The characterisation of concentrated latices by photon correlation spectroscopy.

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The background to the assessment of the size distribution of a suspension of particulates using correlation spectroscopy to characterise the apparent diffusion coefficient will be presented. It is realised that multiple scattering may prevent accurate measurements being made at even moderate concentrations, << 1% phase volume. Methods that have been used to avoid these effects will be discussed including the use of short path length cells and the dual wavelength cross correlation technique.

Fibre optic techniques using both single and multimode fibres have been used to study concentrated dispersions, up to 40% phase volume or more. The use of monomode fibres has been shown to largely avoid multiple scattering (5) by virtue of the limited solid angle of light capture, and the fact that the effective scattering angle of 180 degrees automatically compensates for the most probable dual scattered component. Another method using a bundle of multimode fibres works in the limit of severe multiple scattering. (1) The optical configuration for a particular implementation can be characterised to enable the degree of multiple scattering to be compensated for in the analysis of the experimental data.

Having overcome the problem of multiple scattering the fact remains that at high concentrations, or where long range Interactions are present, particles do not diffuse freely and hence a reliable size measurement on an unknown sample usually requires the measurement of a series of dilutions to establish a valid operational concentration. Fortunately many latex dispersions undergo interactions which do not hinder diffusion significantly until concentrations of (say) >10% are reached and may be readily diluted. This is less true of some emulsions.

An instrument is described that utilises single mode optical fibre to measure scattered light from a dispersion of colloidal particles. The instrument utilises a coupler that enables both the illuminating and scattered light to be carried along a common fibre. A 'reference beam' or 'heterodyne' optical configuration is achieved by mixing a portion of the light from the end of the fibre with the scattered light on the detector. This detector is a photon counting photo multiplier, whose output is passed to a multi-bit photon correlator for processing. This instrument has been developed from an earlier analogue device described by Dyott (1), and in a number of papers by Hem et al. (2). It has been used both for particle sizing using the well known photon correlation/Brownian motion method, and for studying particle interaction effects.

The 'reference beam' configuration has the advantage that the contribution of 'dust particles' that may distort the correlation function is greatly reduced and that the first order (G1) correlation function is measured directly rather than needing to be derived via a square root process as is normal with 'homodyne' detection. When the scattering signal is comparable in strength to the reference beam, as will be the case if the sample is of high refractive index and/or very concentrated the signal processing method must take account of the 'homodyne' contribution that will be mixed in: data demonstrating this will be presented. A theoretical treatment of this problem has been performed by Bremer et al (4). Recent work by Van De Meeren et al (4) which uses an alternate analytical method of separating the effects of homodyne mixing will also be presented. An alternative method of avoiding this mixing (Horn (6)) will also be discussed.

The method has potential for use in process monitoring as the fibre probe can potentially be extended to many metres from the electronic base unit, is small, relatively non-intrusive, and robust.