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The Preparation of Oriented Clay Mineral Specimens for X-ray Diffraction Analysis by a Filter-Membrane Peel Technique

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Abstract

Oriented clay mineral mounts for X-ray diffractometry may be prepared by filtering a clay suspension onto a membrane filter, and transferring the clay film directly onto a glass slide. The procedure is rapid and produces highly uniform diffraction mounts.

The most widely used methods of preparing oriented clay specimens for X-ray diffractometry are the pipette-on-glass technique and the suction-onto-ceramic technique. These and other techniques are described by Gibbs (1965); recent descriptions of the suction-onto-ceramic technique are given by Rich (1969) and Shaw (1972). Gibbs (1965) showed that the pipette-on-glass technique, and all techniques which involve gravitational settling or centrifugation of clay particles from suspension give rise to size-segregated specimens which are unsuitable for quantitative analysis. The procedure described here is based on rapid filtration of a clay suspension onto a membrane, so that there is no opportunity for size segregation to occur.

Procedure

A standard 47 mm diameter, 0.45 micron pore-size filter (*e.g.*, Millipore HAWP 047 00, Gelman GA-6) is mounted in a standard filter holder (from the same manufacturers). The membrane is supported in the holder by a flat fritted-glass surface which does not impart any texture to the membrane or the clay film on it. The filter assembly is connected to a vacuum, and sufficient clay suspension is filtered to deposit the required amount of clay on the membrane. In the author's experience, a mount made using approximately 80 mg of clay is satisfactory for qualitative clay mineral identification, and for semi-quantitative estimates of clay mineral percentages using a scheme such as that of Biscaye (1965). This amount does not insure that 95 percent of the diffracted intensity from an infinitely thick sample is obtained; for applications in which an "infinitely thick" sample is required, considerably more clay (up to 250 mg, depending on the mass absorption coefficient of the sample and the angular range to be examined) must be deposited onto the membrane. The concentration of clay in suspension should be greater than one percent in order to minimize filtration time. The author prefers to use unflocculated suspensions, as flocculation sometimes reduces the degree of preferred orientation in the resulting mount. However, if "infinitely thick" mounts are required, it is generally necessary to flocculate the clay by adding $MgCl_2$ solution to the suspension in order to keep the filtration time below 5 minutes. If the clay is to be potassium or magnesium saturated, the appropriate chloride solution is then drawn through the clay on the filter, followed by distilled water to remove excess salts. The filter holder is then disassembled and the membrane, while still moist, is placed clay side down on a clean 1″ x 2″ (25 mm × 50 mm) glass microscope slide. The back of the membrane is rolled gently with a 1″ diameter tube, and the membrane is then peeled off, leaving the clay film on the glass slide. Provided there are no air bubbles between the clay and the glass (easily checked by inspection and removed by additional rolling), the clay will adhere to the glass and the membrane will separate cleanly. The mount may then be dried in air or placed over glycerol at 100°C or ethylene glycol at 60°C prior to analysis. The filter membrane may be rinsed and re-used if desired. The author has also used a 25 mm membrane and 1″ diameter circular glass slide to fit the rotating specimen holder for the Norelco X-ray diffractometer. Although the nominal pore diameter of the membrane used is 0.45 micron, the author has never had a problem of clay material coming through the filter; should this happen, membranes with smaller pore diameters are available. The length of the samples produced by this technique is 40 mm, which may not be sufficient to intercept the entire primary X-ray beam at low angles. The angle at which this occurs depends on the geometry of the particular diffractometer and on the divergence slit employed. For example, on a G.E. XRD-5 diffractometer equipped with a 0.4° divergence slit, a 40 mm sample will intersect the entire primary beam down to 3.0°2θ. If precise intensity data are required at lower angles, either a narrower divergence slit or a larger filter membrane must be used. The membranes are available in a wide range of shapes and sizes, and a filter assembly can be constructed to produce a mount of almost any desired geometry. Although the 40 mm mount does not intercept the entire primary beam below 3°2θ, the sample geometry is constant from mount to mount, so that intensity values at low angles are highly reproducible, even though they are not suitable for certain types of calculations.

Results and Advantages

Twelve mounts of the same material (the clay fraction of a Recent marine sediment) were prepared by this method (using approximately 80 mg of clay per sample) and analyzed by X-ray diffractometry. The variance of the X-ray peak area measurements obtained from this experiment was identical with the variance obtained by repeated analysis of the same mount, indicating that the specimens produced by this technique are highly uniform. Compared to the suction-on-ceramic technique, this technique has the following advantages. 1. The membranes and glass slides are stock items and need no cutting or shaping. The materials are also perfectly uniform, whereas different batches of tile may show large variations in permeability and porosity (Shaw, 1972). 2. There is never a problem of X-ray diffraction peaks from the substrate. 3. The maximum filtration time by this technique is approximately 5 minutes, while longer times are commonly required for suction onto ceramic tiles, although the suction time for ceramic tiles may be reduced by the technique of Rich (1969). This is desirable for minimizing segregation errors, in addition to general economic reasons.

The suction-on-ceramic technique does have the advantage that the tiles are unaffected by heating, whereas standard glass slides tend to warp above 500°C (Vycor glass slides will withstand somewhat higher temperatures). Compared to the smear-on-glass technique advocated by Gibbs (1965), the technique described here is usually more rapid since clay suspensions of various concentrations can be used as a starting point and, in the author's experience, a more uniform specimen is obtained for diffraction analysis.

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