Determination of the refractive index of particles in the clay and sub-micrometer size range

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ABSTRACT

This paper describes a quick and simple method for the determination of the refractive index (RI) of very small particles by measuring the light intensity passing through a suspension of particles. Any commercial spectrophotometer and regular capillaries can be used. Since the RI of the liquid in which the particles are suspended can be prepared as requested, there are almost no limitations with respect to the range of the RI. The only limitations arise from the liquids that are available as well as their properties (e.g., viscosity, health risks). A Gauss-function describes the measured data that simplifies the determination of the RI of the mineral. Because the refractive index depends on the wavelength applied, the use of spectrophotometers allows for the determination of the variation of the RI at different wavelengths.

Keywords: Clay, refractive index, dispersion coefficient, layer silicates, nanocomposites, optical properties, small particles

INTRODUCTION

The microscopic determination of the refractive index (RI) of minerals is a well-known procedure (Bloss 1999). It is an elegant method for the unambiguous identification and determination of minerals and their refractive indices. Mineral grains in the size range of 20 to 30 μm are needed for the Becke line method if a polarizing microscope and a set of refractive index liquids are available. It is also possible to use a gem refractometer, but in this case, a mineral specimen with a polished surface of approximately 2 mm² is needed.

Difficulties arise when the particle size of the mineral lies within the range of the used wavelength. These small sub-micrometer particles, especially those in the lower nanometer-range, are far below the optical resolution and cause additional diffraction that complicates the procedure. For the precise determination of the RI of a mineral, one has to match the RI of the liquid in which the particle is suspended. This is sometimes a tedious task. This publication sheds light on a method proposed by Allen and Sennett (1991) by providing more details and presenting the advantage of the technique. This fast and easy method is based on measuring the light intensity (transmission) passing through a suspension of particles of interest. The RI of the suspension liquid may be prepared as needed and therefore is known. Since the refractive index depends upon the wavelength applied, the use of a spectrophotometer allows for the determination of the RI at different wavelengths.

To determine the robustness and sensitivity of the method, various conditions have been tested. Important factors to consider include the temperature dependency of the RI of the suspension, the concentration of the dispersed particles, and the volume fraction of the two immersion oils mixed.

MATERIAL AND METHODS

A Genesys 10UV spectrophotometer from Thermoelectron Corp. was used for this investigation. The transmission was measured at a wavelength, λ, of 589.6 nm (NaD1-line). The background was determined by measuring an empty, air-filled capillary of 1.5 mL volume.

For the RI-mixture, two liquids were utilized: Immersion-oil from Merck KGaA (Merck Art. Nr. 802 505) with n = 1.516, and cinnamon aldehyde from Merck KGaA (Merck Art. Nr. 802 505) with n = 1.6219. Mixtures in the volume ratio ranging from 0 to 100% in 20% and in 10% steps were prepared such that 6 and 11 mixtures with different RI values were available.

Series containing 20 and 50 mg of sample were dispersed into the RI-mixture with a total volume of 2 mL. Hence, a total mass of 180 to 300 mg of substance was needed. Ultra-sonification of the suspensions was limited to 5 minutes to avoid temperature rise of the suspension.

The method has been tested on a quartz reference from BAM (Federal Institute for Material Research and Testing) CRM BAM-PM-101, an illite provided from B+M Arginotec with an average particle size of 250 nm, and two kaolinite samples denoted as kaolinite-PW (Polwhite-B) and kaolinite-GA (Grade-A) from IMERYS, both in the 1 μm size range.

The relationship between transmission intensity and RI was described by a Gauss-function:

\[ I = b + A \exp\left[-(RI - RI_m)^2/(2\sigma^2)\right] \]

with b = baseline, A = amplitude, RI = RI of immersion oil, RI_m = RI of mineral, σ = parameter related to the width of the Gauss curve. This simple function is easy to apply and is often used in the fitting of transmission data.

For the measurement of the RI and the temperature dependence of the RI-mixtures, an Abbe-refractometer was used. X-ray diffraction was performed at the Water Technology and Geotechnology Division of the Institute for Technical Chemistry (ITC-WGT) on a Siemens D5000 instrument with CuKα radiation.