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### Colloids and Surfaces A: Physicochemical and Engineering Aspects

journal homepage: www.elsevier.com/locate/colsurfa

# Settling of mineral aqueous suspensions. Classification and stability prediction by neural networks



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#### HIGHLIGHTS

#### GRAPHICAL ABSTRACT

- Classification of mineral aqueous suspensions.
- Use neural networks (ANN) to determine prevailing interparticle interactions.
- Propose a method based on ANN to estimate stability regarding sedimentation.

#### ARTICLE INFO

Article history: Received 28 February 2014 Received in revised form 30 June 2014 Accepted 6 July 2014 Available online 12 July 2014

Keywords: Classification Stability Mineral suspension Neural networks Settling

#### ABSTRACT

Aqueous mineral suspensions and pastes are greatly used in industry and waste treatment processes. But unfortunately due to their inherent complexity (numerous parameters to consider and non-linearity of temporal behaviour), their physicochemical stability, controlled by their dispersion state, is difficult to predict. A way to have a better understanding of these systems is to apprehend stability by studying settling behaviour of suspensions in function of solid concentration and interparticle interactions.

To this purpose, previous works on settling optical analysis were used in addition to rheological approach, to determine in some mineral systems, a suspension typology in function of solid mass fraction and predominant particle interaction. In order to generalize this work to various mineral aqueous suspensions, a modelling study is proposed in this paper. The aim of this work is to predict the stability of mineral suspensions based on a specific index: phase separation index (PSI) previously established using 10 discriminating parameters currently measured in industrial and academic areas. Because of their well-known ability to model non-linear processes, a neural networks-based procedure was used to classify the settling behaviour in four classes: diluted, concentrated (cohesive and non cohesive) and solid suspension. The method was proved to be very efficient, delivering 100% of good classification on various sets of test sets up to 60% of the database allowing thus to predict the suspension stability for applications such as inks, paints, cosmetics. In this research, the predominant influence of mass fraction parameter was showed.

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#### 1. Introduction

Aqueous mineral suspensions and pastes are commonly used in several industrial areas or waste treatment process. Their

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http://dx.doi.org/10.1016/j.colsurfa.2014.07.005 0927-7757/© 2014 Elsevier B.V. All rights reserved. macroscopic behaviours such as rheology or physico-chemical stability must be studied to control processes and material properties. However, this control may be difficult because their behaviours depend on various parameters such as solid concentration, particle size, shape or hydrodynamic and interparticle forces.... These latter depend also on a great number of parameters which are linked to the solid or liquid phase characteristics, or particle/liquid interphase. Many rheometry studies showed that, suspensions



behaviour is generally non linear [1,2] with regards to time. These works distinguished three types of suspensions: diluted, concentrated and solid suspensions depending on prevalent interparticle interactions. The multiplicity of systems leads recent studies to use mathematical tools in order to predict, with few experiments and parameters, the settling behaviour of suspensions [3,4]. In the same spirit, a global method of physico-chemical stability analysis, suitable for classification of kaolin and glass beads suspensions as function of interparticle interactions was proposed in previous works [5,6]. They were based on settling measurements carried out using a Turbiscan MA 2000 optical analyser [7]. The authors established a novel index: the phase separation index (PSI), which can be used to a broad range of solid concentrations. The evolution of this index versus the mass solid fraction makes the classification possible as a function of the prevalent type of interparticle interactions. Four classes were derived from the PSI, which can be interpreted in terms of stability behaviour.

To complete and attempt to generalize these studies to all mineral pastes and suspensions, the current work presents an approach based on two steps: the first one study various agglomerated or dispersed suspensions and establishes classification of behaviour based on the PSI index. The second step attempts to generalize the classification and to predict dispersed systems stability as a function of 10 parameters (as particle dimension, density or pH) by a neural networks approach. Neural networks were chosen for their ability to apprehend complex functions and their two fundamental properties: universal approximation and parsimony [8]. Furthermore, among the black-box based modelling tools (SVM, others statistical models), they have the capability to be adjusted to the complexity of the underlying function to identify [9], using a unique formalism, which is not for example the case of the SVM. After the implementation of the neural model, because of its simplicity in operational mode, and low cost, the second phase of the method could of a great interest for industrial sectors as inks, paints, cosmetics

The paper is organized as follows: after the presentation of materials and methods, and PSI calculation in Section 2, a brief overview of neural networks definitions and properties will be proposed in Section 4. The approach of classification will then be presented. Finally the efficiency of the classification will be proved. The conclusion will then be given assuming the real interest of the method as well for industrial than academic users of suspensions.

#### 2. Materials and method

Suspension settling depends strongly on interparticle forces which can be modified at some critical volume fraction ( $\phi_{\nu}$ ). They affect hydrodynamic interactions via the particle drag and mesoscopic organization as size or shape of agglomerates which determine the settling behaviour and sediment structure. In order to propose a general analysis method applicable to a broad range of systems, a phase separation index (PSI) has been proposed [5,6]. This index (Eq. (1) in Section 2.3) is calculated from sedimentation measurements obtained with an optical analyser of concentrated suspensions, the Turbiscan MA2000 from the Formulaction Company.

#### 2.1. Procedure of samples preparation

Samples were prepared from dry powder suspended in deionized water until a total weight of 30 g. Suspensions were stirred during 15 min with a constant speed in order to obtain a homogenous medium. Powders used are kaolin (Prolabo–CAS number 1332-58-7), aluminium hydroxide (Martinswerk) and glass microbeads of various sizes (Potters Europe). Alumina was prepared using aluminium hydroxide treated at 1200 °C in order to obtain  $\alpha$  alumina [10].

#### 2.2. Settling optical analysis

The variation in suspension stability as a function of mass fraction was studied at room temperature  $(21 \,^{\circ}C)$  using the dispersion optical analyser, Turbiscan MA 2000. Although the volume fraction is decisive in theoretical development, the mass fraction has been employed in this work for obvious practical reasons.

The sample was introduced in a glass cylindrical cell (15 mm diameter and 110 mm height) and analysed using a light beam emitted in near infrared (850 nm wavelength) which scanned the entire sedimentation column with a step of 40  $\mu$ m. The time elapsed between two consecutive scans was chosen as a function of destabilization kinetics of suspension. Transmitted and backscattered lights were analysed using detectors placed, respectively, at 0° and 135° from the incident beam direction, so two evolutions of profiles versus times have been obtained: respectively, one in transmission (*T*) and one in backscattering (BS). An example is show in Fig. 1, where we can differentiate three parts to consider in the sedimentation column (which correspond to the sample volume including between the special stopper and the meniscus):

- At the top of the sample, the variations of transmission profiles present a shift on the left of the clarification front, which indicated an increase of the supernatant volume. We can note that a similar variation exists in the backscattering profiles, but they result to secondary reflections of light on the glass cell. Therefore, in this case, one should only consider the transmission signals, and not the BS one.
- The middle of the column is too concentrated to have a transmission signal. So from this part we can study the stability or instability on the BS profiles evolution.
- At the bottom one, we can observe in Fig. 1, an increase of BS profiles values due to sediment formation (increase of  $\phi_v$  value).

#### 2.3. Phase separation index calculation

The phase separation index (PSI) was proposed in a previous sedimentation study [5]. It was calculated from backscattering profiles in the part of sediment formation. It was defined (Eq. (1)) by the product of relative sediment height at 15 min ( $H_s/H_c$ ) and the average value of the percentage of backscattered light ( $B_s^{\times}$ ) in the zone defined as the sediment (i.e. in  $H_s$  in Fig. 1). The choice to measure these values at 15 min is arbitrary, but it seems to be a reasonable time to lead a settling analysis.

$$PSI = \left(\frac{H_s}{H_c}\right) \times \left(B_s^{\%}\right) \tag{1}$$

with  $H_c$ , the sedimentation column height.

So, this index takes into account the quantity and dispersion state of matter which is present in sediment because  $B_s^{\mathcal{X}}$ , the average backscattering percentage of the sediment, is directly linked to the particle volume fraction ( $\phi_v$ ) and the mean particle size (d) as shown in Eqs. (2) and (3). As the sediment structure is directly link to mesostructural agglomeration state of particles during settling processes, the PSI can be a pertinent parameter to characterize suspension stability.

$$B_s^{\varkappa} \approx \frac{1}{\sqrt{\lambda_*}} \tag{2}$$

$$\lambda * (d, \Phi_{\nu}) = \frac{2d}{3\Phi_{\nu}(1-g)Q_s}$$
(3)

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