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A turbidimetric method to measure isoelectric points and particles deposition onto massive substrates

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Abstract

A simple experimental approach was developed to determine the adhesion rate of particles onto massive substrate. Turbidimetry measurements are used to follow the evolution of particle concentration in a suspension in dynamic contact with the walls of a vessel made of different materials. This method allows to rapidly obtain qualitative results about the adhesion of metallic oxides particles on massive substrates. Adhesion of particles of charged latex onto glass was used to validate the approach and was shown to be a method to determine isoelectric points (IEP) of massive substrates. Then, the adhesion of an iron oxide (hematite) particles onto several substrates was studied to determine the reactivity of current labware (glass, polypropylene) and on a metal (aluminum) commonly found in industrial fouling problems. Adhesion of hematite was found to be pH-dependant, and occurs only below ca. 6 (glass) or 7 (polypropylene), and above 7 (aluminum). DLVO calculations were performed to model the hematite/water/glass system and are consistent with the experimental results. Experiments at temperature 7–50 °C have shown an increasing of the adhesion rate from 7 to 40 °C, then a constant value until 50 °C.

Keywords: Hematite adhesion; Latex adhesion; Aluminum fouling

1. Introduction

Deposition of particles in industrial equipments where a fluid is circulating is the cause of several technical problems. For example, such process induces fouling of filters and heat exchangers [1,2], and produces radioactive contamination of cooling circuit of nuclear reactors. Particle deposition proceeds in a two-step process: (1) particles are transported from the bulk of the solution to the walls, according to hydrodynamics, and (2) particles adhere, according to the chemical interactions between the particle and the wall (DLVO theory [3]). Deposition is in competition with detachment of the adhered particles, as a consequence of the drag force [4]. Thus, the deposited amount is the consequence of an equilibrium between these two phenomena.

The simulation of particle transport in turbulent stream has been studied in numerous works (see the reference article by Beal [5]), but it remains a great challenge due to the difficulty to model the coherent structures (bursts, etc.) near the wall and the chemical forces between the colloids and the wall. The chemical interactions were described in the DLVO theory, where the force of adhesion is the sum of the electrostatic forces and the Van der Waals forces. The electrostatic forces are dominant during the deposition phase, and hence the relative charges of the surfaces are the determinant parameters indicating whether the interaction is repulsive or attractive. Isoelectric points (IEP) of the particles and of the substrate indicate the pH range where adhesion could occur, but hydrodynamics force must be taken into account since it can induce a strong drag force which removes the deposited particles, but also can promote the deposition by increasing the collision frequency and the kinetic energy of particles. However, the chemical prediction needs the knowledge of the values of the IEP for both materials, which have to be measured by methods different for particles (zetameters) and for massive substrates (contact angles, streaming potential, etc.).

Several works have been devoted to the direct measurement of adhesion of hematite on steel [6] by the packed column technique

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[7]. A closed circulation loop was designed by Newson et al. [8] to study the deposition of magnetite particles on aluminum tubes. This setup was used by Williamson et al. [9] to measure the deposition kinetics of hematite particles on steel tube. These studies have shown that the pH was a key parameter for the deposition kinetics and equilibrium. The use of charged latex to gain information about surface charge and IEP was described in several articles. Kallay et al. [10] have used the packed column technique to determine the IEP of several pure metallic powders. They have found a good correlation between their IEP values of the oxidized metal surface and IEP value of particles of metal (hydr)oxides. Haque et al. [11] have applied the same method with stainless steel beads. From TEM pictures of goethite-latex suspensions, Blakey and James [12] have obtained the sign of the faces of the acicular particles.

In the present work, we have developed a simple experimental approach to determine the adhesion rate of particles, using turbidity measurements on a suspension in dynamic contact with the walls of a vessel made of different materials. This geometry generates a turbulent flow (contrary to the packed column technique where the flow is laminar), and the suspension is in contact with only the studied material (but the impeller) contrary to a closed circulation loop. The obtained adhesion rates were connected to the IEP of particles and substrates, using ζ potential measurements and literature values. In an opposite approach, we have also studied whether this method could be a simple way to determine the IEP of powdered and massive materials, if the IEP of one of these materials is already known.

Hematite was chosen as a model of oxide particles. Several latexes were also used, since these particles have normally a known surface charge. Glass, polypropylene and aluminum were used as examples of substrate materials in order to explore different domains of IEP values.

2. Experimental

2.1. Materials

In Table 1, particles and substrates are listed. Hematite was a commercial powder from Alfa, thoroughly washed with base, acid and water to remove impurities (sulfate, carbonate, etc.). Spheroidal particles with diameter around 150 nm were observed by SEM (pictures not shown). Latexes (Estapor-Merck) consisted in polystyrene spheres (200–300 nm) with carboxylate groups (“negative latex” K1) or quaternary ammonium/amine groups (“amphoteric” latex K6).

Table 1
Systems investigated in the study

Solids	IEP
Particles[a]	
Hematite	5.4
Latex K6	6
Latex K1	<3
Massive substrates	
Glass	2.4[b]
Poylpropylene	Around 4[c]
Boehmitized aluminum	9[d]

[a] This study.
[b] [27].
[c] Based on IEP of polyethylene [16].
[d] Based on IEP of boehmite particles [15].

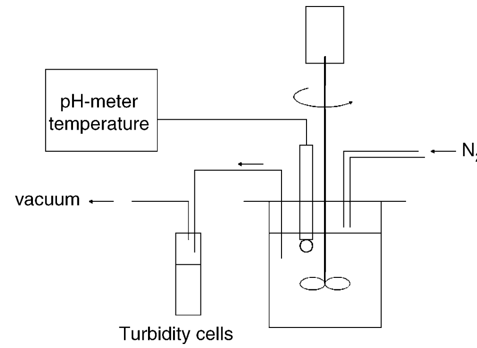

Fig. 1. Experimental setup.

Several substrates were investigated: (1) glass and (2) polypropylene were chosen to determine whether the current labware is inert, (3) aluminum because it is commonly found in industrial fouling problems. Commercial beakers (about 7 cm diameter, 9 cm height) made of glass (Pyrex) and polypropylene was washed with ethanol and water in an ultrasonic bath. Boiling water was poured in aluminum beakers before use to allow the growth of a pseudo-boehmite film [13].

2.2. Setup

Adhesion measurements were carried out with the setup described in Fig. 1. A suspension of particles was poured in a 250-ml beaker and sonicated. Stirring was operated by a four-blade (45°, 14 mm diameter, 1 mm thickness) glass impeller. pH and temperature were continuously measured with a pH electrode and a temperature sensor (Metrohm 6.0228.000). A Tygon tube plunged in the suspension and allowed transferring 30 ml of the suspension in a turbidity cell with help by a vacuum line. Adhesion experiments were carried out with a continuous flow of nitrogen over the suspension to avoid CO_2 contamination. A thermostated double-wall jacket allowed a temperature control at ±0.5 °C.

2.3. Experiments

Each experiment consisted of different steps:

(i) Washing of the reactor and checking that turbidity of sonicated water in the reactor is negligible.
(ii) Filling the reactor with the suspension (usually 200 ml) and sonication during 10 min to destroy the aggregates [14].
(iii) Starting the stirring, pH measurement, N_2 flowing.
(iv) After 5 min, withdrawing 30 ml into a turbidity cell and measurement of the turbidity.
(v) Pouring back the analyzed suspension into the reactor.
(vi) Repeating steps (iv)–(v) typically four times during 30 min.