PETROGRAPHIC STUDY OF CLAY MATERIALS

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ABSTRACT

Some investigators have used petrographic methods as the sole means of making clay mineral analyses, whereas others have considered petrographic methods to be useless for materials as fine grained as clays. The value of the method lies between these extremes. It has considerable value, but it has limitations and must be used with caution.

By the use of various auxiliary techniques it is usually possible to obtain fairly precise values for the indices of refraction and birefringence of the clay mineral components of a clay material. Frequently an interference figure can be obtained which yields further data on the optical characteristics. The presence of very fine nonclay minerals, such as quartz and carbonate, and organic material and iron oxide or hydroxide may prevent the making of satisfactory optical measurements.

It is necessary to decide if the optical data obtained are influenced by contamination with nonclay minerals, and if they indicate a single clay mineral or mixture of clay minerals. Fortunately the optical values themselves and the character of the particles on which they were determined give good clues regarding purity and monomineral characteristics.

Optical data in general will not permit the detection of relatively small amounts of a clay mineral in a clay mineral mixture. Also such data may be very misleading in studies of mixed-layer clay minerals.

In addition to optical data, petrographic studies of clay materials yield other important information. They are of value in determining the texture of the rock and the relative abundance and size of the nonclay minerals. In general microscopic studies are easy to perform and the probable value from them is so great, that they should be made in all clay material researches.

Introduction. A reader of the literature on clay mineralogy and clay technology would be told by some authors that optical microscopic methods cannot be applied to clays because of their very fine-grained character. On the other hand he would find some investigators using optical microscopic techniques as the sole means of identifying clay minerals and of providing analytical data for extensive clay-mineral studies. In the writer's opinion the best practice lies between these extreme views. Such methods can frequently be used to produce accurate and precise analytical data. They have limitations and must be used with caution. Under some conditions and for some types of problems, they may produce very misleading results, particularly to the inexperienced investigator. Perhaps it should be remarked at this point that no single technique for clay-mineral analysis is always infallible.

The object of the present paper is to consider the application of petrographic techniques (not including electron microscopy) to clay studies. Consideration will be given particularly to the data which can be obtained, their interpretation, and the limitations of the techniques.

Measurement of Optical Values. It is frequently possible to determine with fair accuracy the indices of refraction, birefringence, optical sign, and axial angle of the clay mineral component of clay materials. Sometimes this can be done by simply mounting slivers of the dried clay as received in index of refraction liquids. This can be done only if the clay material contains substantially no nonclay minerals, and if the clay-mineral particles show some degree of aggregate orientation. It can be done on some bentonites, some fairly pure kaolins, and some shales composed of illites. For such materials, an experienced investigator can obtain a good idea of the clay-mineral composition in a few minutes by an examination with the petrographic microscope.

Many, perhaps most clay materials, require preliminary treatment before satisfactory optical data for the clay-mineral components can be obtained. Such treatment consists of separating the nonclay-mineral components and preparing oriented aggregates (Grim, R. E., 1934) of the clay minerals.

The separation of the nonclay minerals present in discrete particles, like quartz, is a matter of wet sedimentation. In the author's laboratory a separation is frequently made at one micron; the minus one micron fraction is used for clay mineral identification. Sometimes several separations must be made at other size grades, or perhaps another size grade is more desirable. The optimum point for fractionation to obtain the best concentration of clay minerals for analysis varies with the clay material and the problem at hand. Sometimes a preliminary microscopic examination will indicate the optimum size grade into which to separate the clay mineral fraction. Sometimes actual test separations must be made.

It is frequently necessary to separate the clay fraction by a series of wet washings so that essentially a complete split is made of a given particle size. In clay composed of mixtures of clay minerals some components may be more easily dispersed than others so that the mineral composition of the first washing is not representative of the clay-mineral composition.

In the writer's laboratory the clay is first washed free of soluble salts with distilled water; then a suspension usually results without the use of a dispersing agent. If a dispersing agent is required, ammonia is used to avoid the development of a salt when the material is later dried.

Oriented aggregates are formed by allowing the dispersed suspension to stand. The clay-mineral particles tend to settle on a flat surface—which may be the bottom of a beaker or a horizontally suspended glass slide—one flake on top of another with the flat surfaces substantially parallel. In the case of the elongate clay minerals, an aggregate orientation of laths or fibers frequently develops. The accumulated clay is removed from the suspension, dried, and then slivers are mounted in index liquids for study.

The examination of such aggregates will yield usually reasonably accurate values for the α and γ indices. If the orientation of the flakes is about parallel and if 2V is small, as in the case of many of the clay minerals, the variation of the values obtained for α and γ from the true values is negligible. In cases of a single clay mineral, when there is no contaminating nonclay mineral, aggregates may be obtained in which there is orientation parallel to the a- and b-axes and consequently which yield interference figures giving data on the optical sign and axial angle. The development of such aggregates represents something closely akin to crystal growth.

In clay materials containing pigmentary iron or organic compounds it is frequently difficult to eliminate such material from the oriented aggregates. The presence

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of such pigmentary material may make the aggregates worthless for optical measurements. Sometimes such material can be removed by mild treatment with oxidizing agents, supplemented in the case of iron, with acids. Great caution must be used in such treatment as many of the clay minerals are susceptible to attack by such reagents. For example, some montmorillonites and the palygorskites-sepiolites are very soluble in acid.

What Use Can be Made of Optical Data? As illustrated in table 1, the various clay-mineral groups have fairly distinctive optical properties, so that clay-mineral identifications should be possible from optical measurements. Before identifications can be made from optical values, it is necessary to decide if such values were obtained on material uncontaminated by nonclay minerals and composed of a single clay mineral.

With regard to the presence of nonclay minerals, the contaminants are usually quartz (or cristobalite in bentonites) and/or calcite. These minerals may be present in particles less than one micron in diameter so that they cannot be separated from the clay minerals and their detection may be very difficult or, at times, impossible. Usually such material gives a granular appearance to the aggregates as seen when mounted in the index liquids, and it is impossible to obtain optical values with any precision. No interference figures are obtainable; in the case of quartz suspiciously low birefringence may appear, and in the case of calcite the birefringence may be suspiciously high.

An investigator with some experience with the petrographic microscope in the study of clays can usually tell when the clay-mineral aggregates are contaminated with nonclay minerals, although it is not always possible. Thus the presence of some cristobalite mixed with montmorillonite in bentonites frequently escapes detection, but even then experience frequently permits detection of this contaminant.

The determination of the presence of a single clay mineral versus a clay-mineral mixture in aggregates is a more difficult matter. A single mineral rather than a mixture is suggested by the determination of precise optical values, and the presence of good interference figures indicating some parallel alignment of particles parallel to the a and b axes. In addition there are particular characteristics of the clay minerals themselves which tend to reveal their presence when seen under the microscope. Thus montmorillonite has relatively low indices (except nontronite) and high birefringence. The indices of the mineral vary with the loss of adsorbed water, and may change after they are mounted in the index liquid because of adsorption of the liquid. Also the aggregates of montmorillonite have a unique appearance, perhaps because they are composed of very small particles. The individual particles of the aggregate cannot be seen and the aggregate often has the appearance of a single crystal somewhat strained. Again, the appearance alone of montmorillonite aggregates under the microscope often is enough to indicate its presence. Montmorillonites are most easily confused with the attapulgite-palygorskite minerals as they may have the same appearance under the microscope and about the same indices and birefringence. Fortunately this group of minerals is rather rare and the indices are likely to be

a little higher than for most montmorillonites. One would have to be suspicious of montmorillonite-appearing aggregates which had indices in the range of the attapulgite-palygorskites.

The optical properties of kaolinite and halloysite are fairly distinctive so that aggregates yielding these values usually permit safe identification. It is difficult to separate kaolinite and halloysite under the microscope. The hydrated variety of halloysite may be revealed by the lower indices and by the change in the indices as the mineral changes to the lower-hydration form. A suggestion of the presence of the lower-hydrated form may be obtained from slightly lower indices and birefringence than for kaolinite. Halloysite aggregates may seem to be nearly isotropic. Aggregates of kaolinite and halloysite usually have a distinct granular appearance. Care must be taken to avoid confusion of aggregates with low indices because of contaminating quartz. Many kaolinitic clays contain relatively large (10 microns) kaolinite worms which can be seen in a microscopic study of the crude clay. These worms will, of course, yield good optical data, and their character will show the presence of kaolinite.

Aggregates with indices and birefringence equal to illite, which have a granular appearance, are almost certain to be composed of this mineral. Nontronite has similar optical properties but aggregates of this mineral do not have a granular appearance. Like other montmorillonite minerals, nontronite aggregates appear as a single particle that has been subjected to strain. Many shales are composed of illite with a natural aggregate orientation parallel to the lamination. The clay mineral can be identified petrographically in such shales without the special preparation of aggregates.

Experience is too scant with clay-mineral chlorites and vermiculites to specify how much can be done with their identification by petrographic methods. In many of the shales of Pennsylvanian age that the writer has studied, chlorite occurs in large enough and pure enough particles that it can be identified on the basis of its optical properties. In general its optical properties are sufficiently different from those of the other clay minerals so that any relatively pure material should not be missed. If the mineral is intimately mixed with other clay minerals in small amounts, as seems frequently to be the case, it would probably be missed. Apparently it is frequently mixed with illite where its identification is very difficult. Unfortunately chlorite mixed with other clay minerals in small amounts is very difficult to identify by any available analytical technique.

There is even less experience with clay-mineral vermiculite than with chlorite, so that the problem of its identification petrographically can only be surmised. Its optical properties are about equal to those of an iron-rich montmorillonite, and it is unlikely that the two could be differentiated optically. Perhaps the larger particle size of the vermiculite would be suggestive of this mineral.

Optical methods of identification encounter great difficulties when mixtures of clay minerals are involved. In some mixtures where the components are present in about equal abundance, fairly certain identifications can be made. As an example, many Pennsylvanian under-

Table 1.   Optical properties of the clay minerals.

	α	γ	$\gamma-\alpha$	Sign	2V	Dispersion	Remarks
Kaolinite	1.553-1.563	1.560-1.570	.006-.007	(—)	24°-50° 42° mean value	p>V weak	Optic plane and Z ⊥ to (010) X ∧ ⊥(001)=3° ±
Dickite	1.560-1.562	1.566-1.571	.006-.009	(+)	52°-80°	p<V weak	Z=b; X∧C=+15° to 20°
Nacrite	1.557-1.560	1.563-1.566	.006	(—) (+)	40° 90°	p>V p<V	Z—b; X ∧ ⊥ (001) 10°-12°
Allophane	n=1.468-1.512						
Halloysite 4H₂O	Mean value	1.526-1.532					
Halloysite 2H₂O	Mean value	1.548-1.556	.002-.001				
Montmorillonite	1.480-1.590	1.515-1.630	.025-.040	(—)	0-30° ±		X about ⊥ (001)
Hectorite	1.485	1.516	.031	(—)	small		
Saponite	1.480-1.490	1.510-1.525	.030-.035	(—)	moderate		
Nontronite	1.565-1.60	1.600-1.640	.035-.040	(—)	moderate		Pleochroic; yellow-brown-green
Volchonskoite	1.551	1.585	.034	(—)	small		
Sauconite	1.550-1.575	1.592-1.615	.035-.042	(—)	small		
Illite	1.545-1.63	1.57-1.67	.022-.055	(—)	small		X about ⊥ (001); some pleochroic
Glauconite	1.545-1.63	1.57-1.66	.022-.030	(—)	0-20° ±	p>V	Pleochroic; yellow-green
Hydrobiotite	1.59-1.62	1.64-1.67	.045-1.055	(—)	small		Pleochroic; green-brown-yellow
Chlorite	1.57-1.64	1.575-1.645	.003-.007	(∓)	small		Pleochroic; green-brown
Vermiculite	1.525-1.526	1.545-1.585	.020-.030	(—)	small		X about ⊥ (001); pleochroic; green-brown
Sepiolite	1.490-1.520	1.505-1.530	.009-.015	(—)	0-60°		Z about=c (elongation)
Attapulgite	1.510-1.520	1.540-1.555	.025-.032	(—)	small		
Palygorskite	Mean value	1.50-1.555	.025-.035	(—)	small to large		Z//elongation

clays in Illinois are mixtures of illite and kaolinites. Aggregates with substantially no nonclay minerals can be obtained which give easily measurable and fairly precise indices which are intermediate between these clay minerals. In general, small amounts of a second clay mineral may not be determinable optically. This is an extremely serious matter as clay-mineral analyses for many purposes must be complete if they are to be of value. Ten percent of montmorillonite in a clay material may largely determine its physical properties, so that clay-mineral analyses made to predict physical properties or utility must be complete. In many cases, clay-mineral analyses had better not be made unless they are complete.

Identifications based on optical data encounter the greatest difficulty in mixed-layer minerals. Such mixed-layer materials are likely to give almost every indication under the microscope of being a single clay mineral. The indices and birefringence can be measured precisely and often fairly good interference figures can be obtained. Actually the interference figure is likely to afford the best suggestion that the material is a mixture. It can be shown that a stacking of sheet units will give an interference figure with isogyres sharply defined throughout, only if the sheets are uniformly distributed and there is orientation in the a and b axes. If there is variation in the parallelism in the a and b axes and the sheets are not uniformly distributed, the isogyres will first lose their sharpness at the center and then become fuzzy and indistinct throughout as randomness and mixing increase. Thus the character of the isogyres may give a clue to possible mixing of minerals.

The literature is filled with identifications of material as a single mineral by optical methods which has been proven to be mixtures by X-ray and thermal methods.

Bravaisite from the type locality is an example (Grim and Rowland, 1942). Many materials which have been listed as beidellite are other examples.

Other Values of Petrographic Methods in Clay Investigations. Studies of thin sections of clay materials reveal information on the texture of the materials and the paragenesis of the minerals which may be very valuable in some types of investigations. In general the clay minerals are very difficult or impossible to identify in thin sections. Many examples of the value of thin-section studies in clay problems could be given, but a few will suffice. In the study of bentonites, the presence of shard structures is the best evidence of the genesis of the clay by the alteration of volcanic ash. The study of wall-rock alteration associated with ore bodies, the paragenesis of the clay minerals, their possible zonal arrangement, the relation of secondary minerals to primary minerals, and the relation of the clay minerals to the ore minerals all require the study of thin sections. In soil investigations the relation of clay minerals to profile development often requires the study of thin sections.

The value of thin-section studies would be greatly enhanced if a method were available to cut a section without the elimination of adsorbed moisture by drying. In some cases at least, for example, in loosely bonded sands and silts, this adsorbed water is an integral part of the texture and it cannot be removed without changing the characteristics of the material. Thin sections of dried material do not in all cases reveal the texture of the natural material. In the writer's laboratory an attempt was made some years ago to freeze the material suddenly and then cut and examine the section while frozen. The method seemed promising, but other activities forced it to be put aside before it could be worked out.

In the writer's laboratory it is customary to examine all samples microscopically as the first step in any clay-mineral analysis. Such an examination provides information on the kind and abundance of the nonclay minerals. It often gives a good clue as to the probable identity of the clay mineral. Such a preliminary microscopic study aids greatly in planning the further detailed analysis. It indicates, for example, what the optimum particle size is likely to be for the separation of the clay mineral and nonclay mineral fractions. It indicates the size of sample necessary to provide an adequate amount of clay-mineral fraction for analysis. Also, information on the probable kind of clay mineral suggests the sensitivity of the differential thermal apparatus that will be necessary. It may indicate the kind and amount of any pigmentary material and thereby aid in determining what, if any, procedure may be used safely to eliminate it.

When one is faced with problem of analyzing a large number of clay samples, a microscopic study may permit a classification of the samples into types so that complete analytical data need not be obtained on all samples in order that the clay minerals in all samples can become known.

The first reaction of a novice to the petrographic study of clays is to conclude that the method is of no value. With experience he finds that more and more can be done with the method, and he should conclude that it is a very valuable tool. It is particularly valuable as an auxiliary procedure and as a preliminary method of evaluating and planning the whole analysis. As a means of identifying clay minerals, it must be used with caution. When used alone, it will sometimes give trustworthy analyses. Almost always it should be supplemented by other analytical data and this is particularly true when a complete analysis is required. Experience using the clues mentioned herein will serve as a guide to determining when the method can be used safely.

SELECTED REFERENCES

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