EXCHANGE OF LYOTROPIC SERIES CATIONS BY MICAEOUS VERMICULITE AND ITS WEATHERING PRODUCTS DETERMINED BY ELECTRON MICROSCOPY AND RADIOCHEMICAL ANALYSIS

Progress Report

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Abstract

Swelling of micaceous vermiculite layers in blister-like areas of lower charge, which occurred in the lyotropic series: \( \text{Li}^+ > \text{Na}^+ > \text{Be}^{2+} > \text{Mg}^{2+} > \text{Ca}^{2+} \), was found to be repressed by fixing monovalent cations. That swelling was not completely prevented was traced, by electron microscopy and electron probe analysis, to trapping of hydrated cations by fixing cations in the order \( \text{Cs}^+ > \text{Rb}^+ > \text{NH}_4^+ > \text{K}^+ \). Layer charge of biotitic vermiculites, which affects \( ^{137}\text{Cs}^+ \) fixation capacity, was unaffected by oxidation and/or reduction of 7 to 90 mmole/100 g of octahedral Fe. The layer charge change mechanism appears to involve tetrahedral \( \text{Al}^{3+} \).

The presence of \( ^{238}\text{U} \) fission particle tracks in micaceous vermiculites, observed by scanning electron microscopy, increased the diffusion rate of interlayer \( \text{K}^+ \), structural \( \text{Fe} \) (forming crusts on layers) and \( \text{Si(OH)}_4 \), selectivity of \( \text{K}^+ \) over \( \text{Ca}^{2+} \), and cation exchange capacity. Specific sorption of the divalent cations \( \text{Mg}^{2+} \), \( \text{Ca}^{2+} \), \( \text{Ba}^{2+} \) and \( \text{Sr}^{2+} \) on iron and aluminum gels occurred in the presence of \( 1\text{N NaNO}_3 \) with the release of approximately one proton per sorbed ion.

The surface meter of a basalt-derived chronosequence of Australian soils was found by oxygen isotope analysis to be \( \frac{1}{4} \) to \( \frac{1}{2} \) eolian quartz. Sorption of \( ^{90}\text{Sr}^{2+} \) and \( ^{137}\text{Cs}^+ \) was weak in these soils dominated by amorphous material, kaolinite and halloysite. New Zealand chronosequence soils and aerosolic dusts, with abundant micaceous vermiculite, strongly sorbed these isotopes.
Documentation

This progress report is submitted pursuant to Article B-XX1 of the Contract, No. AT(11-1)-1515 which has been in operation since June 1, 1965. Under this contract to April 6, 1973, 26 scientific papers have been published or accepted for publication, four additional papers have been submitted for publication, and six others are in various stages of development for publication (all listed near the end of this report).

I. Layer charge changes in relation to cation fixation.

Swelling of the layers of micaceous vermiculite in blister-like areas of lower charge, which occurred (Sridhar, Jackson, and Syers, 1972) according to the lyotropic cation series: Li⁺ > Na⁺ > Be²⁺ > Mg²⁺ > Ca²⁺, was found to be repressed by fixing monovalent cations. That swelling was not completely prevented was traced (Sridhar and Jackson, 1973), by electron microscopy and electron probe analysis, to trapping of hydrated cations by fixing cations in the order Cs⁺ > Rb⁺ > NH₄⁺ > K⁺. Layer charge of biotitic vermiculites, which affects ¹³⁷Cs⁺ fixation capacity, was unaffected by 7 to 90 mmole/100 g of octahedral Fe oxidation and/or reduction (Veith and Jackson, 1973). An OH⁻ group is deprotonated when Fe²⁺ → Fe³⁺. A phlogopite with little iron was found to weather through vermiculite to saponite involving a substantial layer charge decrease from 210 to 140 during the
process (Sridhar and Jackson, in preparation). The above indicates that iron oxidation is not the mechanism of the layer charge decrease, but rather, merely occurs concurrently. Preliminary investigations under this project revealed that layer charge decreases with the removal of tetrahedral Al$^{3+}$ in vermiculites (Sridhar and Jackson, in preparation). During the current year, research on the layer charge decrease by tetrahedral Al$^{3+}$ removal and its influence on the swelling properties has been carried out and prepared for publication (Sridhar and Jackson, 1973). Project studies (Dolcater, Jackson, and Syers, 1972) revealed that layer charge density in addition to cation charge and hydration control cation exchange selectivity (CES) properties of micaceous vermiculite. Completion of the study of layer charge decrease mechanisms during the weathering of phlogopite through vermiculite to saponite will further understanding of the cation exchange selectivity process by micaceous layer silicates (Sridhar and Jackson, in preparation).

II. Specific adsorption of divalent cations. Previous work has shown that the hydrous oxides of Fe and Al show a remarkable ability to sorb trace quantities of $^{45}$Ca$^{2+}$ and $^{90}$Sr$^{2+}$ from solutions containing a million times more Na$^+$ (Kinniburgh, Syers, and Jackson, 1973). Sorption occurred by release of protons from the weakly acidic surface OH functional groups which therefore caused the sorption
reaction to be highly pH dependent. In contrast to non-specific adsorption, most of the cation sorption occurred while the solids still possessed a net positive charge (Kinniburgh, Syers and Jackson, 1973). Furthermore, since approximately one $\text{H}^+$ was released for each $\text{Ca}^{2+}$ or $\text{Sr}^{2+}$ sorbed, the net positive charge of the surface is further increased by sorption.

It seems that the results of this study can be generalized to both a wide range of divalent cations (Kinniburgh, Jackson and Syers, in preparation) and a wide range of naturally occurring soil minerals. The specific adsorption of the transition and heavy metal cations occurs at an appreciably lower pH than that of $\text{Ca}^{2+}$ and $\text{Sr}^{2+}$, and this has important implications to the mobility and availability of these ions in soils. Somewhat surprisingly, the hydrous oxides appear to serve as a good model for the specific adsorption of many divalent cations on phyllosilicates, such as montmorillonite. This is presumably because those cations are sorbed on the broken edges, defects and coatings of these phyllosilicates and such sites resemble the model hydrous oxides studied in this project. Mainly ferruginous sesquioxide coatings on micaceous vermiculite surfaces appeared in the early stages of soil development (less than 250 years) and increased in thickness and amount in the older soils of a chronosequence of soils developed in greywacke and mica-
schist materials of New Zealand (Mokma, Jackson, Syers, and Stevens, 1973). Redistribution of iron oxides was indicated by the increase in the quantity of discrete goethite particles in the older soils. The amount of $^{90}\text{Sr}^{2+}$ sorbed varied within a relatively narrow range but that fixed against replacement by $\text{Ca}^{2+}$ in 0.01N CaCl$_2$ varied over a much wider range. The tendency for a decrease in the amount of $^{90}\text{Sr}^{2+}$ sorbed and fixed with increasing age in the older soils indicates that the free iron oxides which sorb and fix $\text{Sr}^{2+}$ increase in crystallinity and become less active. Removal of ferruginous sesquioxides tended to decrease the extent of sorption and fixation of $^{90}\text{Sr}^{2+}$ but increased the extent of sorption and fixation of $^{137}\text{Cs}^+$, owing to increased layer charge density.

III. Cation exchange and chemical weathering of micaceous vermiculite in relation to fission particle tracks and hydrous metal oxide crusts observed by electron microscopy.

Perforations such as holes and cracks in micaceous vermiculites caused by fission of $^{238}\text{U}$ in the crystal layers and weathering were found (Jackson, Lee, Brown, Sachs, and Syers, 1973)* by scanning electron microscopy (SEM). Hydrous metal oxide crusts intercalated on occasional (001) cleavages and

*(Authors, date) refers to publications or manuscripts accepted for publication given in the first and second list, respectively, of references appended.
layer partings of micaceous vermiculite were observed by SEM through pores, holes, or fractures along (hk0) planes. The coatings of (Fe,Al) oxides were shown to occur as a spatter-pattern over intermittent cleavages while other cleavages were clean. The oxide distribution may build up over $^{238}\text{U}$ fission particle tracks, the latter being found in both primary micas and in vermiculite. Considerable number of $^{238}\text{U}$ fission particle tracks (200 to 500 cm$^{-2}$) were also observed in micas and vermiculites after HF etching (Lee, Jackson, and Sachs, 1973). Diffusion of interlayer $K^+$ and structural Fe and Si(OH)$_4$ through the tracks produced by thermal neutron activation of $^{235}\text{U}$. An increase of selectivity of $K^+$ over $\text{Ca}^{2+}$ and cation exchange capacity during the extensive salt treatments of micaceous minerals after activation showed an important role of uranium fission particle tracks on chemical weathering of the micaceous mineral in nature (Lee, Jackson, and Sachs, 1973). The results indicate that the presence of fission particle tracks was partially responsible for the higher cation exchange capacity of muscovite macroflakes, determined radiochemically, compared to the theoretical value (Mokma, Syers, and Jackson, 1970). Also, in micaceous New Zealand soils (Mokma, Jackson, Syers, and Stevens, 1973), the micaceous vermiculite content largely determined the sorption and fixation of $^{137}\text{Cs}^+$. The strength of fixation suggests that the fixed $^{137}\text{Cs}^+$ may not
be released short of the decomposition of the micaceous vermiculite by weathering.

Ultramicrotomy and high resolution electron microscopy permitted direct photography of layers and (Fe,Al) hydroxy interlayers of phyllosilicates (Brown and Jackson, 1973). Micrographs made before and after an appreciable irradiation from the electron beam revealed images of the 7A structural plane spacings of chloritized materials, representing the interlayer hydroxyl to layer hydroxyl (002) electron density nodes. Loss of the images at 7A intervals and reenforcement of those at 14A intervals was caused by heating in the electron beam. A finding of small micaceous domains in kaolinites observed by high resolution electron microscopy after ultramicrotome cutting of sections perpendicular to the layers (Lee, Jackson, and Brown, in preparation)* are expected to affect the structure and physicochemical properties of kaolinite weathering product of mica. Establishment by these studies of the presence of micaceous vermiculite in clays of highly weathered kaolinitic soils help explain properties important to fixations of radioactive wastes such as $^{137}\text{Cs}^+$ by clays. Furthermore, the high resolution electron microscopy carried out so far (Lee, Jackson, Brown, and

*(Authors, in preparation) refers to manuscripts in the third list of references appended.
Veith, in preparation) is providing relevant knowledge on mica weathering to vermiculite, on the nature of interstratification, on interlayering of aluminum hydroxide in authigenic micaceous minerals, and on the relationship to cation exchange and sorption of $^{90}$Sr$^{2+}$.

IV. Aerosolic (tropospheric) dusts containing micaceous vermiculite react with radiocesium and radiostrontium. The aerosolic (tropospheric) dusts, found in this project by the use of the oxygen isotope composition of the mineral quartz to have accreted in soils and sediments in a wide range of locations throughout the world (Clayton et al., 1972; Mokma et al., 1972), have been shown to contain micaceous vermiculite which is responsible for the sorption and strong fixation of $^{137}$Cs$^+$ (Syers et al., 1972). In the North Pacific area, the $\delta^{18}O$ values of quartz isolated from 25 soils developed over quartz-free basalt in several Hawaiian Islands (Jackson, Levelt, Syers, Rex, Clayton, Sherman, and Uehara, 1971) and from 22 North Pacific, deep-sea, pelagic sediments (Clayton, Rex, Syers, and Jackson, 1972) were remarkably similar (averaging 17.6 and 17.8, respectively), suggesting a common eolian origin.

The dusts, which vary in their $^{137}$Cs$^+$ content depending on locality and time of deposition, all sorbed significant amounts of added $^{137}$Cs$^+$ and strongly fixed it against replacement by Ca$^{2+}$ in 0.01N CaCl$_2$ solution used to simulate
natural soil conditions.

The specific activity of $^{137}\text{Cs}^+$ on eight aerosolic dusts collected from snow and ice, from mud-rains, and from the atmosphere at several localities in the world ranged from 0 to 264 pc/g (Syers, Mokma, Jackson, Dolcater, and Rex, 1972). Zero values were obtained for dust samples collected from a mud-rain in England (deposited in 1968) and from an ice-core in Greenland (deposited pre-1940). A sample separated from melted ice collected on the Franz Josef glacier in New Zealand contained 264 pc/g of $^{137}\text{Cs}^+$; a relatively high proportion of the $^{137}\text{Cs}^+$ on the latter sample could not be replaced by five consecutive washings with 0.01N CaCl$_2$, indicating a strong fixation by the micaceous vermiculite component of the dust. The fact that all of the dust samples sorbed significant amounts (30 to 50%) of added carrier-free $^{137}\text{Cs}^+$, suggests that the absence or presence of only small amounts of $^{137}\text{Cs}^+$ on dusts is related to the lack of nuclear testing at the time when the dust was transported. A pelagic sediment from the north central Pacific and a soil from the Hawaiian Islands, both of which contain weathered micaceous minerals of eolian origin, also sorbed appreciable amounts of added $^{137}\text{Cs}^+$. This implies that aerosolic dusts following deposition as sediments or soils have the ability to sorb $^{137}\text{Cs}^+$ incident in rainwater.
Eolian influenced soils developed in basalt in Australia (Jackson, Gibbons, Syers, and Mokma, 1972) were much less effective than soils and dusts containing micaceous vermiculites in fixing $^{137}\text{Cs}^+$ and $^{90}\text{Sr}^{2+}$ against $0.01\text{N CaCl}_2$ (Mokma, Jackson, Syers, and Gibbons, 1973). The oxygen isotope abundance (13.6 to 15.4 o/oo) in quartz from soils developed in this chronosequence of quartz-free basalt and from associated soils in sedimentary rocks and the particle size distribution of the quartz (flöttsand, coarser than aerosolic silt) indicate that the quartz originated by eolian transport from stranded beach ridges and dunes occurring windward (Jackson, Gibbons, Syers, and Mokma, 1972).

Because aerosolic dusts, which appear to constitute a major vehicle for the global transport and fixation of $^{137}\text{Cs}^+$ and other radioactive cations occur as widespread additions to soils and sediments, established in this study, an examination has been undertaken of the meteorological conditions controlling global dustfall (Jackson, Gillette, Blifford, Danielsen, Bryson, and Syers, in preparation). Variations in oxygen isotopic composition of quartz in dustfall at different latitudes has led to investigation of principles governing mixing of minerals and accumulation in sediments (Churchman, Clayton, Sridhar, and Jackson, in preparation). Control experiments on isotopic exchange in quartz are underway (Sridhar, Jackson, and Clayton, in preparation).
PUBLICATIONS

and Manuscripts Accepted for Publication

Under AEC contract AT(11-1)-1515, initiated June 1, 1965

1967

1. de Villiers, J. M., and M. L. Jackson
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(Paper COO-1515-5)

2. Roth, C. B., M. L. Jackson, J. M. de Villiers, and V. V. Volk
1967. SURFACE COLLOIDS ON MICACEOUS VERMICULITE.
(Paper COO-1515-3)

1968

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(Paper COO-1515-15)

4. Jackson, M. L.
1968. WEATHERING OF PRIMARY AND SECONDARY MINERALS IN SOILS.
(Paper COO-1515-14)

5. Roth, C. B., M. L. Jackson, E. G. Lotse, and J. K. Syers
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(Paper COO-1515-13)

1968. QUARTZ ISOLATION FROM ROCKS, SEDIMENTS AND SOILS FOR DETERMINATION OF OXYGEN ISOTOPIC COMPOSITION.
(Paper COO-1515-16)
1969

7. Rex, R. W., J. K. Syers, M. L. Jackson, and R. N. Clayton
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    1970. MICACEOUS VERMICULITE BASAL CLEAVAGES WITH BLISTER-LIKE LIFTING OF LAYERS AS A FUNCTION OF EXCHANGEABLE CATION CHARGE AND HYDRATION.
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15. Sridhar, K., M. L. Jackson, and J. K. Syers  
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1972

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17. Dolcater, D. L., M. L. Jackson, and J. K. Syers  
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   (Paper COO-1515-31) (Accepted for publication)
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AS RELATED TO CHEMICAL WEATHERING AND CATION EXCHANGE
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   R. A. Bryson and J. K. Syers 
1973. GLOBAL DUSTFALL DURING THE QUATERNARY AS RELATED TO 
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*Numbers refer to published papers and Technical Progress Reports, including COO-1515-6 (1967), COO-1515-18 (1968), and COO-1515-20 (1969), with abstracts of papers published under the contract. Numbers in () incorrectly used for other than Technical Progress Reports. na = Not applicable.
### Key to COO-1515 Report Series (Cont.)

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xx = Sent with this Technical Progress Report (COO-1515-47).

na = Not applicable
Effort level:

Principal Investigator, Marion L. Jackson,
0.25 man year equivalent
(25% of time to date, and to end of current
term of contract)

Co-Principal Investigator, J. Keith Syers,
0.2 man-year equivalent
(to present and to end of current term of
contract -- working on manuscripts for
publication -- See reference list).