



Potassium availability and mineralogical relationships of selected Montana soil clays and silts
by Steven Jake Phillips

A thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of
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Abstract:

Soil clay mineralogical relationships of K availability were evaluated by chemical analysis, X-ray diffraction and biological extraction from 18 selected soil locations in eastern and western Montana.

Montmorillonite-vermiculite, mica, kaolin, quartz, plagioclase, and K-feldspar were identified by X-ray analysis in all soils. Allo-phane was detected in one western Montana soil. The 10 Å mica component of all eastern Montana soil clays was found to increase in peak amplitude by K-saturation at the expense of the 14 Å component.

Quantitative analysis of K fixation, percent montmorillonite and percent vermiculite of soil clays was determined by evaluating the cation exchange capacity before and after K fixation.

Potassium availability as determined by NH₄ OAc extraction was negatively correlated with cation exchange capacity, percent vermiculite in clay, percent sand, and K fixation. However, available K was positively correlated with percent clay. A decrease in the quantity of montmorillonite-vermiculite relative to mica was found to correspond to an increase in available K.

Soil K extraction by fungi was initiated in order to evaluate the release of K in soil minerals. Untreated soils, soils alternately wetted and dried, and silt and clay fractions were evaluated. Alternately wetted and dried soils were found to decrease in the amount of K extracted as opposed to the untreated soil. Approximately 2.5 times as much K was extracted by fungi in the clay fraction as compared to the silt, suggesting an increase in K release with decreasing particle size. Potassium extraction by fungi caused a transformation of mica to interstratified mica-vermiculite. The vermiculite component at 14 Å increased in peak amplitude as the mica component at 10 Å decreased in peak amplitude and sharpness.

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STEVEN JAKE PHILLIPS

A thesis submitted to the Graduate Faculty in partial
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Soils

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ABSTRACT

Soil clay mineralogical relationships of K availability were evaluated by chemical analysis, X-ray diffraction and biological extraction from 18 selected soil locations in eastern and western Montana.

Montmorillonite-vermiculite, mica, kaolin, quartz, plagioclase, and K-feldspar were identified by X-ray analysis in all soils. Allophane was detected in one western Montana soil. The 10 Å mica component of all eastern Montana soil clays was found to increase in peak amplitude by K-saturation at the expense of the 14 Å component.

Quantitative analysis of K fixation, percent montmorillonite and percent vermiculite of soil clays was determined by evaluating the cation exchange capacity before and after K fixation.

Potassium availability as determined by NH_4OAc extraction was negatively correlated with cation exchange capacity, percent vermiculite in clay, percent sand, and K fixation. However, available K was positively correlated with percent clay. A decrease in the quantity of montmorillonite-vermiculite relative to mica was found to correspond to an increase in available K.

Soil K extraction by fungi was initiated in order to evaluate the release of K in soil minerals. Untreated soils, soils alternately wetted and dried, and silt and clay fractions were evaluated. Alternately wetted and dried soils were found to decrease in the amount of K extracted as opposed to the untreated soil. Approximately 2.5 times as much K was extracted by fungi in the clay fraction as compared to the silt, suggesting an increase in K release with decreasing particle size. Potassium extraction by fungi caused a transformation of mica to interstratified mica-vermiculite. The vermiculite component at 14 Å increased in peak amplitude as the mica component at 10 Å decreased in peak amplitude and sharpness.

INTRODUCTION

Potassium (K) in agricultural soils is usually the most abundant of the plant nutrient elements. During the growing season an appreciable amount of K becomes available to plants. The amount that is not available and not exchangeable resides in the K-bearing minerals of the soil. It has long been established that one of the primary sources of K in soils, available for plant growth, is layer silicate minerals. A precise correlation between K availability, cation exchange properties, and K fixation, however, has not been totally established. This is attributed to the uncertainty of identification of micaceous and mica-like minerals which may contribute to differential behavior with respect to K exchange phenomena. To attain an understanding of K reactions in soils, it is advantageous to study the naturally occurring soil minerals including micas, vermiculites, smectites, and K-feldspars. This study was initiated with three principle objectives: 1) to correlate K-availability with soil mineralogical relationships of selected eastern and western Montana soils; 2) to determine the mineralogical effect of K saturated and K depleted soil clays; and, 3) to determine the availability of mineral K to growing fungi.

LITERATURE REVIEW

Potassium and Soil Mineralogical Factors

Potassium is normally found in great abundance in agricultural soils. The lithosphere contains approximately 2.3% K (Ernst, 1969). Potassium in most mineral soils ranges from approximately 0.05 to 3.5%. Most agricultural soils, however, average between 1 and 2% in the upper 6 inch depth (Jackson, 1964). Potassium in agricultural soils, for the most part, is derived from K-bearing feldspars and micaceous minerals. Feldspars and micas constitute greater than 63 percent of igneous rocks and 27 percent of sedimentary rocks (Ernst, 1969; Jackson, 1964).

Potassium-feldspars are tectosilicates consisting of $(\text{Si},\text{Al})\text{O}_4$ tetrahedra linked in all directions through the O of adjacent tetrahedra. The tetrahedra are arranged in horizontal rings. These rings have been distorted, i.e., tilted and rotated from the horizontal (Deer et al., 1963). The resultant tetrahedral configuration contains large interstices which contain K ions. The tetrahedra commonly contain Si and Al ion substitutions for Si. Where a local negative charge occurs, caused by Al substitutions, it is compensated for by the positive charge of interstitial cations. The idealized formula for K-feldspars is KAlSi_3O_8 , indicating approximately 25 percent substitution of tetrahedral Al for Si (Jackson, 1964).

K-feldspar polymorphs include: sanadine, microcline, adularia, and orthoclase.

Micas are common constituents of most soils. The basic structural units as proposed by Deer et al. (1963) are hexagonally linked cation-O tetrahedra and OH-cation-O octahedra. Individual tetrahedra consist of a central cation and four coordinating O's. The octahedra consist of a central cation and 6 coordinating OH or O's. Apical O's from adjacent tetrahedra are shared by the octahedral layer providing a linkage between layers. Replacement of Si for Al via isomorphous substitution in the tetrahedral layer causes a negative charge which is then balanced by interlayer cations including K.

Two general types of micas occur. Dioctahedral micas are those having two thirds of the octahedral positions filled with trivalent cations, usually Al. Trioctahedral micas are those in which all of the possible octahedral positions are occupied primarily by divalent cations.

The idealized formulas for muscovite, biotite, and illite are $K_2Al_2Si_6Al_4O_{20}(OH)_4$, $K_2Al_2Si_6(Fe^{+2},Mg)_6O_{20}(OH)_4$, and $K_{1-1.5}(Si_{8-y},Al_y)(Al_4,Fe_4,Mg_4,Mg_6)O_{20}(OH)_4$, respectively (Grim, 1968). Muscovite and biotite are normally derived from igneous rocks, whereas illite is derived from sediments (Grim et al., 1937).

Grim (1968) suggests that all gradations can exist between illite, muscovite, and biotite, and illite and montmorillonite. It

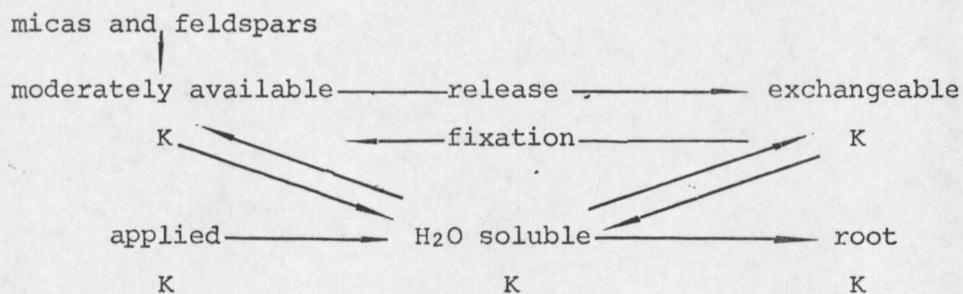
has also been suggested that illites are randomly interstratified mica montmorillonites, mica vermiculites, mica chlorites, or intergrades of any number of these mineral assemblages (Jackson et al., 1952; Mehra and Jackson, 1959). Illites differ from well-crystallized micas in that there is less substitution of Al for Si. As a consequence of less substitution, the Si to Al molecular ratio of illite is higher than that of more crystalline micas and the charge deficiency is reduced from 2 per unit cell to approximately 1.3 (Grim, 1968).

Potassium of layer silicates in the interlayer position is accommodated in pseudo-hexagonal or ditrigonal voids formed by the basal tetrahedral atomic configuration. With proxying of cations of different size and valance, distortion of the lattice occurs. When the tetrahedral layer is relatively larger than the octahedral layer, individual tetrahedra rotate and tilt from the idealized horizontal planes in order to accommodate local charge balance (Radoslovich and Norrish, 1962). The resultant basal tetrahedral voids become ditrigonal and the interlayer K coordination number is 6, consisting of 3 O's from adjacent mica layers. Coulomb's law states that for electrostatic charge, the strength varies as to the reciprocal of the distance squared between the charge and the ion. In tetrahedrally substituted clays, the distance between the K ion

and the lattice charge is approximately half that of the octahedrally substituted clays. Thus, the electrostatic force between the K ion and the tetrahedral charge is four times that of the octahedral charge. Radoslovich and Norrish (1962) concluded that K adsorption onto lattice silicates was favored by the negative charge originating in the tetrahedral layer. It was also concluded that high negative charge density favored K adsorption.

Forms of Potassium and Potassium
Equilibria

Potassium equilibrium in the soil and K availability to plants is dependent, not only on the total amount of K present, but also the stage of mineral weathering and the chemical relationship of various K forms. Jackson (1964) suggests the following reactions for the chemical relationship of soil K:



Attoe and Truog (1945) concluded that 97 to 98 percent of the soil K exists in the mineral form. They also concluded that

approximately 90 percent of the available K exists in the exchangeable form. Jackson et al. (1952) indicated that K occupies interlayer positions in mica is difficultly available and release occurs when the charge of the layer is reduced and the interlayer position expands.

The immediate sources of K to plants are water soluble and exchangeable (Jackson, 1964). Nonexchangeable and strongly adsorbed K are of considerable importance to plant nutrition during the growing season. The equilibrium level of K becomes unbalanced during the growing season by K removal in the available form by plants. However, K is equilibrium quickly replenished when growth stops and nonexchangeable forms of K from K-bearing minerals become available.

Wood and DeTurk (1940) observed that fixation of K occurred when added to Illinois soils stored under moist conditions. With no K additions, these soils either fixed or released K depending on the specific soil K equilibrium level. Bolt et al. (1963), Cook and Hutcheson (1960), and Dowdy and Hutcheson (1963) have proposed an exchange equilibrium level in most agricultural soils to approximate 0.5 meq per 100 g. Potassium release from the nonexchangeable form usually maintains a somewhat constant level (Berland et al., 1950; Smith and Matthews, 1957; MacLean, 1961; Matthews and Sherrell, 1960).

According to Moss (1963), an increasing dilution of the soil water solution facilitated a greater increase in water soluble K.

Many soils have a different value for exchangeable K if the displacing solution is added to field-moist as opposed to air-dried samples. Higher values are usually found in air-dried samples. However, the opposite may occur especially in soils high in exchangeable K. Luebs et al. (1956) and Hanway et al. (1961,1962) conducted a large number of field experiments in North Central United States and obtained a higher correlation between uptake of exchangeable K by plants on undried soils as opposed to samples air-dried before analysis. These researchers concluded that analysis of exchangeable K should be on field-moist samples if used as an indication of availability to plants or of exchangeable K in the field.

The law of mass action indicates that the amount of K released from mineral forms at equilibrium increases with increased concentration of cations other than K in solution. In addition, K release will occur with an increase in the volume of solution and with a decrease of K in the solution before the reaction (Scott and Smith, 1966).

The difference between the cation exchange capacity and the charge density for montmorillonite, muscovite, and vermiculite is 0, 230, and 50 meq per 100 g., respectively (Kardos, 1964). This is the amount of K not exchanged by normal extraction procedures.

Potassium Release

Potassium release from nonexchangeable forms is primarily a cation exchange reaction. Mortland et al. (1956) concluded that a reduction in the percentage of K from biotite after 4 successive crops of wheat increased the cation exchange capacity and was attributed to K removal by plants. Cations other than K were thought to satisfy the negative charge of the mineral layers and consequent spreading of the layers involved water entry into the interlayer position.

The vertical distribution of micaceous minerals in soils verifies the loss of interlayer K from micas. Normally, micas of sand and silt size increase with depth. The inverse is true of vermiculite. Soil vermiculite is presumably a weathering product of K micas originally present throughout the soil profile. Rich (1958) using X-ray diffraction intensities, reported that vermiculite decreased relative to mica with depth and also decreased with successively large particle size.

Conyers and McLean (1968) using X-ray analysis reported a decrease in the 10, 5, and 3.3 Å peak intensities and an increase in the intensities of the 17 and 14 Å peaks of plant degraded layer silicates. Degradation of illite to expanding layer silicates, i.e., vermiculite and smectite, was suggested due to the removal of interlayer K.

Attoe (1946) reported that air-dried samples not fertilized with K increased in exchangeable K in 9 out of 19 soils analyzed. With K fertilization, fixation occurred in 8 out of 10 samples stored in a moist condition and when air-dried, fixation occurred in all samples.

Rich (1964) and Rich and Black (1964) concluded that K removal was pH dependent. An inverse relationship was found to exist between K release and pH, i.e., K release is increased as the pH is lowered. The hydronium ion (H_3O) was reported to be more effective in replacing K than larger hydrated ions, however, after K replacement H_3O ions were rapidly replaced by other cations allowing for lattice expansion.

Potassium Fixation

Fixation of K is the conversion of the exchangeable form to the nonexchangeable form. For the most part, K fixation is a property of expanded layer-silicates. In these minerals, K is held electrostatically between layers outside the aluminosilicate structural network as a charge balancing ion subject to fixation in the ditrigonal voids in nonexchangeable form.

Potassium fixation occurs in soils containing vermiculite and vermiculite-like minerals under moist conditions, and the fixation is a consequence of prior K removal. This was demonstrated by

Mortland (1961) using mixtures of biotite and Na-saturated layer silicates. It was found that the initial K content in the layer silicates was inversely proportional to the amount of K fixed.

Luebs et al. (1956) found that K fixation was dependent on relative humidity. Soils of different H₂O vapor content were analyzed, and it was demonstrated that an increase in relative humidity caused a decrease in exchangeable K and the inverse was true of decreased relative humidity.

Potassium in Particle Size Fractions

Cook and Hutcheson (1960) reported that the total K content of the Ap, A₂ and C horizons of selected Kentucky soils decreased as the particle size decreased. Illite was found to be the predominant layer silicate mineral in the coarse fractions and less predominant in the fine fractions.

Smith et al. (1968) treated several silt and clay size fractions of Marshall soil with sodium chloride-sodium tetraphenylboron (NaTPB). It was found that as the particle size increased, the amount of K extracted increased reaching a maximum at the 20 μ equivalent spherical diameter (e.s.d.) size fraction, then decreased as the particle size was further increased. Scott (1968) analyzed micaceous minerals as to susceptibility of interlayer K to exchange

with NaTPB. It was found that as the particle size decreased from 50 to 0.08 μ (e.s.d.), the rate and the amount of K exchange increased then decreased. The maximum exchange rate was found to be considerably higher in the 0.08 to 20 μ (e.s.d.) fraction than in the 20 to 50 μ (e.s.d.) fraction.

MATERIALS AND METHODS

Potassium in Nitrogen Topdressing Study

During the years 1970 and 1971, spring N-topdressing studies were conducted at several locations throughout eastern Montana (east of the Continental Divide). Potassium-topdressing in the form of KCl applications were included in the study in order to determine the predictability of K response and to obtain qualitative and quantitative information regarding K deficiency for winter wheat and for correlation of soil properties. The study sites were seeded with Winalta, Cheyenne, Froid, Warrior, or Itana varieties of winter wheat (*Triticum aestivum* L.). Grain yield and response data for eastern Montana locations are listed in Table 1.

Soil Descriptions

Soil descriptions and classifications were obtained from the United States Soil Conservation Service, Bozeman, Montana.^{1/}

Accl soil at location 1 is a silty clay loam classified as a member of the fine, montmorillonitic family of Abruptic Aridic Argiborolls. The soil profile consists of deep alluvium over glacial

^{1/}Personal communication, Shelby Brownfield, Soil Scientist (Correlator); soil classification, a comprehensive system-7th approximation. U.S. Dept. Agr. U.S. Govt. Printing Office, Washington, D.C.; established series revised memoranda.

Table 1. Grain yield and potassium response of eastern Montana locations.

Location No.	Series	Grain yield (kg/ha)			K response (kg/ha)	
		treatment 40-40-0	treatment 40-40-40	treatment 40-40-100	maximum	average
1.	Acel	1730.40	2427.93	2222.97	697.5	595.4
2.	Scobey	1486.46	1384.99	1427.33	-101.5	- 80.6
3.	Marias	2513.28	2526.72	2674.56	161.3	87.4
4.	Bainville	2271.36	2493.12	2506.56	235.2	228.5
5.	Cherry	2358.72	2485.40	2365.44	120.9	63.8
6.	Devon	1695.45	1714.94	1745.30	47.0	32.3
7.	Dooley	873.60	1041.60	1182.72	309.1	238.6
8.	Williams	2983.68	3120.09	2984.35	154.6	68.5
9.	Giltedge	2984.35	3177.21	3105.98	166.0	177.4
10.	Amsterdam	3128.83	3307.58	3175.20	178.8	112.9
11.	Coburn	1339.96	1663.20	1548.96	323.2	266.1
12.	Absarokee	2460.86	3042.81	3049.53	588.7	585.3
13.	Cargill	1064.44	1342.65	1412.54	348.1	313.2
14.	Scobey	2921.85	3121.44	3013.92	199.6	145.8
15.	Assinniboine	2974.27	3024.81	3157.72	183.5	126.3

till. Dark green shale and siltstone of the Colorado Formation underlie the solum.

Scobey soil at location 2 and 14 is a clay loam classified as a member of the fine, montmorillonitic family of Typic Argiborolls. The lithology of the profile and substrate resemble that of the Acel series.

Marias soil at location 3 consists predominantly of clay sized minerals of the fine, montmorillonitic, calcareous, frigid family of Ustertic Torriorthents. The soil consists of alluvial clays derived from the Colorado Formation.

Bainville clay loam at location 4 is a member of the fine-silty, mixed mesic family of Ustic Torriorthents. The parent material is composed of interbedded shaly clay, mudstone and sandstone derived from the Hell Creek Formation.

Cherry soil at location 5 is a silty clay loam classified as a fine-silty, mixed family of Aridic Haploborolls. The substrate is composed of loamy and silty shales and siltstones of the Fort Union Formation.

Devon soil at location 6 is a loam classified as a member of the fine-loamy, mixed family of Aridic Argiborolls. The regolith of the Devon series is similar to that of the Cherry series.

Dooley at location 7 consists of fine sandy loam classified as a fine-loamy, mixed family of Typic Argiborolls. The profile is underlain by gravels, sand and silt of the Flaxville Gravels.

Williams heavy loam at location 8 is classified as a member of the fine-loamy, mixed family of Typic Argiborolls. The bedrock consists of clay, shale and sandstone of the Fort Union Formation.

Giltedge at location 9 is a silty clay loam classified as a fine, montmorillonitic, mesic family of Ustollic Natrargids. The lithology of the substrate consists of gravels, sand and silt deposits of fluvial terrace remnants.

Amsterdam soil at location 10 is a silt loam classified as a member of the fine-loamy, mixed family of Typic Cryoborolls. The profile consists of aeolian or lacustrine loess deposited on Tertiary undifferentiated sediments.

Coburn silty clay loam at location 11 is a fine, montmorillonitic family member of Abruptic Argiborolls. The solum is underlain by deep alluvium over weathered shale. Thermopolis dark gray-green shale and sandstone underlies the soil.

Absarokee clay loam at location 12 is classified as a fine, montmorillonitic family member of Typic Argiborolls. The soil is predominantly derived from gravel, sand and silty alluvial material deposited as valley fill sediments.

Cargill soil at location 13 is a silty clay loam member of the fine-silty, mixed family of Borollic Calciorthids. The regolith is similar to that of the Acel, Marias and Scobey soils.

Assinniboine soil at location 15 consists of fine-loamy, mixed family, classified as Aridic Argiborolls. The substrate is composed of fine textured shales much like the parent material of Bainville soil.

The sample location at township 27N, range 34W at location 16 is unclassified at this time. The parent material for this soil is composed of glaciolacustrine sediments.

The sample site at township 6N, range 20W, location 17, is unnamed at this time. It is classified as a loam member of the fine-loamy, mixed family of Typic Argiborolls. The soil profile is developed over gravelly, sandy, silty valley fill sediments.

Hamilton at location 18 is a silty loam classified as a coarse-silty, mixed family member of Fluventic Haploborolls. The profile is composed of silty loess alluvium derived from valley fill sediments.

Soil Preparations

In 1972, selected soil sub-samples of the N-topdressing study experiments were obtained. In addition, 3 bulk samples from western Montana (west of the Continental Divide) were obtained and sub-samples taken. Sub-samples of these soils at 0 to 6 and 6 to 12 inch depths were oven-dried at 60°C, and passed through a 200 mesh sieve. The samples were then placed in metal containers for subsequent analysis. Sample number and appropriate locations are listed in Table 2. Sample site locations are illustrated in Figure 1.

X-ray Mineralogical Analysis

Mineral identification and mineralogical composition were determined by X-ray diffraction criteria outlined by Whittig (1965). Silt and clay separates were Mg and K-saturated and deposited on glass petrographic slides as proposed by Kittrick (1961) following glycerol solvation according to the procedure of Klages and Southard (1968).

X-ray diffractograms were obtained using a General Electric XRD5 Diffractometer with Ni-filtered Cu-radiation ($\lambda 1.5405 \text{ \AA}$). A 1° beam divergent slit, medium resolution soller slit, and a 0.1° detector slit were used. The maximum scan angle covered 60° , from 3 to $63^\circ 2\theta$. A linear recording mode of 1000 cps full scale with a 2 second time constant was used on the recording apparatus. The diffractometer and recording scan rate was 2° per minute.

Chemical Analysis

The cation exchange capacity was determined in two ways in order to quantify the amount of montmorillonite and vermiculite in the soil following a procedure outlined by Alexiades and Jackson (1965). The cation exchange capacity was determined by saturating the clay with Ca using CaCl_2 , removal of excess salts, and replacing the Ca with MgCl_2 , designated as (C E C, Ca:Mg). For determination of the exchange capacity after K fixation, the sample was saturated by washing with

Table 2. Sample No., location, series, and cooperator names of eastern and western Montana soils.

Location		Cooperator		Legal description		
No.	Series	Name	County	sec.*	twp.†	rge.‡
1.	Acel	Gray	Chouteau	36	23N	7E
2.	Scobey	N. Station	Hill	16	31N	15E
3.	Marias	Bergstrom	Pondera	31	27N	1E
4.	Bainville	Erikson	Yellowstone	11	4N	23E
5.	Cherry	Coulter	Garfield	4	19N	34E
6.	Devon	Stanton	Garfield	31	20N	35E
7.	Dooley	Brenden	Daniels	15	35N	49E
8.	Williams	Schnitzler	Sheridan	19	31N	55E
9.	Giltedge	Torske	Big Horn	7	2S	33E
10.	Amsterdam	Bates	Gallatin	1	1S	3E
11.	Coburn	Daum	Yellowstone	7	4S	26E
12.	Absarokee	Roland	Carbon	9	4S	22E
13.	Cargill	Gettel	Cascade	17	22N	1E
14.	Scobey	Holtz	Cascade	4	22N	4E
15.	Assinniboine	Erpelding	Rosebud	15	6N	41E
16.	--	W. Station	Sanders	32	27N	34W
17.	--	W. Station	Ravalli	27	6N	20W
18.	Hamilton	W. Station	Ravalli	28	7N	20W

* sec.-section † twp.-township ‡ rge.-range

