Chapter 8

Particle sizing with ENFS.

One of the most important applications of Light Scattering technique, from the industrial point of view, is particle sizing. Industrial particle sizers generally include some sensors, which measure the scattered intensity $I(q)$, both at small and high angles. Generally, a mechanical system makes the powder or the colloid flow in a cell, so that a good statistical sample can be obtained. An algorithm, based on Mie theory, tries to find the distribution of particle diameters, which best fits the measured scattered intensity.

In order to asses the reliability of ENFS applied to particle sizing, we analyzed some mixtures of two colloids. We prepared two colloidal solution of polystyrene spheres. In order that the density of the solvent matches the density of the colloid, we used a solution of equal volumes of water and heavy water: the colloid was quite stable, and did not sediment evidently over some hours. The diameters of the two colloids are $5.2\mu\text{m} \pm 0.5\mu\text{m}$ and $10.0\mu\text{m} \pm 0.3\mu\text{m}$ (samples A and B). The refraction index of the polystyrene is 1.59, while the solvent has the refraction index of water, 1.33. Then, we prepared three mixtures of them, respectively with volume fractions of 1:1, 1:2, 2:1 of samples A and B. The scattered intensity was measured both with ENFS and a state-of-the-art SALS instrument. The data are presented in Figs. 8.1, 8.2, 8.3, 8.4, 8.5

We define α and β the volume fractions of samples A and B in each mixture; the scattered intensity of the mixture with a given α and β is $I_{\alpha,\beta}(q)$. The scattered intensities $I_{\alpha,\beta}(q)$, obtained for the three mixtures, are compared with the scattered intensities $I_A(q)$ and $I_B(q)$ of the two samples A and B. We evaluate the values of α' and β' for which $I_{\alpha,\beta}(q) \approx \alpha' I_A(q) + \beta' I_B(q)$, by looking for the minima of the mean square deviation:

$$\left\{ \begin{array}{l} \alpha' = \frac{\sum_q I_{\alpha,\beta}(q) I_A(q) \sum_q I_B^2(q) - \sum_q I_{\alpha,\beta}(q) I_B(q) \sum_q I_A(q) I_B(q)}{\sum_q I_A^2(q) \sum_q I_B^2(q) - \left[\sum_q I_A(q) I_B(q)\right]^2} \\ \beta' = \frac{\sum_q I_{\alpha,\beta}(q) I_B(q) \sum_q I_A^2(q) - \sum_q I_{\alpha,\beta}(q) I_A(q) \sum_q I_A(q) I_B(q)}{\sum_q I_A^2(q) \sum_q I_B^2(q) - \left[\sum_q I_A(q) I_B(q)\right]^2} \end{array} \right. \tag{8.1}$$

The values of α' and β' are the measured colloid concentrations, and must be compared with α and β. Table 8.1 shows the measured values, α' and β',

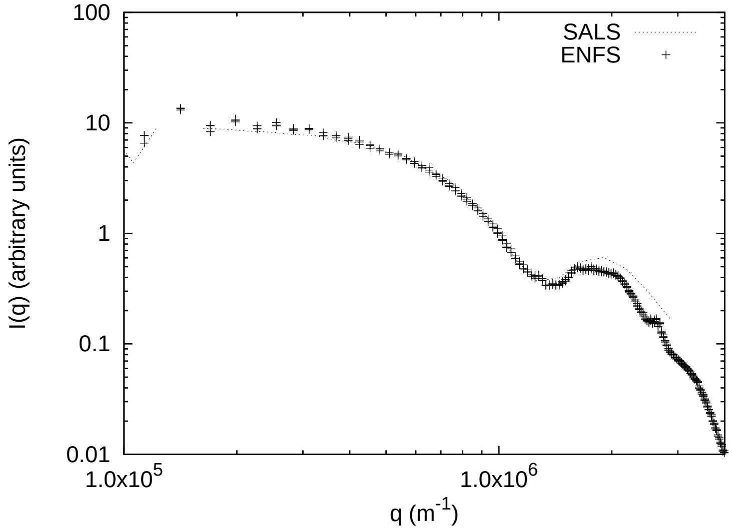

Figure 8.1: Scattered light intensity measurement of a 5.2μm colloid (sample A).

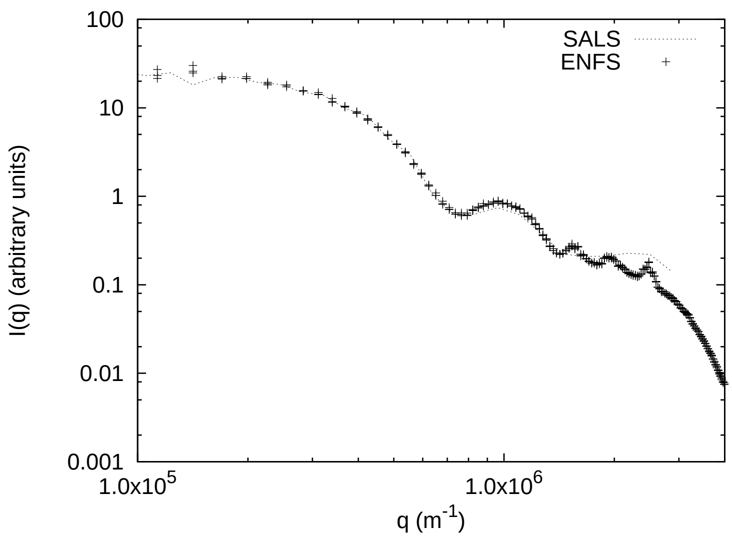

Figure 8.2: Scattered light intensity measurement of a 10.0μm colloid (sample B).

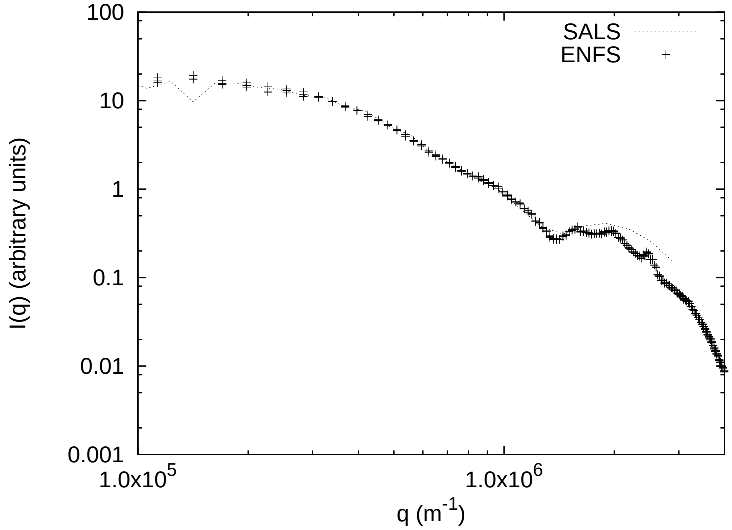

Figure 8.3: Scattered light intensity measurement of a mixture of the two samples. Volume fractions: 1/2 A, 1/2 B

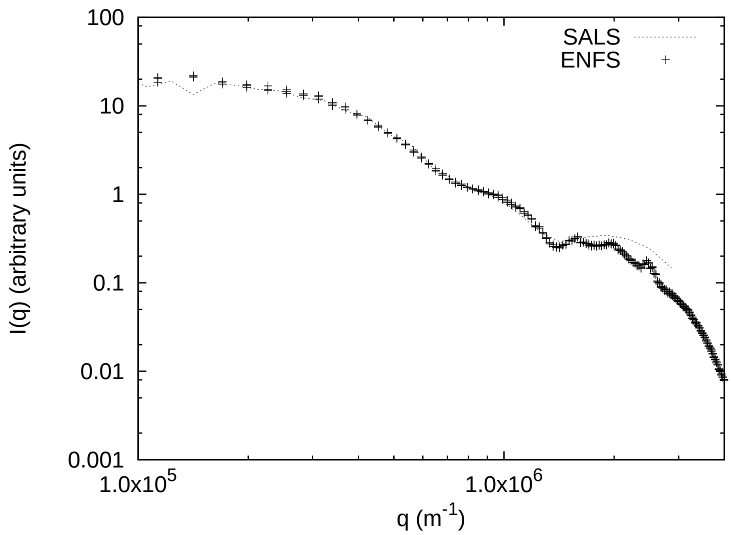

Figure 8.4: Scattered light intensity measurement of a mixture of the two samples. Volume fractions: 1/3 A, 2/3 B

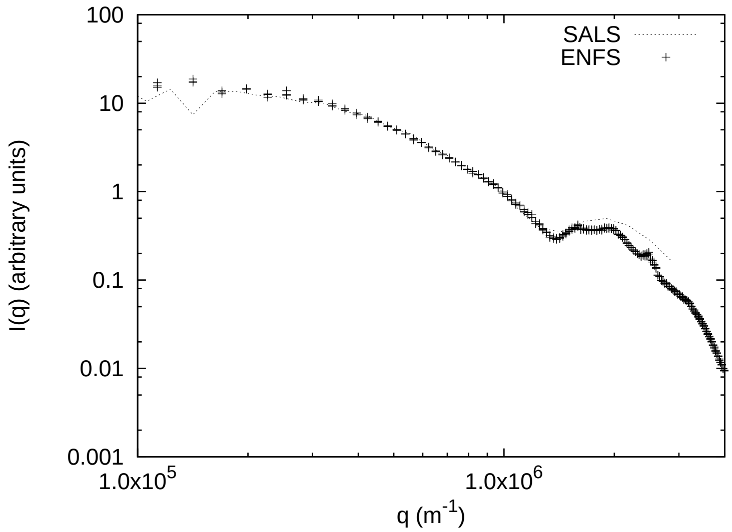

Figure 8.5: Scattered light intensity measurement of a mixture of the two samples. Volume fractions: 2/3 A, 1/3 B

Measured α', β'	Actual α, β
$0.52A, 0.46B$	$1/2A, 1/2B$
$0.69A, 0.32B$	$2/3A, 1/3B$
$0.31A, 0.68B$	$1/3A, 2/3B$

Table 8.1: Measured and actual values of volume concentrations of colloid A and B in the three mixtures.

compared with the actual ones, α and β. The agreement is quite good: this shows that ENFS is suited for particle sizing.

The scattering data has been analyzed by an inversion algorithm based on Mie theory. Mie theory allows to evaluate the scattered intensity $I(q)$ generated by a given diameter distribution $\rho(d)$ of dielectric spheres; the inversion algorithm looks for the distribution $\rho(d)$ which gives the best approximation to the measured $I(q)$. The results are shown in Figs. 8.6 and 8.7. Two peaks are quite evident: they are centered on the diameters of $5\mu m$ and $10\mu m$. The small peak centered around 8μm in the histogram of Sample A corresponds to the scattering of the dymers: the colloid is partially aggregated. The height of the peaks in Fig. 8.7 change accordingly to the fraction of the samples A and B in the mixture.

It should be noticed that ENFS measures the intensity of the scattered beams with reference to the main beam. This allows to evaluate the particle concen-

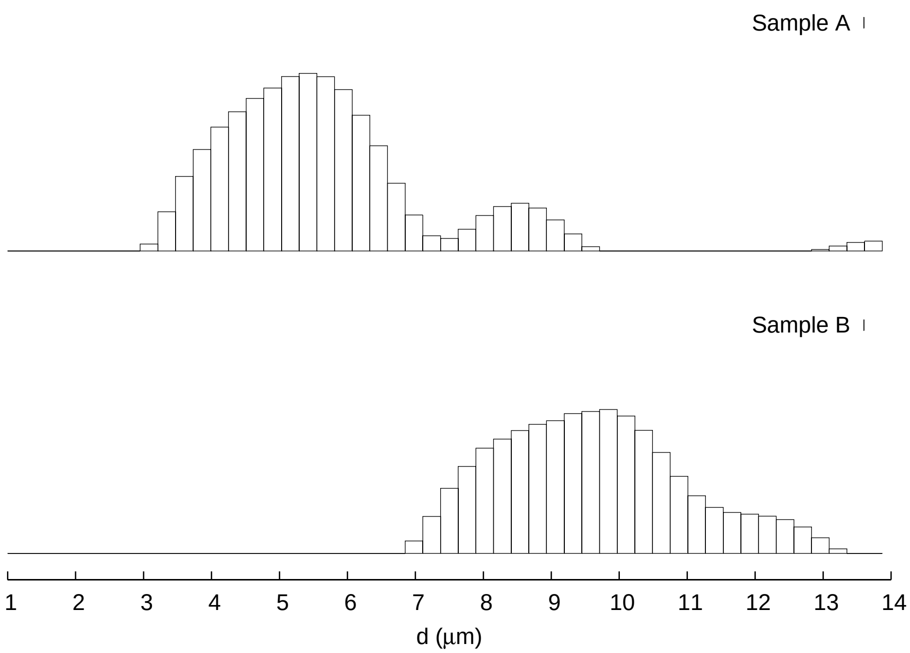

Figure 8.6: Diameter distribution of the two colloidal samples measured by ENFS, obtained by an inversion algorithm based on Mie theory. The height of the bars is proportional to the intensity of light scattered by the particles in the range covered by the horizontal extension of the bar. Sample A is a 5.2μm colloid, and sample B is a 10μm colloid. The two peaks are evident. Sample A shows a small peak centered around 8μm: it corresponds to the scattering of the dymers.

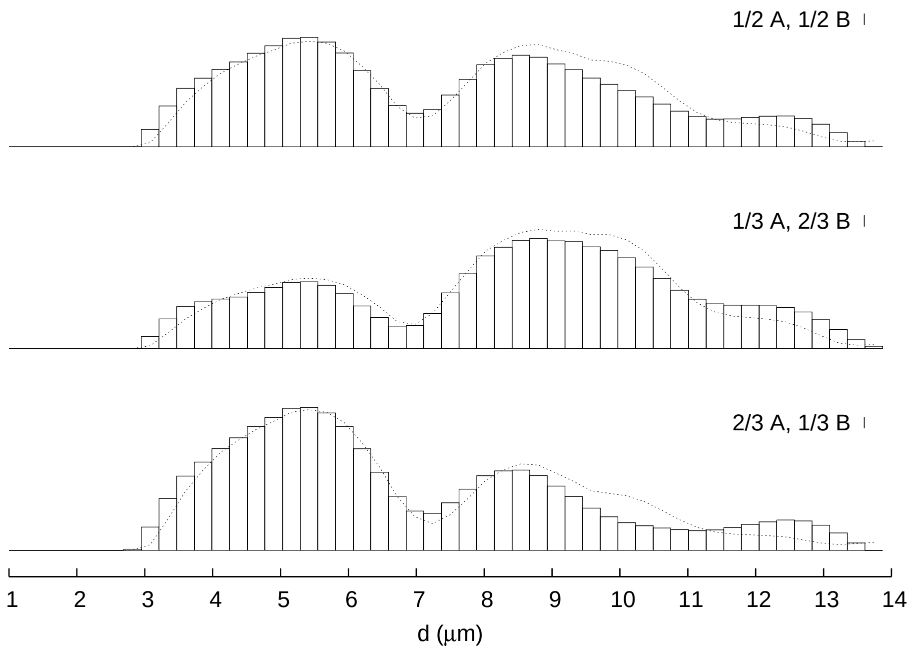

Figure 8.7: Diameter distribution of the mixtures of colloidal samples, measured by ENFS, obtained by an inversion algorithm based on Mie theory. The height of the bars is proportional to the intensity of light scattered by the particles in the range covered by the horizontal extension of the bar. The dotted curves are obtained by combining the values measured for samples A and B, shown in Fig. 8.6 with coefficients given by the volume fractions of the two samples.

tration, and not only the relative concentration of different particles. This is accomplished by using a single sensor; on the contrary, with SALS, the transmitted and the scattered beams must be measured by independent sensors, because the intensities are generally extremely different. This difference comes from the fact that SALS sensors measure the intensity of scattered beams, while ENFS measures the interference of them. For example, consider a sample that generates a single scattered beam, whose intensity is 1/10000 than the transmitted one. For SALS, we need two sensors, one for measuring the scattered beam and one for the transmitted beam, and they require an accurate calibration. A single sensor could be used without calibration, but its dynamic range should cover 4 decades, and in this range it should be quite linear. For ENFS, the interference of the two beams generates a modulation of about 1/100. A single CCD array can easily measure such a modulation.