INVESTIGATION OF THE MICACEOUS MINERALS IN SLATE

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Abstract

A mineral of the illite (hydromica) group forms the bulk of the fine micaceous material in the slates from the Lehigh-Northampton district of northeastern Pennsylvania. Although similar to the type illite from Fithian, Illinois, in all other respects, electron microscope studies reveal that the mineral in the slate occurs in flakes which morphologically are larger and much better defined than the fuzzy aggregates of minute shreds or fibers which characterize electron micrographs of illite obtained from shales. *X*-ray orientation studies show that the flakes are arranged in parallel orientation in the slate and that the amount of alignment is related to the degree of metamorphism. In the specimens studied the development and alignment of illite plates by metamorphic processes is believed to be the cause of slaty cleavage.

Concentrates of the illite mineral were obtained by froth flotation and supercentrifuge techniques. Due to the presence in the slate of sericite and chlorite, a number of methods were used to determine the nature of the mineral in the fine fractions. Although chemical analyses and thermal tests are particularly diagnostic, data provided by *x*-ray units, the petrographic microscope, and the electron microscope are also significant.

Introduction

As part of a project of the Mineral Industries Experiment Station of The Pennsylvania State College investigating the nature and future utilization of slate, thorough studies of the minerals have been made. While much of the mineralogy of slate has been discussed (1, 2, 3, 4, 5), this investigation has revealed interesting facts pertaining to a number of the minerals in slate and particularly to the micaceous minerals.

The samples studied were from the Lehigh-Northampton slate belt of northeastern Pennsylvania. Detailed investigations were made of a typical slate from the Parsons Brothers slate quarry on the Diamond Vein at Pen Argyl, and other slates were compared with this as a standard.

The investigation was made largely with the petrographic microscope and *x*-ray diffraction units, but firing tests, differential thermal analyses, chemical analyses, and electron micrographs and diffraction photographs have provided much of the significant data.

When the study was begun, microscopic work indicated that the cleavage of this slate was due to a mineral of the illite (hydromica) group. Such a possibility had been suggested by Fairbairn (5) as the result of *x*-ray studies of foliated rocks. Figure 1 is a photomicrograph of a slate section cut perpendicular to the cleavage. The dark veinlets are composed of minute shreds and fibers individually half a micron or less in width. These tiny fragments are subparallel with a prominent cleavage parallel to the foliation of the rock. They show optical orientation which is easily recognized by a marked increase in absorption when the veinlets are parallel to the vibration direction of the incident polarized light. Larger sericite and chlorite flakes are easily distinguished but the mineral forming the veinlets differs from sericite in color and pleochroism, and from chlorite in birefringence. The investigation has not only shown that the mineral is a member of the illite group but has furnished data of possible significance in the interpretation of the mineralogical changes which accompany the development of slaty cleavage during metamorphism.

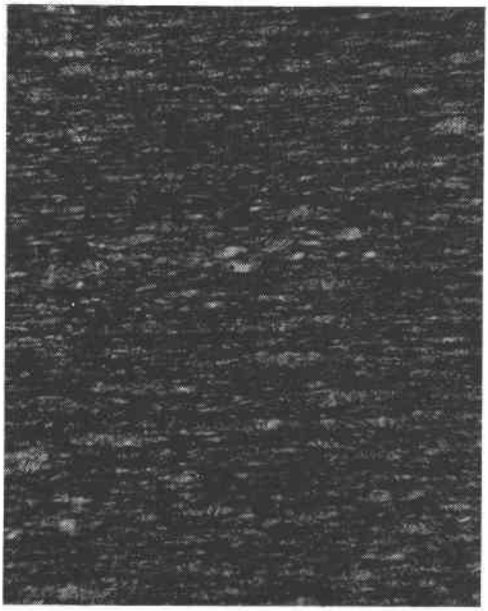

FIG. 1. Photomicrograph of slate, section cut perpendicular to cleavage (×175).

SAMPLE PREPARATION

In addition to the micaceous minerals, the slate contains quartz, calcite, dolomite, feldspar, pyrite, rutile, graphite, and carbonaceous matter. In consequence, one of the main problems in the detailed study of any single mineral was to separate it from the others. This was particularly difficult in the case of the illite mineral because the flakes of sericite and chlorite range from less than five to more than 500 microns in length. It was found that pure fractions of the unknown could not be obtained in sufficient quantity to justify the time expended, but by a combination of methods, relatively pure concentrates of the micaceous minerals could be made.

Slate "flour" was used in all fractionation work. 85% of this material is finer than 0.044 mm. since it will pass a 325 mesh sieve whereas all of it goes through a 200 sieve with an opening of 0.074 mm. The pyrite and carbonaceous matter were first removed by froth flotation in a 500 gram Fagergren cell. To 450 grams of slate 50 mg. of potassium ethyl xanthate was added to act as a collector for pyrite while three drops of pine oil and one of mineral oil served as flotation reagents for the carbonaceous material. The pine oil also acted as a frother. The froth was collected and the remainder of the material dispersed by adding 250 mg. of sodium lignin sulphonate. After dispersion, the preparation was allowed to settle until particles 50 microns or less were the only ones still in suspension.

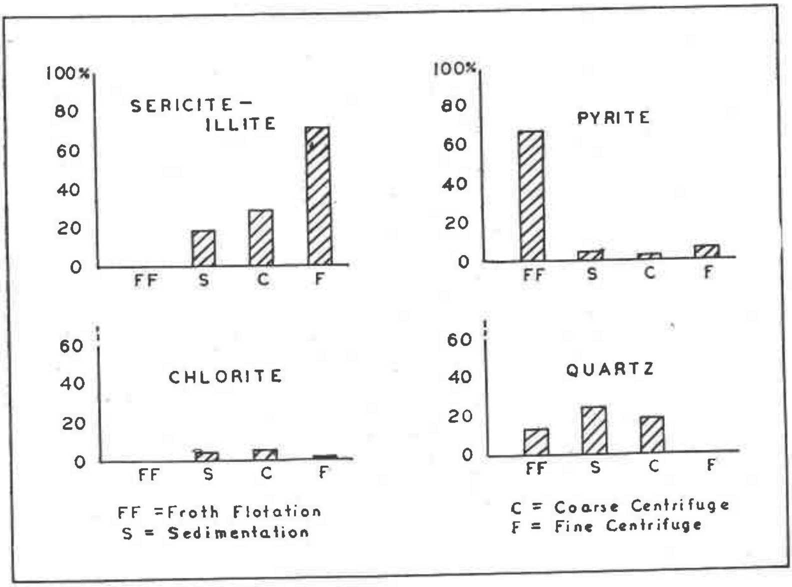

FIG. 2. Concentrates obtained by fractionation of slate.

Further fractionation was made with the Sharples supercentrifuge. Several fractions were obtained by varying the speed and the rate of the feed. For detailed information on the use of this method reference may be made to papers by Fancher and Oliphant (6) and by Oliphant, Houssiere, and Fancher (9). Figure 2 shows the approximate percentage of some of the minerals in several of the fractions. Since the percentages were obtained by x-ray methods no attempt was made to distinguish between sericite and illite in the histogram. The concentrate of fine grained micaceous material provided by the fine centrifuge fraction was found to be of sufficient purity to permit detailed study of the mineral in question.

Using this fraction, tests were made comparing the concentrate with varieties of chlorite and sericite, and with the illite mineral from Fithian, Illinois, described by Grim, Bray, and Bradley (7). Due to the presence of sericite and chlorite and the frequent similarity in behavior of these minerals to that of illite the results were checked by a number of methods.

MICROSCOPIC DATA

Table 1 compares the optical data of sericite, chlorite, and type illite with that of the unknown. Since the chlorite in the slate is apparently the penninite variety the tabulated figures apply to this species. Attempts to obtain the diagnostic interference figure on oriented aggregates were unsuccessful, but pronounced absorption in addition to marked birefringence suggest that the material is illite rather than sericite or chlorite.

TABLE 1. MICROSCOPIC DATA

	Color	Orientation	Absorption	n_γ	$n_\gamma - n_\alpha$
ILLITE	Gray Light Green Light Brown	$X \wedge c = 0^\circ \pm$	X and Y<Z	1.588–1.610	.033–.035
CONCENTRATE from slate	Gray Green	$X \wedge c = 0^\circ \pm$	X and Y<Z	1.587–1.602	.027–.032
SERICITE	Colorless Pale Green	$X \wedge c = 0^\circ \pm$		1.593–1.611	.037–.041
CHLORITE (penninite)	Green	$Z \wedge c = 0^\circ \pm$	X and Y>Z	1.576–1.583	.001–.004

X-RAY STUDY

As Grim et al. (7) have pointed out, there is no significant variation in the interplanar spacings of muscovite, sericite, and illite, although the unit cell appears to be slightly larger in the case of illite. The breadth of the diffraction lines increases from muscovite to illite and this criterion may be employed provided it is kept in mind that the difference may be in part or entirely due to variation in particle size. Figure 3 shows how a

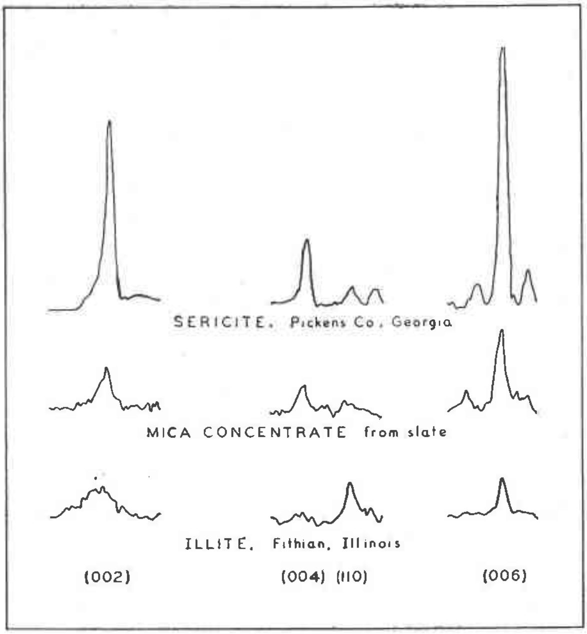

FIG. 3. *X*-ray spectrometer patterns.

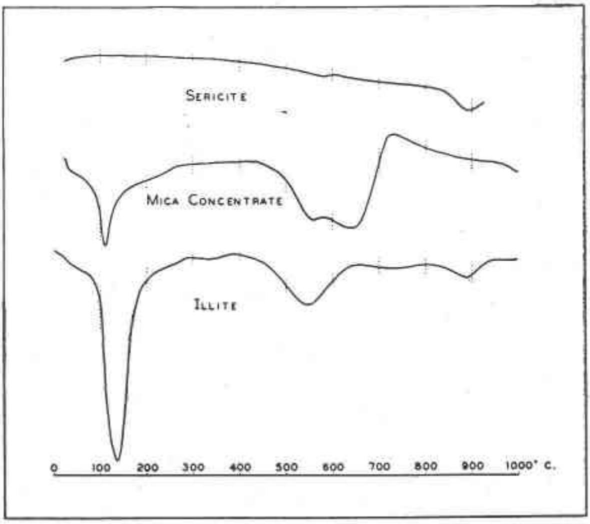

FIG. 4. Differential thermal analysis curves.

pattern made with the Norelco x-ray spectrometer compares with similar patterns of illite and sericite. Thus, although the mica concentrate resembles the illite more than the sericite, the somewhat sharper peaks may represent: (1) coarse-grained illite; (2) very fine-grained sericite; or (3) a mixture of sericite and illite. X-ray diffraction data shows that chlorite is present in the fine concentrate but only in small amount.

Differential Thermal Analyses

Figure 4 shows differential thermal analysis curves of the known and unknown materials. Since x-ray study shows no indication of endellite or any mineral of the montmorillonite group, the low temperature endothermal peak together with the reaction at 560° C. indicates that there is appreciable illite in the fine fraction from the slate. The peak at 640° C. is probably due to the presence of chlorite (10) since even small amounts can give a large endothermal reaction in this temperature range.

Firing Tests

The effect of heat upon the minerals was also studied by firing them to different temperatures and noting changes in structure and the new minerals formed. The procedure consisted of heating a group of samples to a given temperature, allowing them to remain there for two hours, and then removing them from the furnace to permit rapid cooling. A new group of specimens from the original set of samples was used for each firing and the temperatures were 250, 600, 650, 900, 1000, 1100 degrees Centigrade. After cooling each specimen was studied microscopically and

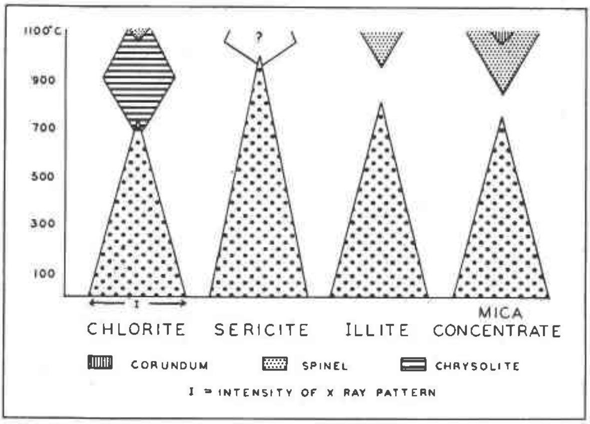

FIG. 5. Firing Data.

x-rayed. Figure 5 summarizes diagrammatically the data obtained and shows the similarity in behavior of the fine slate fraction to the illite mineral from the type locality. In the figure the width of each area represents the intensity of the x-rays diffracted by the phase which exists at the given temperature.

CHEMICAL ANALYSES

Chemical analyses support the evidence that most of the fine fraction from the slate is an illite mineral rather than sericite. Analyses of the mica concentrate and of illite are given in Table 2 and the diagnostic ratios are shown in Table 3. Since TiO_2 and probably CaO are present as

TABLE 2. CHEMICAL ANALYSES

	1	2	1-R	2-R
SiO_2	45.65%	48.95%	47.76%	48.25%
Al_2O_3	25.05	25.03	26.13	24.69
Fe_2O_3	5.41	7.29	5.66	7.20
FeO	—	1.61		1.59
TiO_2	1.95	0.51		
CaO	1.59	0.29		
MgO	3.40	3.10	3.56	3.05
Na_2O	0.51	0.15	0.53	0.15
K_2O	6.50	6.03	6.81	5.95
H_2O at 110° C.	0.60	1.70	0.63	1.67
H_2O above 110° C.	8.53	7.56	8.92	7.45
Total	99.19%	102.22%	100.00%	100.00%

1. Concentrate from slate: Analyzed by R. J. Grace, School of Mineral Industries, The Pennsylvania State College.
2. Illite: Average of five analyses, Grim et al. (7).

1-R. Analysis #1 minus TiO_2 and CaO and recomputed to 100%.
2-R. Analysis #2 minus TiO_2 and CaO and recomputed to 100%.

TABLE 3. DATA FROM CHEMICAL ANALYSES

	SiO_2/K_2O	SiO_2/R_2O_3	R_2O_3/K_2O	SiO_2/Al_2O_3
1. Muscovite	6.0	2.0–2.56 average: 2.4	2.35–3.10 average: 2.80	1.0–3.0
2. Concentrate from slate	11.05	2.74	4.04	3.11
3. Illite	12.75	2.80	4.56	3.32

1. Hallimond, A. F. (8).
2. From analysis 1-R, Table 2.
3. From analysis 2-R, Table 2.

impurities, the original analyses 1 and 2 have been recomputed without these constituents in order to better represent the chemical nature of the illite and the mineral forming the slate concentrate.

ELECTRON MICROSCOPE STUDY

From the foregoing data, particularly that supplied by chemical analyses, firing tests, and differential thermal analyses, it seems evident

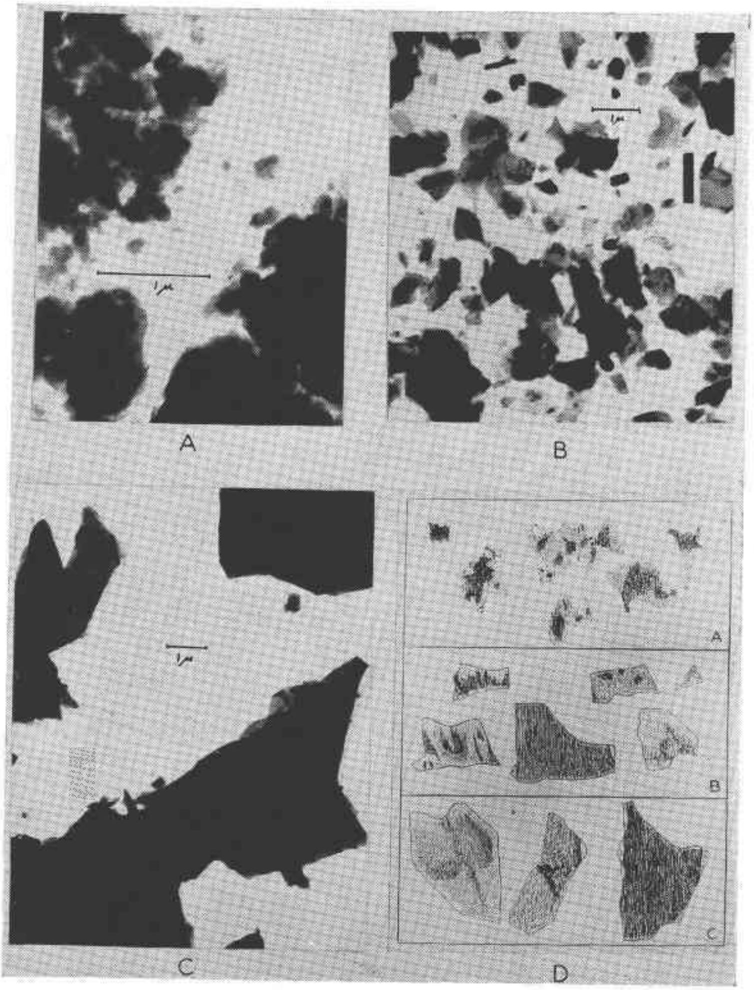

FIG. 6. Electron micrographs. A. Illite mineral from shale, Fithian, Illinois. B. Illite mineral from slate, northeastern Pennsylvania. C. Sericite, Pickens County, Georgia. D. Sketch of characteristic particles from A, B, and C.

that a member of the illite group forms the bulk of the fine slate fraction and is the mineral forming the veinlets shown in Figure 1. Sericite and chlorite apparently account for only a small percentage of the fine micaceous material although larger grains of these minerals are common. In consequence, the electron micrographs in Fig. 6 are of particular interest since the shreds in the picture of the slate fraction do not resemble the fibrous aggregate typical of illite from shale but are similar to sericite both in shape and structure, although smaller in size. Since this mineral differs from type illite only in morphology, it is logical to assume that the development of larger, well-defined flakes is a result of metamorphism.

ORIENTATION DATA

In order to better understand the effect of metamorphism upon the illite, orientation studies have been made upon two slates, one from the "soft-slate" belt from which all slate for commercial use is taken at the present time, the other from the adjacent "hard-slate" belt where the

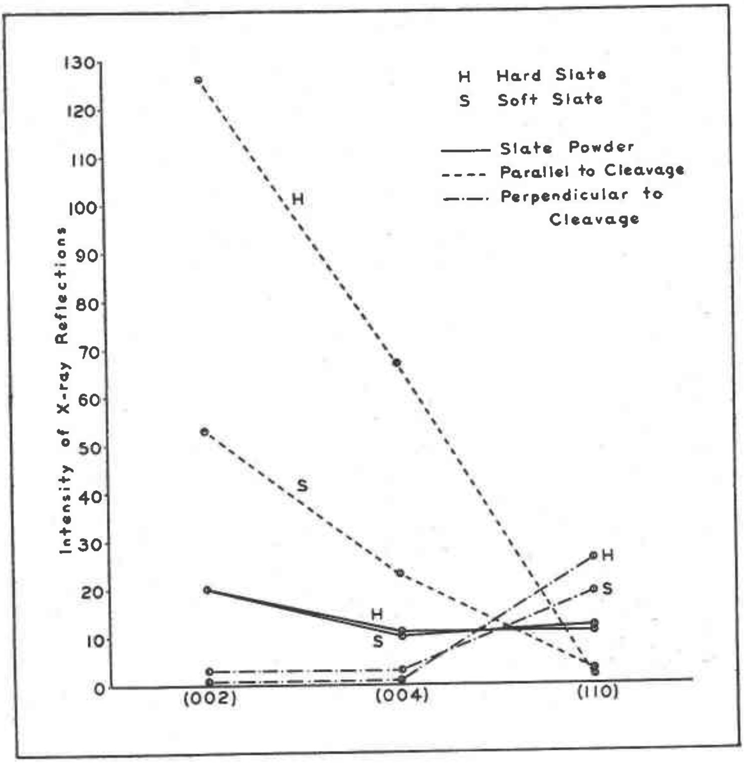

FIG. 7. Orientation study of slate.

rock is much more fissile as a result of a greater degree of metamorphism. The degree of orientation of the illite shreds is shown by Geiger counter measurements of x-ray reflections from the (001) and (110) planes of the illite in slate sections cut both parallel and perpendicular to the cleavage. Data presented in Fig. 7 show that in slate sections cut parallel to the cleavage, reflections from the (001) illite planes are very strong while (110) reflections are weak, while in sections cut perpendicular to the cleavage the opposite is true. Since the particle size in the two slates is nearly the same, the difference in intensity of reflections from the same set of planes is an indication of the degree of orientation in the two slates. Figure 8 shows that in proportion to every five illite shreds in the hard slate that are oriented with (001) parallel to slate cleavage only two shreds are so oriented in the soft slate.

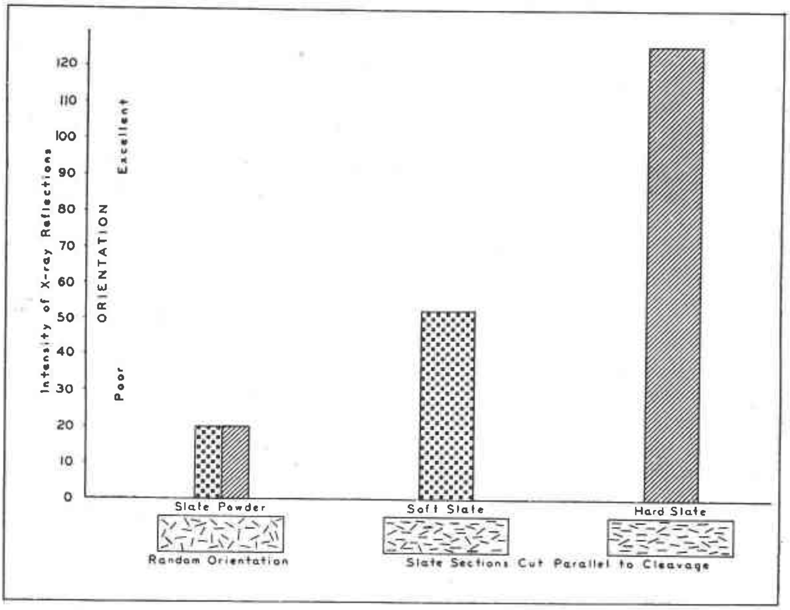

FIG. 8. Degree of orientation of (001) planes of illite in cleavage plane of slate.

INTERPRETATION OF DATA

It is not known whether the illite mineral in the slate was developed from micas and clay minerals during metamorphism or whether illite was present in the original shale and has simply been recrystallized. In either case the effect of metamorphism was twofold: (1) well defined micaceous plates of illite were developed, and (2) presumably during their development, these plates were arranged with basal planes in parallel orientation.

Thus, in the specimens studied, it seems probable that slaty cleavage is caused by the parallel alignment of illite plates. Each dark "veinlet" seen in Fig. 1 is the edge of a broad sheet made up of innumerable parallel flakes. The cleavability of a particular slate sample depends directly upon the number, morphology, and especially the degree of alignment of these minute particles.

SUMMARY

Although optical and *x*-ray studies are not conclusive, chemical analysis and thermal tests indicate that a mineral of the illite (hydromica) group is a major constituent of the slates of northeastern Pennsylvania. Electron micrographs reveal that the illite from the slate resembles sericite in that it is more coarsely crystalline and platy than typical illite obtained from shale, the difference apparently being due to metamorphism. In the "hard slate" which has undergone a greater degree of metamorphism than the adjacent "soft slate," the plates are more perfectly oriented with the (001) planes parallel. In the specimens studied the development and alignment of illite plates by metamorphic processes is believed to be the cause of slaty cleavage.

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