Characterization of Cephalopod Ink Dispersions Using Tunable Resistive Pulse Sensing Technology. Comparison with SEM and DLS.

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Abstract

We have investigated the utility of Tunable Resistive Pulse Sensing (TRPS) technology for characterization aqueous suspensions of cephalopod ink nanoparticles, which are being studied as surrogates for assessing the environmental dispersion of pathogenic virus. We aim to compare TRPS with two techniques frequently used for determining particle size: scanning electron microscopy (SEM) and dynamic light scattering (DLS), and (ii) to examine the influence of the pore diameter in the TRPS measurements.

SEM images (Figure 1) showed sepia ink as quasi-spherical particles, slightly rough, and a variable size close to 100 nm. DLS data (Cordouan Technology VASCO-2 particle size analyzer) showed the size distribution based on the hydrodynamic diameter (Figure 2). The majority of the particles have a diameter less than 211±18 (CV=8 %) nm (Table 1).

TRPS analysis (IZON's qNano) was done on an ink sample diluted (1/50) in TRIS buffer (tris(hidroximetil)aminometano, 15 mM, pH 8), and filtered by 0.45 µm. The nanopore used in this experiment was an NP200, with a size range from 100 to 400nm.Figure 3 shows two of the size distributions carried out with different parameters (voltage and stretch of the nanopore), and the mean diameter obtained is between 136 and 138 nm. Moreover, we did some experiments without filtering the sample in order to know the concentration of the bigger particles in the ink solution using a nanopore NP400 (for 200-800 nm). The results showed that the concentration of the particles with a smaller diameter (<200nm) is 1.6×10^{11} particles mL^{-1}, while for molecules larger (between 200-800 nm) is 1.3×10^{9} particles mL^{-1}. Using TRPS we can obtain the surface potential too (Figure 4). For ink (in TRIS buffer) it is ranged between 41 and 46 mV.

The majority of the particles are in a range of 100 to 180 nm, but, as can be observed in Table 1, with the DLS the size is slightly larger. This may be because the diameter determined by DLS is a hydrodynamic size, and is bigger than the dry particle size.

We concluded that TRPS gives a size distribution with a higher resolution and sensitivity than SEM and DLS; the replication between samples is acceptable, even using different instrumental adjustments; and data are easily quantified and processed.

References

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Figures

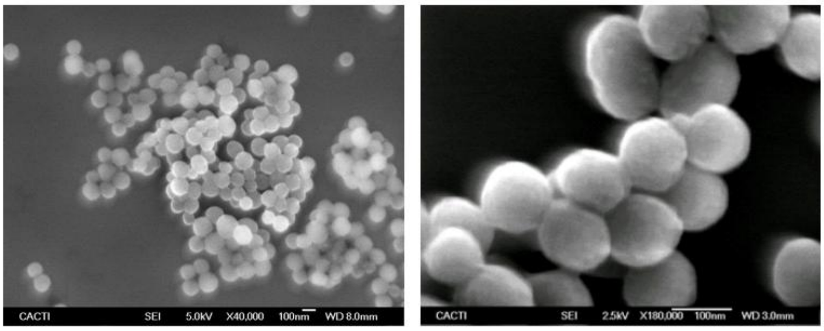

Figure 1. Scanning Electron Microscopy images of sepia ink. Left x40,000; Right x180,000.

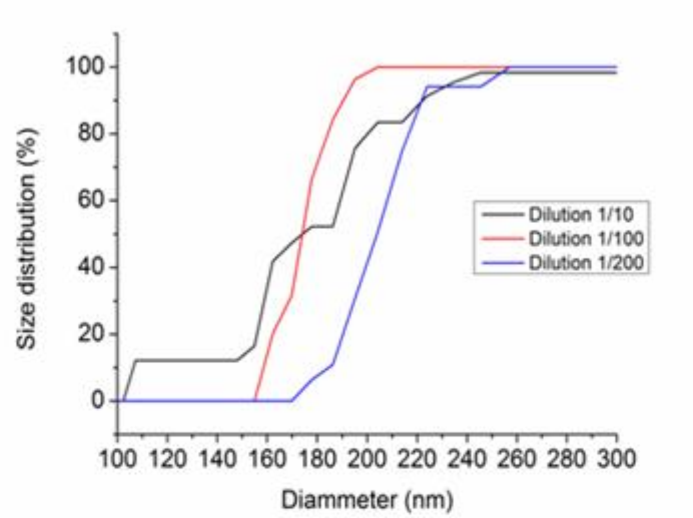

Figure 2. Summary of the cumulative size distribution for the three dilutions: 1/10, 1/100, 1/200.

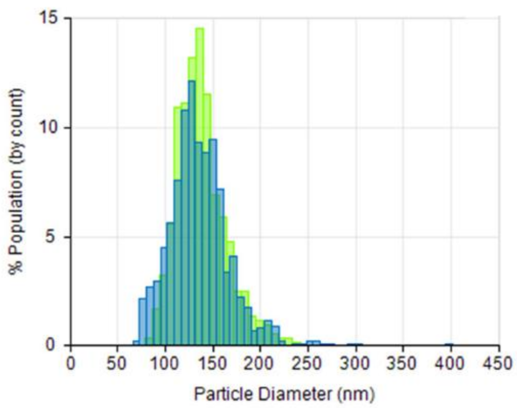

Figure 3: Histogram of the size distribution of two samples with two different instrumental settings. Green: Mean diameter 138 nm; Voltage 0.78 V, Stretch of the nanopore 44.02 mm. Blue: Mean diameter 136 nm; Voltage 1.1 V, Stretch of the nanopore 43.02 mm

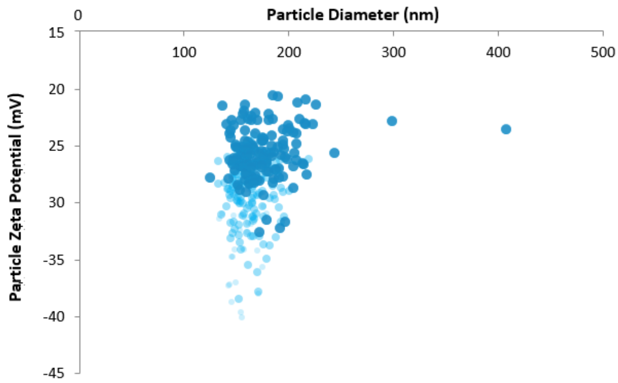

Figure 4: Representation of the surface potential of each particle in an ink sample (filtered 0.45µm, TRIS buffer 15 mM, NP150). The results showed a zeta potential between -41 and -46 mV.

Technique	<149nm diameter	<181 nm diameter	<211nm diameter
DLS	10%	50%	90%
TRPS	79.5-72.4%	91.9-92.2%	96.9-97.7%

Table 1: Comparative percentages obtained with DLS-Padé Laplace and TRPS for particles with smaller diameters than 149, 181 and 211 nm. (Results of TRPS where carried out considering the entire sample, i.e. unfiltered).