and K_F values of the control samples ($K_A = 0$).

K_S . Such a redistribution indicates that the main direct source of K_F was K_E . The probable main movement of K_A to the fixing sites could be visualized to take place in two steps: The first from K_S to K_E , and the second from K_E to K_F . It should be noted, however, that such a scheme for potassium fixation does not contradict the co-existence of other ways of potassium fixation such as the precipitation of potassium as insoluble compounds (Schuffelen and Marel, 1955). The above scheme simply appears to be the main one operative in the soil system studied. Table 5 shows the relation between K_F and K_A, K_S, K_E for the KCl-treated suspensions at 30 and 60 °C. Although these relations were found to fit straight-line equations ($y = ax + b$) with highly significant correlation coefficients, they were, nevertheless, found to fit the parabolic equation $y = ax^b + c$, where a, b and c are constants, with higher positive correlation coefficients; this signifies a better fitness for the parabolic function. A study of these relations shows that at both temperatures, 30 and 60 °C, K_F increased with decreasing rates as each of the variables K_A, K_S and K_E increased. At any level of each of the latter variables, K_F at 60 °C was higher than that at 30 °C; the difference being greater at higher levels of the variables. Furthermore, at 60 °C, the stated K_F relations fitted the straight-line equation better than corresponding ones at 30 °C, as can be judged from the calculated correlation coefficients (table 5). This observation signifies that the K_F relations at 60 °C, especially K_F vs. K_A and K_F vs. K_E , are closer to straight lines than the corresponding ones at 30 °C. Moreover, highly significant correlation coefficients were obtained for the straight-line equations fitting the K_F data at 60 °C in relation to $K_A, K_S,$ and K_E , when the latter three variables were pooled together irrespective of kind of salt or the moisture level of the soil suspensions (fig. 1).

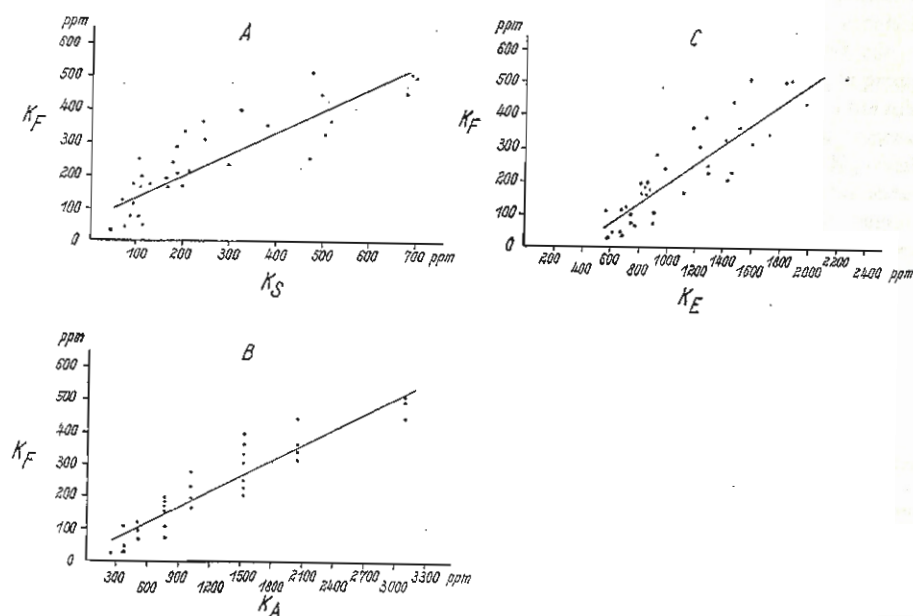


Fig. 1. Fixed potassium, K_F , at 60 °C irrespective of moisture level and kind of added salt in relation to corresponding:

A. soluble potassium, K_S

B. added potassium, K_A

C. exchangeable potassium, K_E

Differences between the K_F versus K_S , K_B and K_A relations at 30 °C and those at 60 °C can be explained mainly on the basis of temperature influences on potassium mobility and on the physical properties of the soil. The increase in temperature, apparently, produced a physical condition conducive to potassium movement and diffusion inside and between soil aggregates and particles. Dissolution and dislocation of lime, gypsum, organic compounds, and other aggregate-binding materials, that could have been caused by aqueous solution and evolved gases at 60 °C, may have resulted in rather open, swollen and perhaps degraded soil aggregates. Such an effect of a temperature increase on the physical condition of soil material can expose new solid surfaces with K-unsaturated fixing sites. With increased rates of reactions and an increased potassium diffusion at 60 °C, the transfer of potassium from the solution phase to the newly fixing sites would be facilitated conceivably. The higher levels of K_A used in this fixation study were apparently sufficient at the prevalent experimental conditions to nearly saturate at 30 °C what were available from these K-fixing sites, and hence the tendency of K_F to level off at higher values of the variables K_S , K_A , and K_B . At 60 °C, on the other hand, there was a greater decrease of potassium as well as better conditions for its transfer so that no such tendency was feasible at the higher levels of these variables. Instead, these relations at 60 °C approach straight lines (fig. 1).

The effect of moisture on the potassium fixation (table 3) appears to be very limited in the moisture range used. This result is generally in line with the observations of *Abmad* and *Davis* (1970), who found practically no influence of moisture in the range of 0.5 to 1.6 of the field capacity on the level of fixed potassium.

The "t" test comparisons between each of the K_F , K_S , and K_B sets of values of the sterilized potassium nitrate-treated suspensions and those of the unsterilized ones showed no significant differences.

As regards the effect of different anions contained in the potassium salts added at 60 °C on the potassium forms, it may be said that it was generally limited to the lower levels of K_A . Fig. 2 shows that the K_S curve for the KCl-treated suspensions was almost identical with

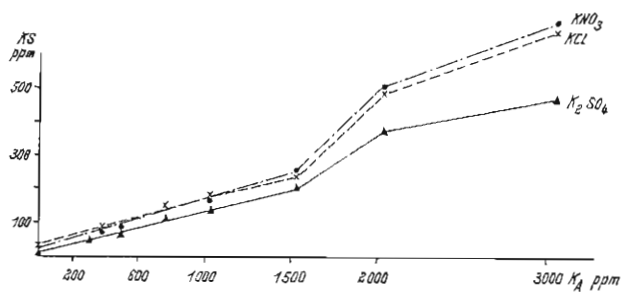


Fig. 2. Soluble potassium, K_S , in relation to added potassium, K_A , derived from different salts at 60 °C

that of the KNO_3 -treated ones, throughout the whole range of K_A . The K_S values of K_2SO_4 -treated suspensions, however, were somewhat lower than comparable values of KCl and KNO_3 -treated suspensions, especially at the three highest levels of K_A . This behaviour was reflected but to a lesser degree in the K_B values, which were generally higher in K_2SO_4 -treated suspensions than those of KNO_3 and KCl ones (fig. 3). In Indian soils, however, K_2SO_4 -treated suspensions were found to have a lower K_B than those of corresponding KNO_3 - and KCl-treated suspensions (*Misra* and *Shanker*, 1971). "t" test comparisons of

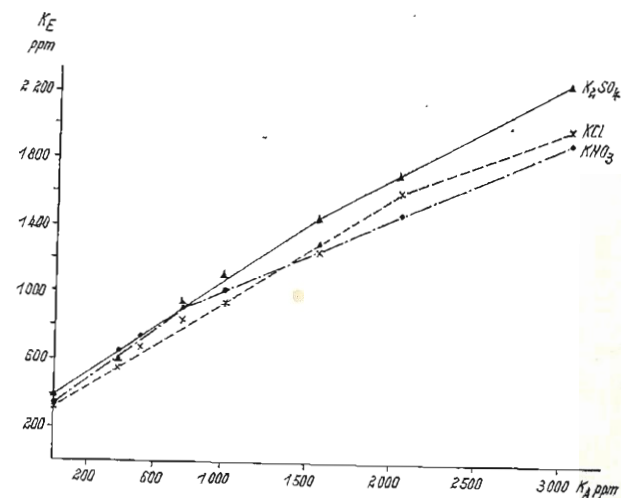


Fig. 3. Exchangeable potassium, K_B , in relation to added potassium, K_A , derived from different salts at 60 °C

sets of the K_F values of the three salts used showed, however, that the anion effect on the K_F values was non-significant. This result is different from that obtained in similar work (*Abmad* and *Davis*, 1970), where KNO_3 - and K_2SO_4 -treated suspensions had comparatively less K_F than KCl-treated ones. It is very likely that the prevalence of ionized, adsorbed, and combined calcium as carbonates and gypsum in Ameriya soil promoted the precipitation of calcium sulfate compounds, especially at the highest three levels of potassium sulfate, added, thus resulting in higher K_B and lower K_S when compared with the corresponding forms derived from added KNO_3 and KCl solutions. Differences in K_S and K_B obtained in this work in the K_2SO_4 -treated suspensions were apparently too small to be reflected in the K_F values. The formation of the ion KSO_4^- appears to be too small in magnitude (*Adams*, 1971) to account for the observed potassium distribution in the K_2SO_4 -treated soil system.

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Summary

Taba Naji, W. Römer, and G. Schilling: Wet fixation of potassium in an Iraqi soil, Ameriya silty clay loam

The wet fixation capacity of K was studied under varying conditions in an alkaline, calcareous, and alluvial Iraqi soil. The variations concerned the soil-water proportion, K concentration, type of K salt added, and temperature, with the time for equilibrium calibration remaining constant at seven days. K fixation increases with increased K addition, i. e. with an increased content of water soluble and exchangeable potassium, but also with an increase in temperature from 30 to 60 °C. Increasing the temperature changes the relation between the level of the K addition and its fixation. Factors like the relation between soil and solution, type of K salt, and microbial activity do not exert any remarkable influence on the fixation.

Taba Naji, W. Römer und G. Schilling: Naßfixierung von Kalium eines irakischen Alluvialbodens (Ameriya, lehmiger Tonboden)

Ein alkalischer karbonathaltiger Alluvialboden aus dem Irak wurde unter verschiedenen Bedingungen auf sein Vermögen zur nassen K-Fixierung untersucht. Variiert wurden Verhältnis Boden zu Wasser, K-Konzentration, Art des zugegebenen K-Salzes und Temperatur bei gleichbleibender Zeit für die Gleichgewichtseinstellung von sieben Tagen. Die K-Fixierung steigt mit der K-Zugabe, d. h. mit der Erhöhung des Gehaltes an wasserlöslichem und austauschbarem Kalium, aber auch mit der Erhöhung der Temperatur von 30 auf 60 °C. Die Temperaturerhöhung verändert die Beziehung zwischen Höhe der K-Zugabe und Fixierung. Die Faktoren Verhältnis Boden zu Lösung, Art des K-Salzes und mikrobiologische Aktivität sind praktisch ohne Einfluß auf die Fixierung.

Taba Naji, W. Römer и G. Schilling: Мокрая фиксация калия в одной иракской аллювиальной почве (Америя, глина)

Щелочная, содержащая карбонат аллювиальная почва из Ирака исследовалась в различных условиях опыта на способность к мокрой фиксации калия. Варировалось соотношение почвы и воды, концентрация калия, вид калийной соли и температура при постоянности времени достижения состояния равновесия 7 дней. Фиксация возрастает с дозой калия, т. е. с увеличением содержания водорастворимого и обменного калия, а также с возрастанием температуры с 30 до 60 °C. Повышение температуры изменяет соотношение между величиной внесённого калия и его фиксацией. Факторы как соотношение почвы и раствора, вид калийной соли и микробиологическая активность практически не влияют на фиксацию калия.

Taba Naji, W. Römer et G. Schilling: Fixation par voie humide de potassium d'un sol alluvial irakien (Ameriya, sol argileux glaiseux)

Un sol alluvial alcalin carbonaté de l'Iraq a été examiné, dans des conditions différentes, en vue de sa capacité de fixation par voie humide de potassium. Ont été variés le rapport sol eau, la concentration de K, le genre du sel de potasse additionné et la température à temps constant pour la mise en équilibre de sept jours. La fixation de potassium augmente avec l'addition de K, c'est-à-dire avec l'augmentation de la teneur en potassium soluble dans l'eau et échangeable, mais aussi avec l'augmentation de la température de 30 à 60 °C. L'augmentation de la température change le rapport entre la quantité de l'addition de K et la fixation. Les facteurs rapport sol solution, genre du sel de potasse et activité microbiologique restent pratiquement sans influence sur la fixation.

Taba Naji, W. Römer y G. Schilling: La fijación en húmedo de potasio de un suelo aluvial iraquí (Ameriya, suelo arcilloso limoso)

Un suelo aluvial alcalino conteniendo carbonato del Irak se analizó bajo condiciones diferentes a su capacidad de fijación de K en húmedo. Se varió la relación suelo a agua, la concentración de K, el tipo de sal potásica y la temperatura manteniendo invariable el tiempo para la regulación del equilibrio de siete días. La fijación de K aumenta con la aplicación de K, es decir, con el aumento del contenido en potasio soluble en agua y inter-

cambiable, pero también con el aumento de la temperatura de 30 a 60 °C. El aumento de la temperatura modifica la relación entre la importancia de la aplicación de K y la fijación. Los factores relación del suelo a la solución, tipo de la sal potásica y actividad microbiológica prácticamente no tienen influencia sobre la fijación.

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