

**A SANS Experiment to Characterize the Structure of
Clay Platelets Dispersed in Water**

or

What Can Neutrons See in Muddy Water?

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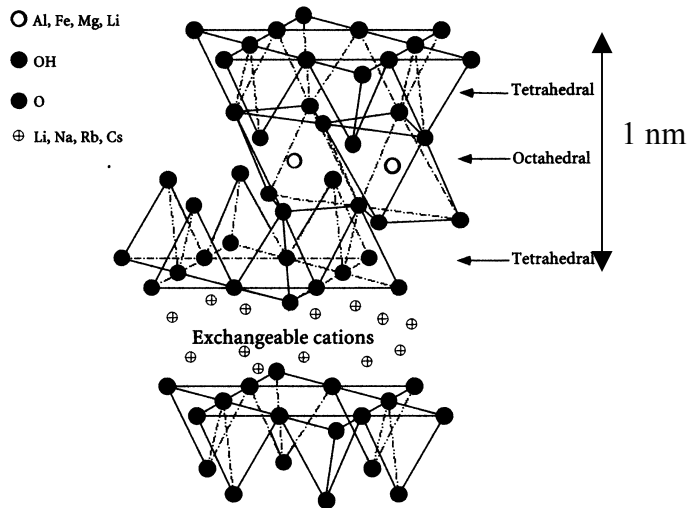
Abstract

Small-angle neutron scattering (SANS) will be used to characterize the solution structure of clay particles dispersed in water. The measurements will be made on an aqueous solution of the common clay, sodium Montmorillonite, to illustrate the application of SANS to a dispersion of highly anisotropic particles. All aspects of the experiment, from the sample preparation and instrument setup through to the data treatment and interpretation will be briefly described and references given for more in-depth study.

I. INTRODUCTION

Clay is an abundant natural mineral that is finding new application as a reinforcing agent in nanocomposite materials. Polymer-clay nanocomposites, for example, have been developed that have improved mechanical strength and fire retardant properties compared with the host polymer matrix. The improved properties are related to the degree of dispersal and exfoliation of the clay particles in the matrix material. SANS is an effective, nondestructive method for characterizing dispersions that can elucidate the relationships between the degree of dispersal and bulk properties. In this experiment, we shall use SANS to characterize an idealized precursor to a clay-reinforced nanocomposite, namely a common clay, Sodium Montmorillonite, dispersed in water¹.

Sodium Montmorillonite (or Bentonite), like all clays, is a crystalline aluminosilicate with a platelet morphology. As shown in the schematic below, each platelet has the 2:1 smectic structure with a middle layer of aluminum hydroxide between two layers of silicon dioxide. The platelets have a net negative charge and are weakly bound through electrostatic interactions with an interlayer of hydrated cations (Na^+ in the present case). The individual platelets are only 1 nm thick, but have lateral dimensions on the order of 1000 nm. Dry clay particles consist of stacks of platelets, called tactoids. The particles are hydrophilic and readily disassociate in water.



Schematic 1. The structure of 2:1 layered silicates. M is a monovalent charge compensating cation in the interlayer and x is the degree of isomorphous substitution, which for the silicates of interest is between 0.5 and 1.3. The degree of isomorphous substitution is also expressed as a cation exchange capacity (CEC) and is measured in milli-equivalents/g.

In this experiment, the SANS from an approximately 2 mass percent suspension of Bentonite in water will be measured.

¹ One technique for producing polymer/clay nanocomposites is to disperse clay in a monomer solution and then polymerize the monomers.

The Objectives of the Experiment are:

- **To determine the predominant shape and average dimensions of the dispersed particles.** This information will be derived from the shape, i.e. the Q-dependence, of the scattering pattern. Since the particles are anticipated to be highly anisotropic, it will be necessary, in order to characterize both the thickness and lateral extent of the particles, to measure over a wide Q-range that encompasses Q-values less than $2\pi/D$, where D is the average diameter of the particles, and greater than $2\pi/2H$, where 2H is the much smaller thickness of the particles.
- **Determine the volume fraction of the dispersed particles.** This information may be derived from the $Q \rightarrow 0$ limit of the scattering curve having scaled the data to absolute units of cross section per unit volume.
- **Determine, possibly, the presence or absence of interparticle self-organization effects.** This will be done by identifying and analyzing deviations in the Q-dependence of the scattering from that expected for a non-interacting, dilute suspension of particles.

II. PLANNING THE EXPERIMENT

Given the stated objectives of the experiment, how do we go about preparing for the experiment so as to maximize our chances of success? Here we discuss some of the issues that bear on this question.

II.1 Scattering Contrast

In order for there to be small-angle scattering, there must be scattering contrast between, in this case, the clay particles and the surrounding water. The scattering is proportional to the scattering contrast, $\Delta\rho$, *squared* where

$$\Delta\rho = \rho_p - \rho_w \quad \leftarrow \text{Scattering Contrast}$$

and ρ_p and ρ_w are the **scattering length densities** (sld) of the particles and the water, respectively. Recall that sld is defined as

$$\rho = \frac{\sum_{i=1}^n b_i}{V} \quad \leftarrow \text{Scattering Length Density}$$

where V is the volume containing n atoms, and b_i is the (bound coherent) **scattering length** of the i th atom in the volume V. V is usually the molecular or molar volume for a homogenous phase in the system of interest.

The sld's for the two phases in the present case, bentonite and water, can be calculated from the above formula, using a table of the scattering lengths (such as Ref. 1) for the elements, or can be calculated using the interactive *SLD Calculator* available at the NCNR's Web pages (<http://www.ncnr.nist.gov/resources/index.html>). The sld's for bentonite and water (both H₂O and D₂O) are given below in Table 1.

Table 1. The scattering length densities (SLD's) for the Bentonite clay (sodium Montmorillonite), light water and heavy water.

Material	Chemical Formula	Mass Density (g/cc)	SLD (cm ⁻²)
Bentonite clay	Na _{.33} [Al _{1.67} Mg _{.33}]Si ₄ O ₁₀ (OH) ₂	2.56	3.67 x 10 ¹⁰
Light water	H ₂ O	1.0	-.56 x 10 ¹⁰
Heavy water	D ₂ O	1.1	6.38 x 10 ¹⁰

From Table 1 we see that scattering contrast for bentonite in H₂O [proportional to (3.67 – (-0.56))² = 17.9] is 2.4 times greater than in D₂O [(3.67 – 6.38)² = 7.34]. However, this is not the only factor to consider. One should also consider the concomitant incoherent scattering from each phase². The incoherent scattering contributes an isotropic background that can obscure weak coherent scattering from the smaller structural features in a material. Here we are interested in both small-scale structure, on the order of the clay platelet thickness of ~ 1nm, and much larger scale structure. Since the incoherent scattering from H₂O is about 30 times greater than that from D₂O, we elect to do the experiment using D₂O as the solvent. The contrast in D₂O is quite adequate and the lower incoherent scattering background will make it easier to distinguish the Q-dependent coherent signal from the Q-independent incoherent background. In addition, the lower incoherent scattering from D₂O allows us to use a thicker sample, as we shall see next, which compensates for the lower coherent scattering contrast vis-à-vis H₂O.

II.2 Sample Thickness

The next decision we face is how thick should the sample be? Recall that the scattered intensity, I_s(Q), is proportional to the product of the sample thickness, d, and the sample transmission, T_s, where T_s, the ratio of the transmitted beam intensity to the incident beam intensity, is given by

$$T_s = e^{-\Sigma_t d}, \quad \Sigma_t = \Sigma_c + \Sigma_i + \Sigma_a$$

where the total cross section per unit sample volume, Σ_t , is the sum of the coherent, incoherent and absorption cross sections per unit volume. The absorption, or neutron capture, cross section, Σ_a , can be computed accurately from the tabulated absorption cross sections of the elements (and isotopes) if the mass density and stoichiometry of the phase

² Incoherent neutron scattering has no counterpart in x ray or light scattering. It arises from the interaction of the neutron with the nucleus, which is described by a scattering length that depends on the particular nuclear isotope and its nuclear spin state. For more information, see, for example, Ref. 5

is known. Σ_a is wavelength dependent, being linearly proportional to λ for nearly all elements. The incoherent cross section, Σ_i , can be *estimated* from the cross section tables for the elements as well, but not as accurately because it depends somewhat on the atomic motions and is, therefore, temperature dependent. The coherent cross section, Σ_c , can also only be estimated since it depends on the details of both the structure and correlated motion of the atoms in the material.

The computations involved in estimated sample transmission are straightforward but tedious. The task is made easier using the NCNR's Web-based *sld calculator* which computes not only scattering length density, but also estimates of the incoherent and absorption cross sections per unit volume. Table 2 gives some of these results for bentonite, H₂O and D₂O.

Table 2. Macroscopic cross sections (i.e. cross sections per unit volume) computed with the *sld calculator* on the NCNR's Web site for the solute and solvents in the experiment. The values for the absorption cross sections are for a wavelength of 6 Å.

	Σ_c (cm ⁻¹)	Σ_i (cm ⁻¹)	Σ_a (6 Å) (cm ⁻¹)	Σ_t (cm ⁻¹)	1/ Σ_t (cm)
Bentonite Na ₃₃ [Al _{1.67} Mg ₃₃]Si ₄ O ₁₀ (OH) ₂	0.282	0.677	0.0271	0.986	1.01
H ₂ O	0.260	5.37	0.0741	5.70	0.175
D ₂ O	0.518	0.136	0.000135	0.654	1.53

The sample to be measured consists of approximately 2 % bentonite (by weight) in D₂O, or 0.869 % bentonite by volume. Hence the total cross section per unit volume for the suspension is .9913 (0.654 cm⁻¹) + 0.00869 (0.986 cm⁻¹) = 0.657 cm⁻¹, and 1/ Σ_t = 1.52 cm. Hence the optimal sample thickness³, the 1/e thickness, is 1.52 cm. The corresponding thickness for the same volume fraction of bentonite in H₂O is 0.177 cm. Hence the large incoherent cross section of hydrogen not only contributes a significant Q-independent background, it also limits the optimal sample thickness.

II.3 Required Q-Range

For this experiment we know we will need to measure over a wide Q-range since the aspect ratio of the clay platelets is so large (platelet diameter/platelet thickness ~ 1000 nm/1 nm = 1000). To get a better idea of the required Q-range, we can use the *SANS Data Simulator* (<http://www.ncnr.nist.gov/resources/simulator.html>) to calculate the Q-dependence of the scattering for the limiting case that the clay particles are fully exfoliated, non-interacting and randomly oriented. From among the 20 different particle models currently included in The *SANS Data Simulator*, we choose the Cylinder model since the scattering from randomly oriented thin disks is a limiting case of this model. The documentation for this model can be found on the NCNR Web site at

³ The scattered intensity is proportional to $d \exp(-\Sigma_t d)$ which has a maximum at $d = 1/\Sigma_t$. However, if Σ_i and Σ_a are small compared with Σ_t , d should be chosen to make $T \sim 0.9$ rather than $1/e = .37$ to avoid multiple scattering.

<http://www.ncnr.nist.gov/resources/sansmodels/cylinder.html>. A plot from the *SANS Data Simulator* for monosized, randomly oriented thin disks is shown in Fig. 1.

Notice in Fig. 1 that the scattering at larger Q is dominated by the solvent scattering from the D_2O . It will be necessary to correctly subtract this scattering in order to reveal the curvature associated with the thickness, $2H$, of the dispersed particles.

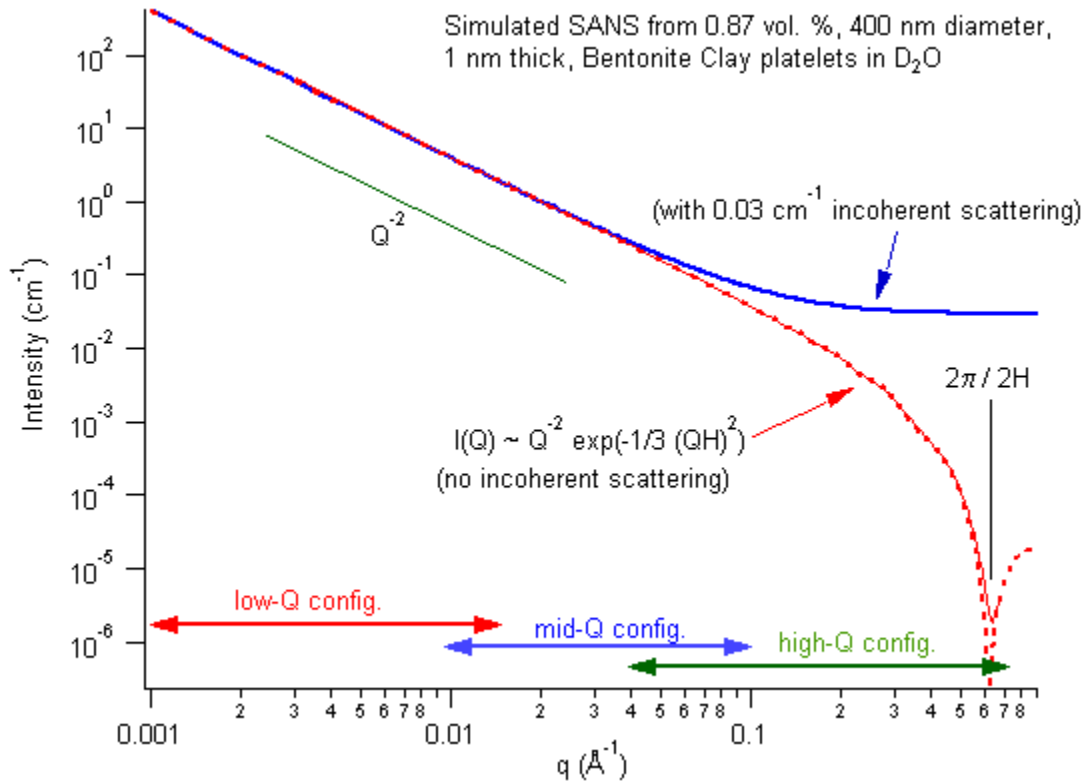


Figure 1. The simulated SANS from monosized, randomly oriented thin disks with diameter $D = 400 \text{ nm}$ and thickness $t = 1 \text{ nm}$. The solid curve includes the incoherent scattering from the D_2O solvent.

III. COLLECTING THE DATA

III.1 How to Configure the SANS Instrument

Now that we know we want to cover as wide a Q -range as possible, we must decide how to configure the SANS instrument to do so efficiently. Here again we can use a computational tool, called SASCALC, as a guide. A schematic of the NCNR's 30-m SANS instruments is shown in Fig. 2, and the instrument configuration parameters, and their allowed range for the NG-7 30-m SANS instrument, are listed in Table 3.

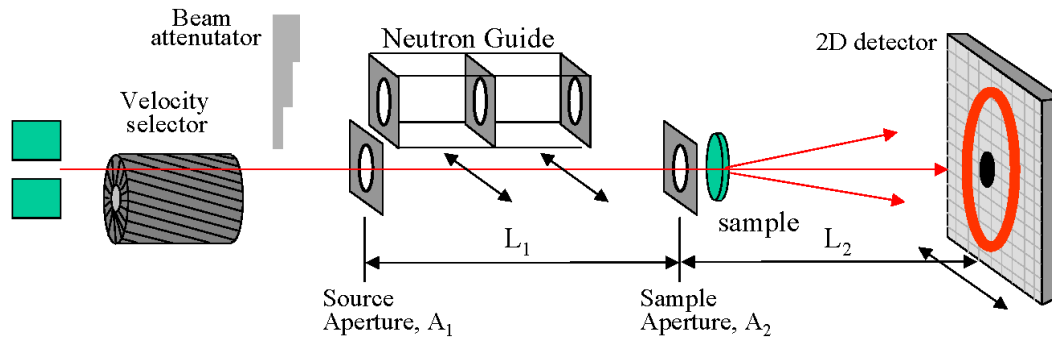


Figure 2. Schematic diagram of the components of the NCNR's 30-m SANS instruments.

Table 3. The instrument configuration parameters, and their range of allowed values, for the NG-7 30-m SANS instrument.

Variable	Allowed Values
Neutron wavelength	5 – 20 Å (determined by the rotational speed of the velocity selector)
Wavelength spread (FWHM)	0.09, 0.11 or 0.22 (determined by the inclination of the velocity selector axis with respect to the beam direction)
Number of neutron guides, Ng	0 – 8 (determines the beam collimation by changing the distance of the source aperture from the sample)
Source aperture diameter	1.43, 2.20 or 3.81 cm for Ng=0; 5.08 cm for Ng=1-8
Sample-to-detector distance (SDD)	100 – 1530 cm
Detector offset	0 – 25 cm (detector translation perpendicular to beam to extend the Q-range covered at a given SDD)
Sample aperture diameter	0 – 2.5 cm
Beamstop diameter	2.54, 5.08, 7.62 or 10.16 cm
Beam Attenuator	10 choices of beam attenuator thickness to reduce beam intensity for sample transmission measurements

For a given set of allowed parameters, SASCALC computes the corresponding Q-range and the beam intensity (n/sec) on the sample. The Q-range for a particular configuration is determined by the choice of wavelength, detector distance and detector offset. To reach the large-Q limit of the instrument, we must use the shortest available wavelength, 5 Å, the shortest sample-to-detector distance, 100 cm, and the maximum detector offset, 25 cm. The number of neutron guides affects primarily the beam intensity on the sample. In general, we choose the largest number of guides, to maximize the beam intensity on the sample, consistent with the desired Q-range. For this large-Q configuration, we can insert all 8 guides. The results from SASCALC for these choices are as follows:

Instrument Configuration for large-Q portion of measurement range

Wavelength:	5.0 A $\Delta\lambda/\lambda$: 0.11 (FWHM)
Number of guides:	8
Sample-to-Detector distance:	100 cm
Detector Offset:	25.00 cm
Intensity at sample:	5.5768E+006 Counts/sec
Qmin:	0.0382 A ⁻¹ Resolution: 20.6494%
Qmax:	0.7174 A ⁻¹ Resolution: 4.5857%
Horizontal Qmax:	0.6438 A ⁻¹
Vertical Qmax:	0.3876 A ⁻¹
Source aperture diameter:	5.08 cm
Sample Aperture diameter:	1.60 cm
Beam diameter at detector:	3.58 cm
Beamstop diameter required:	5.08 cm, (2.0 in)
Attenuator for transmission measurements:	No. 9
Source aperture to sample aperture distance:	387.0 cm

Next we consider how to configure the instrument to reach the low-Q end of the desired measurement range. In this experiment we want to reach very low Q-values both to determine the range and exponent of the expected power-law Q-dependence, and to assure that the data overlap the Q-range of the USANS instrument. For the NG-7 SANS instrument, anytime the required Q_{\min} is less than about 0.0035 A^{-1} , it is advantageous to use the instrument's array of biconcave lenses to focus the beam onto the detector at its maximum distance from the sample. The Q-range and other parameters for this configuration is as follows:

Instrument Configuration for low-Q portion of measurement range

Wavelength:	8.09 A $\Delta\lambda/\lambda$: 0.11 (FWHM)
Number of guides:	0
Sample-to-Detector distance:	1530 cm
Detector Offset:	0 cm
Intensity at sample:	1.7 E+4 Counts/sec
Qmin:	0.0009 A ⁻¹ Resolution: 20.6%
Qmax:	0.0231 A ⁻¹ Resolution: 4.5%
Horizontal Qmax:	0.0163 A ⁻¹
Vertical Qmax:	0.0163 A ⁻¹
Source aperture diameter:	1.43 cm
Sample Aperture diameter:	1.60 cm
Beam diameter at detector:	2.34 cm
Beamstop diameter required:	2.54 cm, (1.0 in)
Attenuator to use for transmission measurements:	No. 3
Source aperture to sample aperture distance:	1627.0 cm

For this experiment we will need a third configuration to cover the gap between the low-Q and high-Q configurations (between $Q \sim 0.016 \text{ A}^{-1}$ and $\sim 0.04 \text{ A}^{-1}$). Here there are

many combinations of wavelength and detector distance that will work. In picking one, we follow the guideline that it is generally preferable to use as short a wavelength as possible, and as long a detector distance as possible, to cover the required Q-range. Hence we choose $\lambda = 5 \text{ \AA}$. We now simply choose a detector distance that gives good overlap with the Q-ranges for the other two configurations. A detector distance of 400 cm will do this nicely. The output from *SASCALC* for this mid-Q configuration is:

Instrument Configuration for mid-Q portion of measurement range

Wavelength:	5.0 A $\Delta\lambda/\lambda$: 0.11 (FWHM)
Number of guides:	4
Sample-to-Detector distance:	400 cm
Detector Offset:	0 cm
Intensity at sample:	1.5 E+6 Counts/sec
Qmin:	0.0096 A ⁻¹ Resolution: 26.4%
Qmax:	0.14 A ⁻¹ Resolution: 4.8%
Horizontal Qmax:	0.10 A ⁻¹
Vertical Qmax:	0.10 A ⁻¹
Source aperture diameter:	5.0 cm
Sample aperture diameter:	1.60 cm
Beam diameter at detector:	4.50 cm
Beamstop diameter required:	5.08 cm, (2.0 in)
Attenuator to use for transmission measurements:	No. 8
Source aperture to sample aperture distance:	1007 cm

III.2 What Measurements to Make

In addition to measuring the scattering from the sample for the three instrument configurations described in the previous section, additional measurements are needed to correct for “background.” Counts recorded by the detector with the sample in place can come from 3 sources: 1) neutrons scattered by the sample itself (the scattering we are interested in); 2) neutrons scattering from something other than the sample, *but which pass through the sample*; and, 3) everything else, including neutrons that reach the detector *without passing through the sample* (stray neutrons or so-called room background) and electronic noise in the detector itself. To separate these three contributions, we need three measurements:

- i) Scattering measured with the sample in place (which contains contribution from all 3 sources listed above), denoted \mathbf{I}_{sam} ;
- ii) Scattering measured with the empty sample holder in place (which contains contributions from the 2nd and 3rd sources listed above), denoted \mathbf{I}_{emp} ; and,

iii) Counts measured with a complete absorber at the sample position (which contains only the contribution from the 3rd source listed above), denoted I_{bdg} .

In addition to these three ‘scattering’ measurements, the transmission (the fraction of the incident beam intensity that passes through the sample without being scattered or absorbed) of the sample and the sample cell must also be measured in order to correctly subtract the contributions to the background and to calibrate the scattering on an absolute cross section scale (the procedure is discussed in Section IV. Data Reduction). The transmission is measured by inserting a calibrated attenuator in the incident beam (to reduce the direct beam intensity to an accurately measurable level) and measuring the direct beam intensity with and without the sample (or the sample cell) in position. The ratio of these two short measurements (typically 1-2 minutes each) is the sample (or sample cell) transmission.

How the scattering and transmission measurements are used to reduce the data to a quantity, called the differential scattering cross section, that is intrinsic to the sample is described in Section IV. Data Reduction.

III.3 How Long to Count

A SANS experiment is an example of the type of counting experiment where the uncertainty, or more precisely the standard deviation, σ , in the number of counts recorded in time, $I(t)$, is $\sigma = \sqrt{I(t)}$. If the scattering is roughly evenly distributed over the SANS detector, then a good rule of thumb is that one should accumulate about 500,000 total detector counts per sample measurement. If the accumulated counts are circularly averaged, one obtains about 50 data points when plotting $I(Q)$ versus Q . This amounts to about 1000 counts per data point with a standard deviation of $\sqrt{1000} \sim 30$ or an uncertainty of about 3 %, which is good enough for most purposes.

A related question, is how long should the background and empty cell measurements be counted relative to the sample measurement. The same $\sigma = \sqrt{I(t)}$ relationship leads to the following approximate result for the optimal relative counting times

$$\frac{t_{\text{background}}}{t_{\text{sample}}} = \sqrt{\frac{\text{Count Rate}_{\text{background}}}{\text{Count Rate}_{\text{sample}}}}.$$

Hence if the scattering from the sample is weak, the background should be counted for as long (but no longer!) as the sample scattering. However, if the sample scattering count rate is, say, 4 times greater than the background rate, the background should be counting only half as long as the sample scattering.

IV. DATA REDUCTION

Data reduction consists of correcting the measured scattering from the sample for the sources of background discussed in Section III.2, and multiplying the corrected counts by

a scaling factor (to remove incidental differences between measurements such as the counting time and sample thickness) that puts the data on an absolute scale of scattering cross section per unit volume. The background-corrected neutron counts, $I_{cor}(Q)$, recorded in a detector pixel in a time interval t are related to absolute cross section, $d\Sigma(Q)/d\Omega$, through the expression

$$I_{cor}(Q) = \phi A \Delta\Omega \varepsilon t d T (d\Sigma(Q)/d\Omega), \quad (1)$$

Where:

ϕ = the neutron flux (neutrons/cm²-sec) at the sample

A = the area of the beam incident on the sample

d = the sample thickness

T = the transmission of the sample (and its container, if there is one)

$\Delta\Omega$ = the solid angle subtended by one pixel of the detector

ε = the detector efficiency, and

t = the counting time.

The incidental instrumental factors can be lumped together into one constant

$$K = \phi A \Delta\Omega \varepsilon t \quad (2)$$

and the intrinsic quantity, $d\Sigma(Q)/d\Omega$, the differential scattering cross section per unit volume, is obtained by scaling the recorded counts

$$d\Sigma(Q)/d\Omega = I_{cor}(Q)/(K d T) \quad (3)$$

We now go over the specific steps involved in extracting $d\Sigma(Q)/d\Omega$ from the raw data. Following equation (1), the raw scattered intensity measured from the sample, I_{sam} , and the empty cell, I_{emp} , can be written as

$$I_{sam} = K d T_{sample+cell} \left(\left(\frac{d\Sigma(Q)}{d\Omega} \right)_{sample} + \left(\frac{d\Sigma(Q)}{d\Omega} \right)_{emp} \right) + I_{bgd} \quad (4)$$

$$I_{emp} = K d T_{cell} \left(\left(\frac{d\Sigma(Q)}{d\Omega} \right)_{emp} \right) + I_{bgd}$$

where $T_{sample+cell}$ and T_{cell} are the measured transmission of the sample (in its container) and the empty container, respectively. From the above, the background corrected scattering, denoted I_{cor} , is given by

$$I_{cor} = (I_{sam} - I_{bgd}) - \left(\frac{T_{sample+cell}}{T_{cell}} \right) (I_{emp} - I_{bgd}) \quad (5)$$

The corrected counts, I_{cor} , are proportional to the quantity of interest, namely the differential scattering cross section. From the above equations,

$$I_{cor} = K d T_{sample+cell} \left(\frac{d\Sigma(Q)}{d\Omega} \right)_{sample} \quad (6)$$

The instrumental scale factor, K , will be determined from a measurement of the attenuated direct beam intensity,

$$I_{direct} = T_{atten} \phi A \Delta\Omega \varepsilon t = T_{atten} K \quad (7)$$

where T_{atten} is the transmission of a calibrated attenuator.

V. DATA ANALYSIS

Since the volume fraction of clay particles in our sample is about 0.01, it is reasonable to analyze the scattering in terms of randomly oriented, non-interacting particles. In this so-called dilute limit, the particles scatter independently, and the total scattering is the sum of the scattered from each particle. The measured intensity (corrected for background and put on an absolute scale) can be expressed as

$$\frac{d\Sigma(Q)}{d\Omega} = (\rho_p - \rho_w)^2 V_p^2 N_p P(Q), \quad (7)$$

where r_p and r_w are the sld's of the clay particles and the D_2O , respectively; V_p is the mean particle volume, and N_p is the number of particles per unit volume. $P(Q)$ is the scattering form factor for the particles,

$$P(Q) = \left| \frac{1}{V_p} \int_{V_p} e^{i\vec{Q}\cdot\vec{r}} d\vec{r} \right|^2, \quad (8)$$

the square of the Fourier transform of the particle shape.

The general expression for the form factor for a cylinder of diameter $2R$, and height, $2H$, is given by

$$P(Q) = \int_0^{2\pi} \frac{\sin^2(QH \cos \beta)}{Q^2 H^2 \cos^2 \beta} \frac{4J_1^2(QR \sin \beta)}{Q^2 R^2 \sin^2 \beta} \sin \beta d\beta, \quad (9)$$

where β is the angle between Q and the cylinder axis, and $J_1(x)$ is a first order Bessel function.

For very thin disks, the first term in the integral for $P(Q)$ becomes unity, i.e., for $QH < 1$, and hence,

$$P(Q) = \int_0^{\pi/2} \frac{4J_1^2(QR \sin \beta)}{Q^2 R^2 \sin^2 \beta} \sin \beta \, d\beta = \frac{2}{Q^2 R^2} \left[1 - \frac{J_1(2QR)}{QR} \right]. \quad (10)$$

In the limit $Q \rightarrow 0$, the above expression becomes

$$P(Q) = \exp(-Q^2 R^2 / 6). \quad (11)$$

The above equation is an example of Guinier's Law which states that for $Q R_g \leq 1$, where R_g is the radius of gyration of the particle, $I(Q) = I(0)\exp(-Q^2 R_g^2/3)$. **Hence for very thin disks, $R_g^2 = R^2/2$.**

For larger values of Q , such that $QR \gg 1$ but $QH < 1$, $P(Q)$ has another simplified form,

$$P(Q) = \frac{2}{Q^2 R^2} \exp(-Q^2 H^2 / 3). \quad (12)$$

The above equation is similar to Guinier's law, as can be seen by rearranging terms to give

$$\ln[P(Q) Q^2] = \ln[2 / R^2] - \frac{1}{3} H^2 Q^2. \quad (13)$$

and hence, **a value for H can be obtained from the slope of the linear region in a plot of $\ln[I(Q) Q^2]$ versus Q^2** . The intercept of such a plot at $Q = 0$ is related to the volume fraction of particles, $\phi = N_p V_p / V$, in solution. This is seen by combining equations (7) and (12) and expressing the results as in equation (13) which gives

$$\ln\left[\frac{d\Sigma(Q)}{d\Omega} Q^2\right] = \ln[4\pi (\Delta\rho)^2 \phi H] - \frac{1}{3} H^2 Q^2. \quad (14)$$

Thus, for a dispersion of homogeneous monodisperse thin platelets, R can be obtained from a plot of $\ln[I(Q)]$ versus Q^2 , provided the Q -values are in the range such that $QR \leq 1$, and the platelet thickness, $2H$, from a plot of $\ln[Q^2 I(Q)]$ versus Q^2 for Q -values such that $QR \gg 1$ but $QH < 1$. In the present case, the clay platelet lateral dimensions (~ 1000 nm) are expected to be too large to be measurable on the NCNR's 30-m SANS instruments; the USANS instrument will be required to determine R in the above expressions, but the 30-m SANS data should be sufficient to determine $2H$.

In analyzing the data, we will use both the modified Guinier law for thin disks, Equation (14), as well as full expression for the scattering from thin disks Equation (10) and compare the results. In both cases, it will be necessary to take into account the incoherent scattering from the sample, as pointed out in Section II.3. This will be done by including a constant as a fitting parameter to represent the featureless incoherent scattering.

VI. REFERENCES AND OTHER RESOURCES

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