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In situ characterisation of a concentrated colloidal titanium dioxide settling suspension and associated bed development: Application of an acoustic backscatter system

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ABSTRACT

The ability to accurately characterise the settling of particulate suspensions is imperative in numerous industries for improved processing, optimisation and control. Characterisation is typically a non-trivial process, and *in situ* measurements that remove the restriction of requiring particular sample sizes are preferred. Here, for the first time, we explore the use of an acoustic backscatter system (ABS) to characterise key settling dynamics within a common colloidal mineral suspension, where backscatter attenuation is heightened due to the associated low scattering cross sections of the particles. Settling titanium dioxide dispersions with concentrations ranging from 0.05 to 3.00 vol% were successfully profiled using ABS transducers of 1, 2 and 4 MHz frequencies. This approach enabled the simultaneous visualisation of both the settling interface and sediment bed formation, generating sedimentation curves and elucidating settling velocities. Furthermore, backscatter attenuation was empirically correlated with the attenuation–concentration relationship established for homogenous dispersions, to obtain concentration profiles of the settling suspensions. The data depicted concentration changes as a function of time in a hindered settling suspension, and allowed observation of the segregation of a size polydisperse suspension. Data were compared with sedimentation data obtained via two common *ex situ* bench scale techniques. Critically, the acoustic backscatter method was validated as a powerful *in situ* characterisation tool for opaque concentrated heterogeneous dispersions, with the ability to provide concentration density information in conjunction with settling kinetics that are not easily attainable via other methods.

1. Introduction

Particulate suspensions and colloidal dispersions are ubiquitous in numerous industries, ranging from pigments, cosmetics, pharmaceutical and foods, to minerals processing and treatment of water, sewage and nuclear waste. Understanding the settling, segregation and bed development behaviours in these typically complex solid–liquid systems is often imperative to process design and efficiency [1]. Although settling generally depends on initial suspension heterogeneity and particle levels, settling can change from a pseudo-constant concentration process to a series of increasing rarefaction fan lines with time [2–4]. Also, if segregation instability leads to a significant gradient in particle density with height, hindered settling will become depth-dependent, leading to further complexities with time. Importantly, the actual verification of concentration profiles in settling suspensions is non-trivial. The ability to monitor settling rates, sediment bed evolution and concentration changes (which are essential knowledge for optimizing process design [5,6]) is typically impeded due to the lack of convenient non-invasive measurement techniques [7].

The characterisation of physically extracted samples is frequently practiced, albeit labour-intensive, intrusive and incapable of providing spatial and temporal profiles with ease [8]. Alternative approaches include those analysing the transmission-attenuation response of laser light in sample-based devices such as the Lumizier (from Lum GmbH) and Turbiscan (Formulation, SA) [9–11]; however, these *ex situ* systems are only suitable for small sample analysis of slowly evolving suspensions. *In situ* devices include light-based transmission/backscatter or fluorescence systems [12–18], CCD video analysis [19–23], gamma ray [24,25] or x-ray CATSCAN [26] and tomographic techniques which measure electrical resistance within suspensions [27,28]. However, a number of these techniques suffer from common limitations to their use, such as complicated set-ups that encompass specific vessel requirements (and hence are not suitable for deployment industrially), while many are highly intrusive, or complex and expensive in application [29]. Additionally, many are only suitable for specific particle concentration regimes.

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Low-intensity diagnostic ultrasonic techniques however have proven to surmount such caveats to a practicable extent [1,30,31], enabling remote and relatively non-intrusive measurement with subsequently high resolution temporal and spatial profiles of suspended concentration, particle size and settling velocities [32,33]. Typical instrumentation include *ex situ* bench scale devices comprising separate transmitter and receiver probes [34–36], and even recent interest in B-scan ultrasound imaging [37]. *In situ* transceiver devices utilizing Doppler shift measurements provide improved flexibility, such as the ultrasonic velocity profiler, which employs a single frequency [9,38], while point profiling can be conducted via acoustic Doppler velocimetry [39,40] and large dilute fluid depths via acoustic Doppler current profiling [41,42]. Furthermore, related acoustic backscatter systems (ABS) offer the capability of employing multiple frequencies, and enable minimally intrusive true *in situ* depth profiling of suspensions without specific sample size requirements [43,44]. Hence, they can be deployed on a laboratory bench scale or potentially in large-scale industrial systems.

Acoustic backscatter systems are typically utilised in estuarine sediment transport studies [45], for the characterisation of dilute (<1 g/L) sand suspensions with particles typically tens of microns in size or larger [46]. In this scenario, acoustic backscatter theory can be applied to determine concentration or particle size from measured backscatter intensity data since particle specific backscatter and attenuation properties are known [46]. This approach is inapplicable in concentrated suspensions where increased concentration enhances inter-particle scattering [43] and where small particle radii augment the influence of viscous attenuation on the backscatter signal [47]. Indeed, there is a challenge in characterising suspensions of small particles for which a highly attenuated acoustic response is anticipated. Due to these caveats, it is impossible to apply rigorous theoretical solutions to quantify the backscatter response, although some advances are currently being made to enable determination of the backscatter and attenuation properties of arbitrary particles [48]. Recently, a phenomenological approach has been demonstrated for the characterisation of concentration in non-dilute suspensions (>2.5 g/L) and is applied in this study. Importantly Hunter et al. conducted a tangential analysis of the gradient of a backscatter depth curve and demonstrated that the gradient is independent of depth. This enables concentration variation to be measured in suspensions irrespective of the depth scale [43,44].

Titanium dioxide was selected in this study owing to its incorporation in numerous consumer products [49]. Its high refractive index renders an opaque suspension, unsuitable for characterisation via optical techniques. Furthermore, its fine and cohesive particles readily aggregate, resulting in broad size distributions [1,50,51], which is more representative of commonly encountered industrial suspensions. Furthermore, at typically 1–2 μm, this acoustically attenuating size is, at minimum, an order of magnitude smaller relative to particulate dispersions that have been traditionally characterised via acoustic backscatter analysis.

Essentially, this paper investigates the capability of a commercially available ABS to characterise a settling, colloidal, industrially relevant mineral suspension. We examine the ABS as a technique for identifying sludge zone and sediment bed positions, monitoring settling dynamics, bed formation, and elucidating potential concentration gradients within polydisperse suspensions. Despite the anticipated challenges, we extend and validate the phenomenological approach for the characterisation of fine colloidal mineral suspensions for the first time here. Indeed, we investigate a tool which is insightful with respect to furthering our understanding of the dynamics of polydisperse suspensions, for which theoretical modelling is very much in development [36,52,53], and colloidal cohesive suspensions for which modelling is not yet well established due to associated complex inter-particle interactions. Moreover, this is of direct value where *in situ* characterisation aids the optimisation of processes involving suspensions.

2. Experimental procedure

2.1. Materials

All experiments were conducted with an anatase titanium dioxide sample (Degussa, Germany) dispersed in deionized water. Since particle size significantly influences acoustic attenuation and the subsequent measured backscatter response [47], distributions of the dispersed particles prepared via stirring (indicative of the conditions in the proceeding experiments) were obtained via a Malvern Mastersizer 2000 (Malvern Instruments, Worcestershire UK). The average size distribution, obtained from three samples each measured three times, is presented in Fig. 1. For comparative purposes, this is presented alongside the average size distribution of suspensions initially prepared via sonication for the same duration (30 min). Essentially, the broader trimodal size distribution of the stirred suspensions illustrates the enhanced level of particle agglomeration encountered within the proceeding experimental suspensions. The median particle diameters (D_{50}) also reflect this, with 7.2 μm for the former and 2.8 μm for the latter.

For further validation, the size and shape of the particles were also characterised via scanning electron microscopy (SEM). Dry powder samples were coated with platinum and imaged with a LEO/Zeiss 1530 FEGSEM (LEO Elektronike GmbH, Germany). The images collected at ×2,500 and ×20,000 magnification are respectively presented in Fig. 2. At lower magnification, Fig. 2(a) depicts a large number of fine particles plus a few relatively larger agglomerates. At higher magnification, Fig. 2(b) suggests that these fines are typically within the 2 μm region with spheroidal structures comprised of aggregated particulates. Currently, the acoustic response of aggregated structures are not well established [54].

2.2. In situ acoustic experimental method

The AQUAscat 1000 ABS (Aquatec Group Ltd, Hampshire, UK) was utilised for experimentation here. It is composed of transceiving probes which propagate selected monochromatic frequencies (0.5–5 MHz) into the suspension. The active diameters of the transducers range from 1.0 to 1.8 cm depending on the frequency. The experimental setup is depicted in Fig. 3. The 1, 2 and 4 MHz transducers were successively submerged below the waterline of a 4 L titanium dioxide–deionised water suspension in a cylindrical Perspex column; 50 cm in height and 10 cm in diameter. Care was taken to degas the suspension via magnetic stirring prior to experimentation to negate the influence of air bubble attenuation [8]. It was assumed that the speed of sound in the suspension was 1485 ms^{-1}, so close to that in water. The actual value may differ due to the presence of particles in suspension and any air entrainment [8]; however, it wasn't expected to have a significant effect on

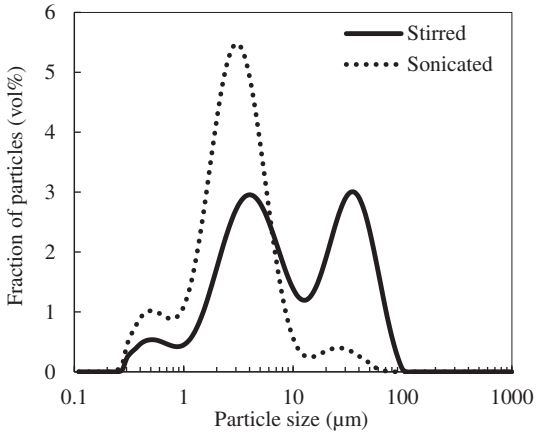

Fig. 1. Particle size distribution.