



Powder wettability at a static air–water interface



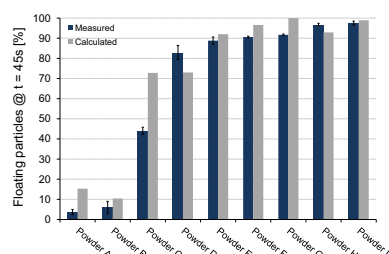
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GRAPHICAL ABSTRACT



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ABSTRACT

The reconstitution of a beverage from a dehydrated powder involves several physical mechanisms that determine the practical difficulty to obtain a homogeneous drink in a convenient way and within an acceptable time for the preparation of a beverage. When pouring powder onto static water, the first hurdle to overcome is the air–water interface. We propose a model to predict the percentage of powder crossing the interface in 45 s, namely the duration relevant for this application. We highlight theoretically the determinant role of the contact angle and of the particle size distribution. We validate experimentally the model for single spheres and use it to predict the wettability performance of commercial food powders for different contact angles and particles sizes. A good agreement is obtained when comparing the predictions and the wettability of the tested powders.

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1. Introduction

Particles floating at the surface of a liquid is a vast topic that is relevant to many industrial applications. In the food industry, continuous reformulation of powders to further improve their nutritional value can sometimes bring along undesired loss of wettability performance which impact the entire reconstitution process [1,2]. Food powders are complex systems made of particles with different sizes and shapes which can either float or sink depending on their wetting properties. In first instance, we will assimilate grains to spheres.

Whatever the sphere size, floating and the existence of an equilibrium position depend primarily on the relative density of the sphere, with respect to the liquid it is poured onto. Furthermore, for a sphere that is small, wetting properties play a paramount role. In the case of a very low contact angle (e.g. clean glass and water) a sphere denser than the liquid will sink whatever its size. Conversely, if the sphere exhibits poor wetting properties (e.g. Teflon), a larger weight will be necessary to ensure sinking.

Several authors (among others Scheludko [3], Ivanov [4], Keller [5] and Vella [6,7]) described theoretically the floating condition of a single cylinder or sphere, determined by the force balance of weight, buoyancy and capillary forces. Liu et al. [8] studied the contribution of those forces when varying the diameter of a hydrophobic glass sphere and made visual observations of the

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floating behavior. Lee et al. [9] described the sinking dynamics of small spheres with regards to sphere density and radius, liquid density, viscosity and surface tension, and partially validated their equations with particle image velocimetry. Shang et al. [10] presented an interesting combination of theoretical and experimental investigations to predict the mobilization of colloidal particles in the vadose zone of soils. The authors successfully compared with experiments the maximum theoretical capillary force generated by the particle–interface contact.

Two spheres can get attracted and sink while individual spheres would not [11–13]. Finally, floating experiments were used to derive the wicking condition in a bed composed of monodisperse or bidisperse spheres [14], considering also the effect of an applied pressure.

In the case of more complex grains, such as food particles, other mechanisms that could modify the flotation behavior should also be considered. First of all, food particles are porous, leading to capillarity penetration and modification of the triple line length [15,16]. Porosity can affect the contact angle depending on the fraction of pores filled with liquid [17,18]. This can result in a modified force balance, influencing the equilibrium immersion depth of the particle. Secondly, many food powders are soluble and this induces a contact angle which varies in time due to hydration at the contact line [19–22]. Depending on the diffusion properties of the liquid into the material, the decrease of the contact angle can be quite rapid which will improve the “sinkability” of the particles. Dissolution can also increase locally the viscosity which can modify the dynamic of sinking as well as the contact angle [23]. This is especially true for high molecular weight carbohydrates. Thirdly, food particles are non-spherical and rough, which conditions the contact angle [18,24] and the force balance [11]. Finally, interactions between particles may occur. In addition to capillary interaction [25], amorphous materials might pick-up moisture, pass the glass transition and thus form viscous bridges [26,27]. Products containing fat can also present stickiness issues due to the formation of fat capillary bridges between grains. Complex flow patterns, for instance the presence of liquid jets can also influence the way particles are sunk into water [28].

In this work, to simplify the problem, we describe the wettability of food powders assuming spherical grains and by making relevant assumptions on the material behavior. We first focus on a single sphere and check experimentally the force balance model. We propose a way to apply the same approach to a mixture of polydisperse spheres and finally apply our equations to powders characterized by various relevant experiments.

2. Materials and methods

We characterized the floating behavior of spherical non-soluble grains measuring the forces that apply on the sphere as a function of its position with respect to the air–water interface. We used a tensiometer (*Kruss K12*) with a force sensitivity of 10^{-4} mN. In this test, the sphere is glued to a metal stem which is connected to the tensiometer (Fig. 1). A liquid bath containing water is moved up the sphere at a slow and constant speed of 1.35 mm/min. The experiment is therefore considered “quasi-static”, i.e. the velocity does not influence the measurements, as confirmed by tests with different heights. The measured force evolves as a function of the sphere’s immersion depth until it is totally immersed. This force represents the sum of capillary and buoyancy forces. Data are collected every 0.15 mm with two types of inert spheres materials: polypropylene (radius 2 or 5 mm characterized by imaging) and glass (radius 1 mm). Glass beads were modified by chemical treatment to vary surface affinity for water (using HCl to increase hydrophilicity and a mixture $\text{Si}(\text{CH}_3)_2\text{Cl}_2$ /Heptane to increase hydrophobicity by silanization after HCl treatment).

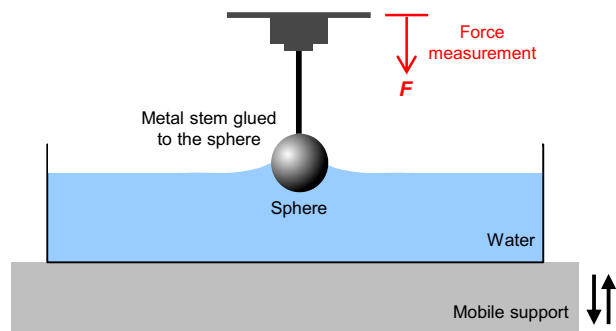


Fig. 1. Force measurement between a hung sphere and a liquid as a function of immersion depth d using a tensiometer.

We did also characterize the floating behavior by imaging spherical grains gently deposited at the air–water interface using tweezers. To obtain lateral pictures of good quality, we built a container using four microscope slides which were previously silanized to obtain a contact angle with water close to 90° and thus prevent meniscus formation.

We also characterized the contact angle between water and our materials. In the case of inert spheres, the contact angle was simply obtained by imaging (Fig. 2a). In the case of the various tested food powders (milk, cocoa, chocolate and coffee powders), we prepared tablets, whereby the compaction should be sufficient to avoid liquid penetration upon contact angle measurement, but not too strong to prevent fat migration. Contact angle between a 3- μL water droplet and the tablet surface was measured as a function of spreading time using a macro-objective and a camera (Fig. 2b). Pictures were analyzed with the “Drop Shape Analysis” software (*Kruss*).

The particle size distribution of the powders was characterized by two different methods. For powders with particle size below 1 mm, we used laser diffraction (Malvern Mastersizer 2000 with dry dispersion module). For larger particles (case of powder B), we used an imaging approach (*Zephyr Occhio*). Those analyses provide a particle size distribution $N(r)$ characterized by the mean radius over volume $r = R = D_{4,3}/2$. Contact angle data and particles sizes are summarized in Tables 1 (beads) and 2 (food powders).

Finally, we assessed the wettability by measuring the percentage of powder remaining at the air–water interface after 45 s (timescale relevant for the preparation of a beverage). Therefore, we used a tailor-made guillotine (see Fig. 3). 1.5 g of powder was released onto 110 mL of 20°C water via a first knife-gate. A second knife-gate, placed 1 cm below the interface is closed after 45 s. The powder still floating at the air–water interface is collected and dissolved in 300 mL water. The concentration of the solution can be deduced from a conductivity measurement. It is therefore possible to compute the percentage of the initial amount of powder remaining at the air–water interface.

3. Floating model

3.1. The case of non-soluble homogeneous spheres

When a homogeneous dense sphere is contacting the surface of a liquid, the sphere can either sink or float depending on the liquid and material properties of the solid. For the sake of clarity and completeness, this paragraph presents the model introduced in previous works (among others Scheludko [3], Ivanov [4], Keller [5] and Vella [6,7]) to predict the sinkability of a sphere.

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