



Wetting dynamics of colloidal dispersions on agar gel surfaces



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ABSTRACT

The effects of silica particle addition on the wetting velocity on flat and fractal agar gel surfaces were analyzed along with the applicability of such particles for controlling the wetting dynamics of water. The contact angles (θ_D) of the colloidal dispersions obeyed the power law, i.e., $\theta_D \propto t^{-x}$, where t is time and x is a constant. Wetting was inhibited by the addition of a suitable amount of 20-nm-diameter silica particles. Specifically, the exponent x reached a minimum value for a silica composition of 0.1 wt%. However, such inhibition effects were not observed upon the addition of silica particles with diameters of 100, 550, and, 1000 nm. The mechanism of the inhibition of the liquid wetting on gel surfaces may be attributed to a slight increase in local viscosity around the contact line during wetting.

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1. Introduction

The wetting dynamics of liquids at biological interfaces are crucial mass transport phenomena contributing to various biological functions in the human body, including nutritional absorption at small intestine walls, occurrence of dry eye syndrome on the corneal surface, and sensitization of the tongue surface [1–3]. Most biological interfaces are generally mucus-coated and complex systems exhibiting hierarchical roughness. Because of the difficulties arising during direct observations or physical evaluations, few reports have attempted to control the wetting dynamics at these interfaces [4–6]. Fractal agar gels, which present rough hydrophilic hierarchical surfaces, have recently been prepared as models for biological surfaces [7]. Their rough structure mimics the hierarchical structure of small intestine walls, which consist of circular folds, villuses, and microvilluses. The quantitative evaluation of the wetting velocity of water on fractal gel surfaces using a high speed camera showed that the ultra-rough structure of the gel surfaces accelerated water wetting. A theoretical model, based on the changes in the free energy associated with wetting phenomena,

was proposed to describe the wetting behavior on rough surfaces [8].

A deeper understanding of the wetting dynamics rests on the observation of the wetting process as well as the experimental and theoretical strategies to control this process. In general, the wetting dynamics of a liquid depend on the interfacial tension and viscosity of the liquid [9]. The characteristic moving velocity of three-phase contact lines can be described by γ/η , where γ and η are the surface tension and viscosity of the liquid, respectively [10]. Our previous study revealed the anomalous wetting behavior of aqueous alcohol solutions on agar gel surfaces and strong Marangoni flow at the three-phase contact line causing extremely fast wetting [11]. In contrast, emulsion droplets in water inhibited wetting on agar gel surfaces [12].

This study focuses on rheology effects on spreading dynamics. Studies on the spreading of droplets of complex fluids containing either surfactants or polymers indicated that both of them slowed down the spreading [13,14]. On the other hand, trisiloxane surfactants induced “superspreading,” in which the droplet spreads out in orders of magnitude faster than usual surfactants. The addition of colloidal particles might diminish liquid wetting because the dispersed solid particles form network structures and induce thickening. Rio et al. showed that a stick-slip phenomenon and a pinning effect resulted from particle aggregation at the three-phase contact line when a colloidal dispersion came in contact with a glass substrate [15]. Furthermore, using video microscopy, Wassen et al. demonstrated that charged nanometer-sized polystyrene spheres

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exhibited two-dimensional crystal-like ordering in water, and this solid-like order occurred in the confined three-phase contact region at the edge of the wetting fluid [16,17]. Koh and Wong utilized reflectance spectroscopy to monitor structural changes during the self-assembly of a monodisperse colloidal system at the three-phase contact line of a sessile drop on an inert substrate [18]. Solids dispersed in a drying drop migrated to the drop edge, forming a so-called “coffee ring.” This migration results from an outward flow within the drop driven by solvent loss through evaporation and geometrical constraints requiring that the drop maintains an equilibrium droplet shape with a fixed boundary [19–22]. Dewetting results in the formation of finger-like patterns near the contact line, leaving behind a deposit of branches. The high particle concentration in the contact line region strongly enhances viscosity, leading to strikingly low dewetting speeds. For pattern formation from drying particle-containing droplets, evaporation also causes dewetting [23]. When the particle concentration exceeds a threshold value, the particle size can be estimated by considering the collective effects of the droplet evaporation and diffusive particle motion within the droplet.

In this study, the wetting behavior of silica particle dispersions with 20-, 100-, 550-, and 1000-nm-diameters on agar gel surfaces was examined using a high speed camera to determine whether the colloidal particle addition affects the dynamics of the liquids on gel surfaces. The liquids' wetting behavior on fractal gel surfaces was also investigated to clarify the effects of colloid size or gel surface roughness on the wetting velocity. Finally, a phenomenological physical model based on the experimental findings was proposed to describe the wetting behavior of colloidal dispersions. Low toxicity silica nanoparticles were chosen because they may provide means to regulate wetting dynamics in consumer product applications, such as skin care products and cleansing products. For example, wetting inhibition agents may be applicable to controlled and effective drug delivery approaches in the future.

2. Experimental

2.1. Materials

Agar powder was purchased from Kanto Chemical Co. (Tokyo, Japan) and used without further purification. Amorphous Aerosil 200 silica particles (mean particle diameter: 20 nm) were obtained from Nippon Aerosil Co., Ltd. (Tokyo, Japan) and spherical COSMO55 silica particles (mean particle diameter: 550 nm) were purchased from JGC Catalysts and Chemicals Ltd. (Kanagawa, Japan). Silica particle dispersions with diameters of 100 nm (sicastar 43-00-102, 50 mg mL⁻¹) and 1000 nm (sicastar 43-00-103, 50 mg mL⁻¹) were obtained from Nacalai Tesque, Inc. (Kyoto, Japan), along with a 30-nm-diameter fluorescent particle dispersion (sicastar green F 42-00-301, 50 mg mL⁻¹). Water was purified using a DX-15 deionizing unit obtained from Kurita Water Industries Ltd. (Tokyo, Japan).

2.2. Preparation

Flat and fractal agar gels were prepared as described in a previous study [7]. Briefly, a mixture of agar powder (6 g) and deionized water (144 g) was heated and agitated until complete dissolution. The 4 wt% agar aqueous solution (150 g) was poured into 9-cm-diameter Petri dishes and allowed to cool and solidify into the flat agar gel. Fractal agar gels were also prepared by a previously reported method [7,11]. Melted alkylketene dimer (AKD) was solidified in Petri dishes to produce fractal surface templates, serving as molds for plaster replicas. Agar aqueous solution was poured into the replica-containing dishes. Box-counting measurements

confirmed that the surface structures were fractals with a fractal dimension D of 2.2. The self-similar features were found to hold from 4 μm to 30 μm of a box size. Agar gel surface geometries were examined using a VK-X100 laser microscope obtained from Keyence (Tokyo, Japan). The roughness was as follows: (1) Flat agar gels exhibited roughness parameters R_a , R_z , and R_y of 0.026, 0.316, and 0.430 μm , respectively, and (2) Fractal agar gels displayed R_a , R_z , and R_y of 2.14, 31.7, and 35.7 μm , respectively. Silica particles were mixed with deionized water for 30 s in a screw-cap test tube using a Vortex-Genie 2 mixer obtained from Scientific Industries Inc. (New York, USA) and for 30 s using an NR-50 M handheld ultrasonic homogenizer obtained from Microtec Co., Ltd. (Chiba, Japan) to obtain silica particle dispersions.

2.3. Measurements

The contact angles of the 0.5- μL -drops of liquids on the gel surfaces were estimated by a sessile drop measuring method using a DM-501 contact angle meter obtained from Kyowa Interface Science (Saitama, Japan). Measurements were conducted in air at 298 K and 50% humidity. Liquid droplets were dropped from the tip of a 22G tetrafluoroethylene-coated syringe (internal diameter: 0.4 mm, external diameter: 0.7 mm) using an automatic dispenser designed for the DM-501 instrument. Images were magnified by approximately 32-folds and displayed through a 17-inch screen at the resolution of 20 μm per pixel. The measurement of contact angles below 20° is normally difficult. Therefore, the accuracy of these values was double-checked: this approach gave an accuracy within 1.6° for a contact angle of 6.7° (see Supporting information Tables S1 and S2) [24]. Temporal changes in contact angles were measured at 1-ms-intervals for ten different points and averaged. The initial volumes of the colloidal dispersions on the flat and fractal agar gel surfaces were determined using the equation $V = (\pi/3)h^2(3r - h)$, where r and h are the radius and height of the droplet. Average apparent initial volumes amounted to 0.28–0.43 and 0.29–0.47 μL on the flat and fractal agar gel surfaces, respectively. The liquid volume decreased by several tens percent within 1 s of instillation due to evaporation. However, assuming that $\theta_D(t)$ obeyed a time-dependent power law of the form $\theta_D(t) \propto t^{-x}$, the exponent x , also called wetting velocity, was constant for initial volumes ranging 0.3–0.7 μL , indicating that initial volume variability and temporal changes had a limited effect on the wetting dynamics of the liquids. The influence of the evaporation-induced variations in wetting droplet volume on the time-related contact angle changes during wetting was investigated. This evaluation confirmed that the changes in the contact angle could be neglected, at least within 1 s of instillation [11]. Liquid surface tensions were estimated by the Young–Laplace method using the DM-501 contact angle meter for 10 μL water drops. The surface tension values of the colloidal dispersions are shown in the Supporting Information (Table S3). Dispersion viscosities at 298 K were determined from their kinetic viscosity and density. The kinetic viscosity was measured using a VM-1A-L vibratory viscometer obtained from Yamaichi Electronics Co., Ltd. (Tokyo, Japan). The density was measured using a pycnometer. The viscosity values of the colloidal dispersions are shown in the Supporting information (Table S2). The amounts of particles or solution were estimated by fluorescence intensity measurements during wetting on agar using an inverted fluorescence microscope (IX71, Olympus) equipped with a 100 W mercury lamp (U-LH100HGAPO, Olympus), as well as UPlan-FLN4X and LUCPlanFLN40X objective lenses (Olympus, Japan). The excitation and emission wavelengths of fluorescent silica beads and rhodamine B were selected using a 460–495 nm bandpass filter (BP460-495) and a 510 nm barrier filter (BA510IF), respectively. Fluorescence images were acquired using a complementary metal oxide semiconductor camera. A sample gel was placed on a cover

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