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Characterization of natural and manufactured nanoparticles by atomic force microscopy: Effect of analysis mode, environment and sample preparation

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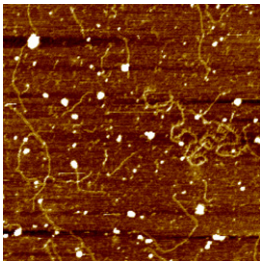
HIGHLIGHTS

- Effect of analysis mode, environment and sample preparation on sizes measured by AFM.
- Sample preparation is the most crucial factor in obtaining accurate size by AFM.
- Sample washing is essential to avoid aggregation artefacts during AFM analysis.
- Sample washing minimizes differences in sizes between liquid and dry condition .
- Non-contact mode is most appropriate for analysis of soft nanoparticles.

GRAPHICAL ABSTRACT



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ABSTRACT

The effect of atomic force microscopy analysis mode (contact and non-contact), the analysis environment (liquid and ambient air) and sample preparation has been systematically investigated on manufactured nanoparticles (NPs; polyvinylpyrrolidone, PVP stabilised gold NPs), extracted Suwannee River humic substances (SRHA) and natural aquatic NPs, with the aim of minimizing sample preparation and analysis artefacts and to obtain the most representative measure of particle size compared to the size of particles in solution. Contact mode results in particle removal due to the strong force applied during analysis and the weak attachment of NPs to the substrate. Lack of sample washing after adsorption to the mica surface or sample preparation by drop deposition and drying can result in the formation of artefacts such as aggregation and salt crystallization. Distinguishing true measurements from sample artefacts are crucially important to our understanding of the size and structure of natural and manufactured nanoparticle analysis by AFM. Consequently, after removing artefacts, our investigations suggest that non-contact mode is most appropriate for the characterisation these NPs. Minimal differences in particle size and morphology were observed between liquid and ambient air condition when thorough washing was performed, and these are comparable between sample analytical variability. This suggests that the more technically challenging liquid mode imaging is not generally necessary for size measurement of manufactured or natural NPs, investigated in this study, when minimally perturbing sample preparation protocols are employed.

1. Introduction

Natural colloids are heterogeneous and polydisperse entities with at least one dimension in the size range of 1–1000 nm [1]. The fraction of these materials in the size range 1–100 nm are currently defined as natural nanoparticles (NPs) [1,2]. Manufactured

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