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Characterization of the effect of food emulsifiers on contact angle and dispersibility of lipid coated neutrally buoyant particles

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Abstract

To investigate the dispersibility of lipid coated particles in food suspensions, contact angle of lipid surface at different pH, salt concentration, protein concentration, surfactant type and concentration was measured. Contact angle decreased with an increase in the emulsifier conecentration when the concentration was low, and reached a fairly constant value at higher concentrations. Whey protein was more efficient compared to other emulsifiers and decreased the contact angle from 100° to 40° at a concentration of 0.5 g/L or higher in water. Tween 20 was more efficient than other tween emulsifiers and it decreased the contact angle from 100° to 67° at a concentration of 1 g/L. Surface pressure area isotherm of 150-80SV soybean oil coated particles at an air–water interface was obtained using Langmuir trough. Contact angle of the particles at the air–liquid interface, inferred from the critical surface pressure in the isotherm, agreed well with the contact angle of planar surface obtained using goniometer. Dispersibility of 150-80SV soybean oil coated particles in aqueous solution was characterized by measuring the bulk particle concentration upon suspension. At pH 2.5, 5 g/L whey protein in the presence of 0.5 mol/L NaCl in 50 g/L citrate buffer showed highest dispersibility.

Keywords: Dispersibility; Contact angle; Goniometer; Whey protein; Tween; Lipid surface

1. Introduction

Micronutrient fortified foods, including beverages, are becoming increasingly important in many countries. Iodine on salts, iron and vitamin C in milk or drink yogurt, and calcium in orange juice are typical examples of micronutrient fortification in various foods. The contribution to micronutrient intakes from fortified foods in the US ranged from 6% for vitamin B6 and folic acid to 24% for iron and vitamin (Lachance, 1989). Iron deficiency affects approximately 20% of the world population and is considered to be the commonest nutritional deficiency (Martinez-Navarrete, Camacho, Martinez-Lahuerta, Martinez-Monzo, & Fito, 2002). Iron deficiency persists although it is abundant in the food supply and the nutritional requirements for it are low. The Surgeon General (HHS, 1988) and the National Research Council (1988) recommended that adolescents and women increase their intake of iron rich foods (Martinez-Navarrete et al., 2002). Iron fortification could increase nutritive value and consumer appeal of dairy and other food products. Main barriers of iron fortification are finding an iron compound that adequately absorbed but no sensory changes to the food systems and overcoming the inhibitory effect on iron absorption of dietary components such as phytic acid, phenolic compounds and calcium (Hurrell, 2002). Water-soluble iron compounds, which are readily bioavailable, often lead to the development of unacceptable color and flavor changes in the food system, while insoluble iron powders give little or no nutritional benefit with very poor absorption causing no sensory changes. Ferrous sulphate and ferrous fumarate can be used instead of elemental iron for better absorption.

Success of iron fortification in dairy and other food products depends on encapsulation of ferrous sulphate and/or ferrous fumarate to prevent lipid unacceptable color and flavor change. In addition, it is necessary to ensure that iron particles are uniformly suspended in the

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food system. Microencapsulation of iron by an inert would alleviate these problems by first minimizing the effects of lipid oxidation and secondly making the particles neutrally buoyant. Spray drying accounts for the majority of commercial encapsulated materials in food products. Coacervation, coating with fat and spray chilling are the other techniques employed for encapsulation (Schafer & Schafer, 2003). Encapsulation has been shown to result in ease of handling, stability against oxidation, retention of volatile ingredients and flavor, taste masking and enhanced bioavailability and efficacy. Microencapsulation has been evaluated by several investigators (Pakarek, Jacob, & Mathiowitz, 1994; Teipel, Heintz, & Krober, 2001) and a comprehensive review of this technique is given by Clark (2002). Excellent discussions of various factors that influence adhesion of particles onto air–liquid interface as related to flotation are given by Gaudin (1957) and Fuerstenau (1980). Wettability and flotability of minerals as a function of surface tension were investigated by Kelebek, Finch, Yoruk, and Smith (1986). Theoretical models for the prediction of flotability of minerals at air–solution interfaces are discussed by Jowett (1980) and Clarke and Wilson (1983). However, the inert coating material should be sufficiently hydrophilic to ensure wettability of coated iron particles so that they are easily dispersed in the aqueous medium. This paper addresses the effect of surface active agents on wettability and dispersability of neutrally buoyant lipid coated particles.

2. Materials

Tween 20, Tween 40, Tween 60, Tween 80 and 2-propanol (Sigma-Aldrich, USA); whey protein (SCIFIT, PA); NaCl, citric acid and Na-citrate (Mallincktodt Baker, NJ), 150-80VS (Loders Croklaan, IL, lot # E 3009-H) encapsulated citric acid with a 20% coating of a hydrogenated soybean oil (melting point of 152–158 °F with an IV of around 5–10) and particle size maximum of 840 μm.

3. Methods

3.1. Contact angle by goniometer

Microscopic glass slides were coated with hydrogenated soybean oil (melting point of 152–158 °F with an IV of around 5–10) by dipping the slide in hot-molten oil and cooling thereafter. Soybean oil-coated slides were immersed in surfactant solutions overnight (16 h) in order to allow the surfactants to adsorb onto the lipid surface and attain equilibrium. The slides were then removed from the solution and dried. A 2 mm diameter droplet of surfactant solution of the same concentration as that employed for treating the lipid surface was formed on the modified slides. The slide with the droplet was placed in a humid chamber in order to prevent evaporation of solvent from the droplet. Contact angle was measured by contact angle meter (Ramehart 50-00) at different times. These experiments were repeated several times.

3.2. Contact angle by langmuir-trough

Fifty g/L citrate buffer (pH 4.0) was poured into a computer-controlled Langmuir minitrough (KSV, Finland) and aspiration was applied carefully on the surface to remove possible surface-impurities. Hydrophobic particles 150-80VS dispersed in 2-propanol were placed at the air–water interface (Pauvnov (2003)). Two-propanol was allowed to evaporate for 15 min. The surface was then compressed at a compression rate of 5 cm^2/min. The surface pressure and surface area were recorded simultaneously.

3.3. Particle dispersion

Fifty g/L citrate buffer (pH 4.0) was prepared. One g/L of whey protein was dissolved in the buffer. One hundred and twenty mL of this solution was poured into a 400 mL beaker (ID = 73 mm). Desired amount of 150-80VS particle was added into the solution. After magnetic stirring for 5 min, the dispersion was allowed to rest for 5 min. A layer of particles was observed at the air–liquid interface. Bulk dispersion was taken out from the middle of the dispersion using a 5 mL pipet. Forty mL solution was taken out and filtered with S&S quantitative filter paper (Aldrich, Milwaukee, USA). The dry mass of filter paper was measured before filtration. The wet paper with particles was allowed to dry overnight and the total mass of filter paper with particles was measured. A control experiment was performed without whey protein in the solution.

4. Results

4.1. Dynamics of contact angle measurement

Typical plot of contact angle versus time for pure water on the lipid surface is shown in Fig. 1. The contact angle does remain fairly constant from 10 to 50 min. At much longer times, there is a decrease in contact angle possibly because of evaporation of water from the droplet. Only the measurements within the range of 10–50 min are reported below.

4.2. Contact angle by langmuir-trough

Π–A isotherm of the hydrophobic particles is shown in Fig. 2. Applying Clint and Taylor's method (Clint & Taylor, 1992), critical surface pressure Π_c was obtained and related to contact angle by

$$\Pi_c = \frac{\pi\gamma_{LA}(1 \pm \cos\theta)^2}{2\sqrt{3}},$$

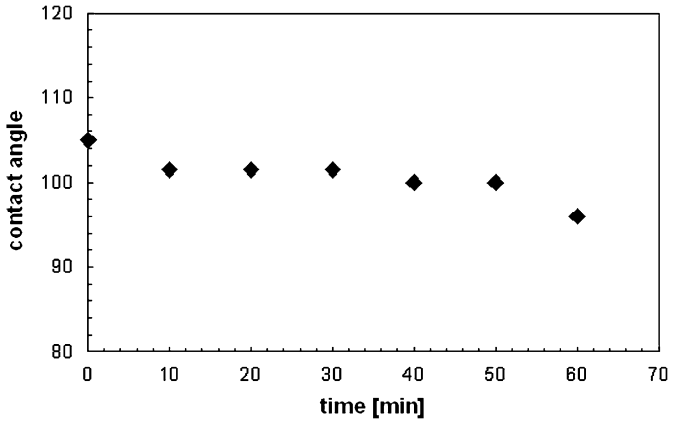

Fig. 1. Contact angle of pure water on lipid-coated slides at different times.

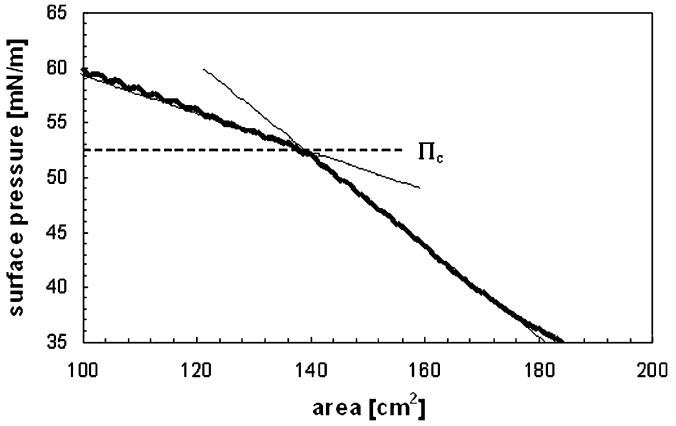

Fig. 2. Π–A isotherm of particle 150-80VS at air–water interface. Liquid phase is 50 g/L citrate buffer (pH 4.0).

where γ_{LA} is the air–liquid surface tension and θ is the contact angle. The "$\pm$" means there are two possible situations to give the same Π_c, which depends on whether the particles move into air or liquid phase when they are compressed. The contact angle for the lipid coated particles obtained by the Π–A isotherm is $95.8 \pm 0.9^\circ$ assuming that particles move into air when compressed. This value agreed well with that of planar surface as measured by goniometer. The contact angle obtained using goniometer is the contact angle of a small droplet on a planar solid surface, whereas that obtained by Langmuir trough is the average contact angle of small particles at a liquid surface. It is impossible to get the Π–A isotherm of particles when protein/surfactant is present in the liquid phase, because the protein/surfactant would also adsorb at the interface. The compression of protein/surfactant adsorption layer would also contribute to the surface pressure variation. As a result, the variation of surface pressure is not solely due to the compression of particles. The good agreement between the contact angles from goniometer and Langmuir trough measurements suggest that the contact angle of a particle is close to that of a planar surface.

4.3. Evaluation of emulsifiers

The effect of Tween 20 and whey protein concentrations on contact angle are shown in Figs. 3 and 4, respectively. As expected, the contact angle decreased at higher surfactant concentration. This decrease was pronounced only at lower Tween 20 concentrations (up to 1 g/L). At higher Tween 20 concentrations, however, the contact angle became fairly constant. In case of whey protein, most of the decrease in contact angle occurred at much lower concentrations (Fig. 4). Fig. 5 compares the contact angles of lipid surface treated with different surfactant solutions for 16 h. Among the surfactants, Tween 20 reduced the contact angle the most whereas whey was the most efficient in that it resulted in the minimum contact angle.

4.4. Effect of pH

The results of contact angle measurements for lipid coated glass slides at different pH in the presence of 0.5 g/L

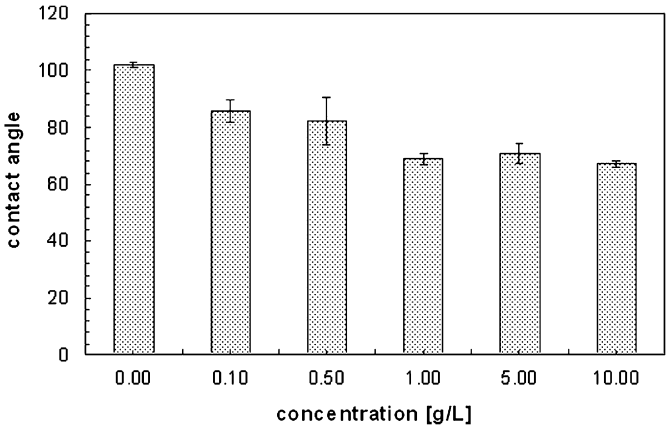

Fig. 3. Contact angle of lipid coated slides treated with different concentrations of Tween 20 solutions. Slides were immersed in surfactant solutions for 16 h and the measurement was made after 15 min.

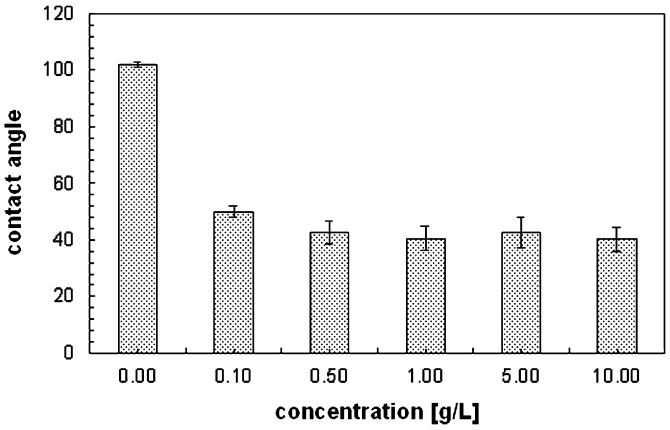

Fig. 4. Contact angle of lipid coated glass slide treated with whey protein of different concentrations. Slides were immersed in protein solutions for 16 h and the measurement was made after 15 min.

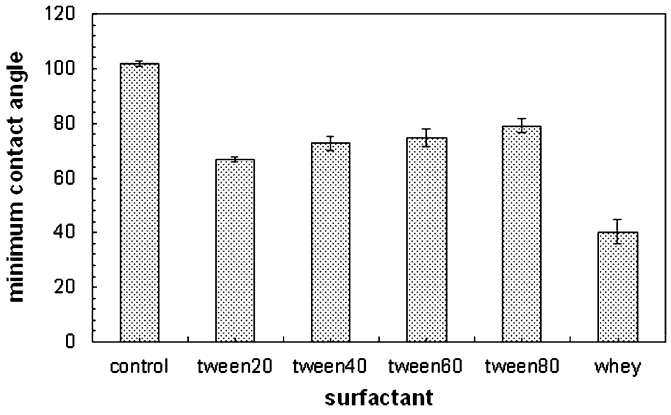

Fig. 5. Minimum contact angle of lipid coated glass slide treated with different surfactant solutions. Slides were immersed into the surfactant solution for 16 h. Contact angles were measured after 15 min.

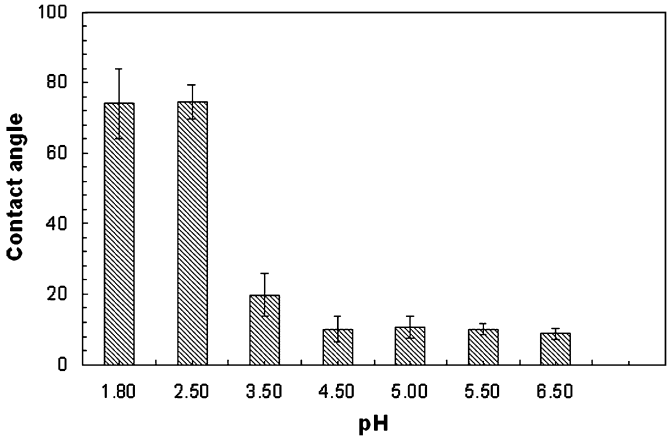

Fig. 6. Contact angle of lipid coated slides treated with 0.5 g/L whey protein solution (50 g/L citrate buffer). Slides were immersed in the solution for 16 h and the measurement was made after 15 min.

whey protein solution are shown in Fig. 6. The contact angle reduces dramatically from a high value of around 70° at pH 2.5 and below to a limiting low value of around 10° for pH 4.5 and above. Therefore, whey protein in the presence of citric acid buffer is found to be very efficient in increasing the wettability of lipid surface. Fig. 7 compares the contact angle of lipid coated glass slide with different treatments. Citrate buffer did not reduce the contact angle of lipid coated glass slide significantly. However, 0.5 g/L whey protein solution in citrate buffer at pH 3.5 did considerably reduce the contact angle. Interestingly, 0.5 g/L whey protein solution in water was able to reduce the contact angle only to around 40°. Also, the contact angle of the surface was even higher (around 50°) when exposed to 0.5 g/L whey protein solution in phosphate buffer at pH 3.5.

Results of contact angle measurements for Tween-20 at different pH are shown in Fig. 8. In the range of pH 2.0–7.0, pH does not show much effect on the contact angle. Contact angles at low pH are slightly lower than those at high pH.

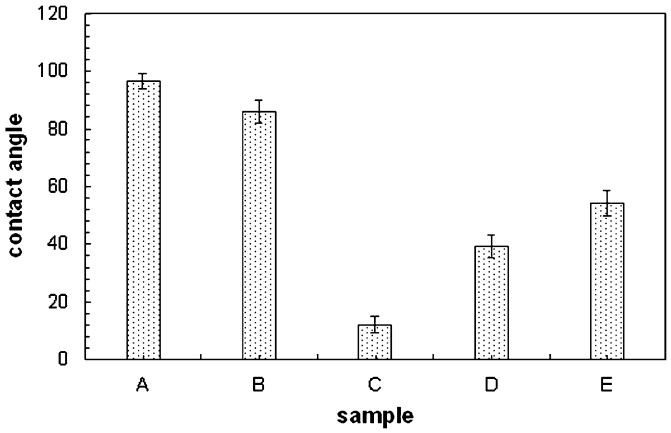

Fig. 7. Contact angle of lipid coated slides treated with different solutions. (A) Pure water; (B) 50 g/L citrate buffer, pH 3.5; (C) 0.5 g/L whey protein in 50 g/L citrate buffer, pH 3.5; (D) 0.5 g/L whey protein in pure water and (E) 0.5 g/L whey protein in 0.01 mol/L phosphate buffer, pH 3.5. Slides were immersed in the solution for 16 h and the measurement was made after 15 min.

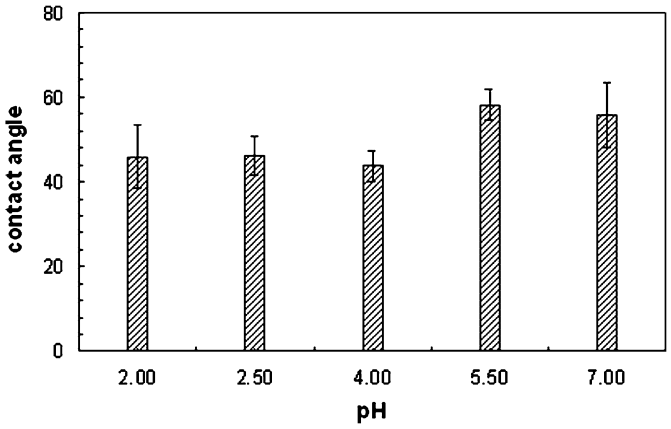

Fig. 8. Contact angle of lipid coated slides treated with 1 g/L Tween-20 solution at different pH (50 g/L citrate buffer). Slides were immersed in the solution for 16 h.

4.5. Effect of ionic strength

At low pH, e.g. pH 2.5, even high concentration of whey protein does not decrease the contact angle very much. At higher pH, however, the contact angle is found to be much lower. This may be due to the effect of ionic strength. At higher pH, higher concentration of Na-citrate in the buffer may contribute to higher ionic strength which may play a role in the reduction of contact angle. The results of contact angle measurements for lipid coated glass slides at different ionic strength in the presence of different concentration of whey protein solution at pH 2.5 are shown in Fig. 9. The contact angle decreased with increasing ionic strength at each protein concentration. At each ionic strength, contact angle decreased with increasing protein concentration. Therefore, high protein concentration and high ionic strength are favorable for

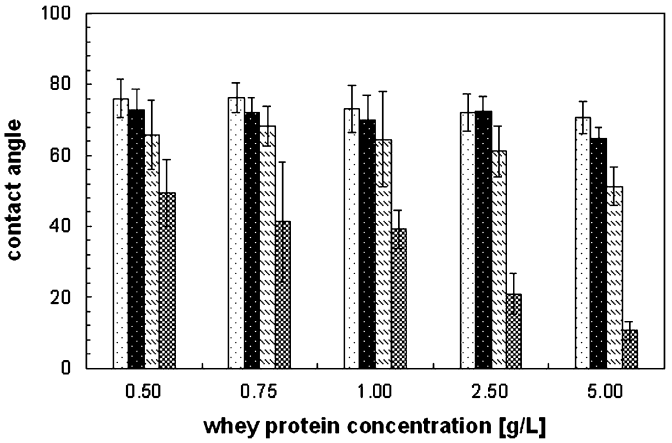

Fig. 9. Contact angle of lipid coated slides treated with whey protein solution at different concentration and different NaCl concentration (50 g/L citate buffer, pH 2.5). Slides were immersed in the solution for 16 h. [NaCl] = 0 mol/L [NaCl] = 0.05 mol/L [NaCl] = 0.1 mol/L; [NaCl] = 0.5 mol/L.

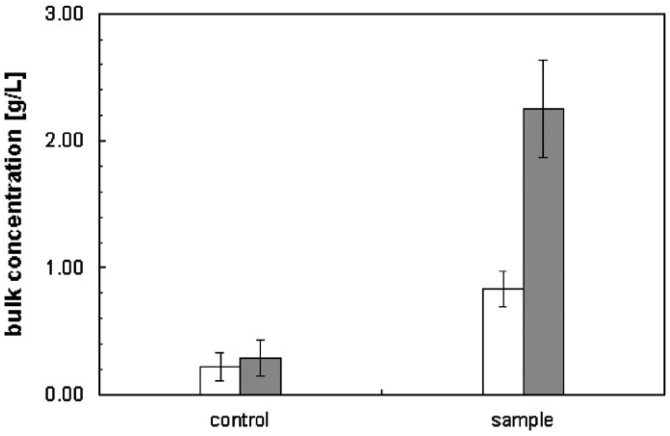

Fig. 10. Bulk concentration of particle dispersion with low (5 g/L) and high (15 g/L) initial concentration. □ initial concentration 5 g/L; ■ initial concentration 15 g/L.

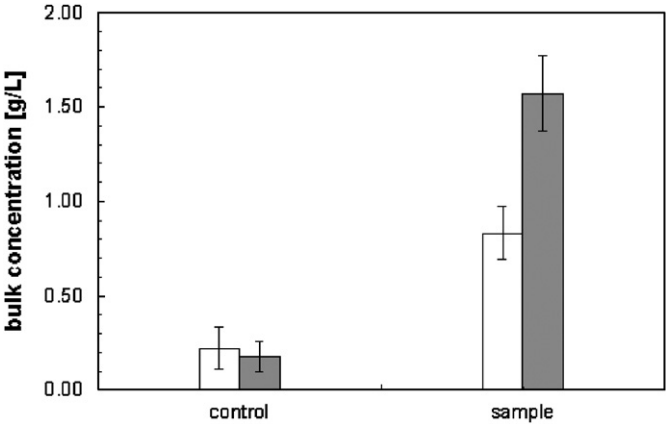

Fig. 11. Bulk concentration of particle dispersion with stir and 5 min rest after stir was stopped. Initial particle concentration is 5 g/L. □ rest for 5 min; ■ with stir.

wettability of the hydrophobic surface. When the whey protein concentration is 5 g/L and the NaCl concentration is 0.5 mol/L, the contact angle decreases to $10.6 \pm 2.7^{\circ}$.

4.6. Particle dispersion

A set of experiments were conducted to investigate the effect of whey protein in the prevention of flotation of lipid coated particles. Lipid coated particles were suspended in 1 g/L whey protein solution in citric acid buffer at pH 4 for 5 min by stirring. Stirring was then stopped and samples were then taken from the bulk solution after 5 min to determine the particle concentration. Since all the particles were not neutrally buoyant, particles that were lighter rose to the top to form a cream layer. As a result of this loss of particles, there was a decrease in particle concentration in the bulk. The final bulk particle concentration was found to be about 1/7th of the initial concentration for low as well as high initial particle concentrations indicating thereby that the particles were not really neutrally buoyant. Interestingly, upon creaming, lipid coated particles in the presence of whey formed a cream layer in which they were fully wetted. However, in case of lipid coated particles suspended in citric acid buffer (control), most of the particles were lost to the air–water interface (Fig. 10). Also, in this case, the particles resided at the interface since they were not fully wetted. In fact, at higher initial particle concentration, one could observe dry particles floating at the surface. In another set of experiments (Fig. 11), particles of 5 g/L initial concentration were suspended for 5 min by stirring. In one set of experiments, a sample was withdrawn from the bulk. In the other set, stirring was stopped and a sample was withdrawn from the bulk after 5 min. In case of control, there was insignificant difference in the particle concentration between the two sets. However, in case of particles in 1 g/L whey solution, stirring during sampling gave a much higher particle concentration. This difference can be attributed to the fact that whey protein solution wets the particles.

The effect of protein concentration on the wettability of whey protein in the presence of 0.5 mol/L NaCl at pH 2.5 is shown in Fig. 12. For the initial concentration of 10 g/L, the bulk concentration was about 2 g/L when the protein concentration was 5 g/L, compared to the bulk concentration of 0.45 g/L when protein concentration was 1 g/L. Bulk concentration of particle dispersion in 1 g/L Tween-20 solution at pH 2.5 is also shown in Fig. 12. The bulk concentration was about 0.7 g/L, which was higher than that in 1 g/L whey protein solution, but was much lower than that in 5 g/L whey protein solution.

4.7. Effect of Ca^{2+} on contact angle

To investigate the effect of purity of whey protein and hardness of water on the contact angle, β-lactoglobulin (Sigma L0130) and α-lactalbumin (Sigma L5385) at the ratio of 4:1 was used to mimic the whey protein. DI water (18 MΩ) was used and 0.1 g/L $CaCl_2$ was added to increase

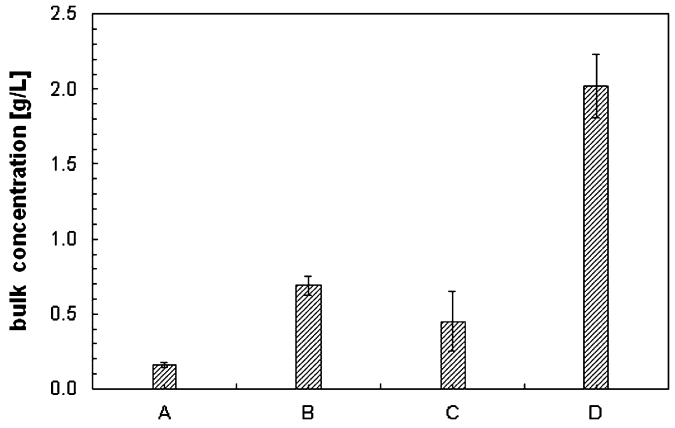

Fig. 12. Bulk concentration of particle dispersion with 5 min rest after stir was stopped. Fifty g/L citrate buffer (pH 2.5) and initial particle concentration is 10 g/L. (A) control; (B) 1 g/L Tween 20; (C) 1 g/L whey protein with 0.5 mol/L NaCl; (D) 5 g/L whey protein with 0.5 mol/L NaCl.

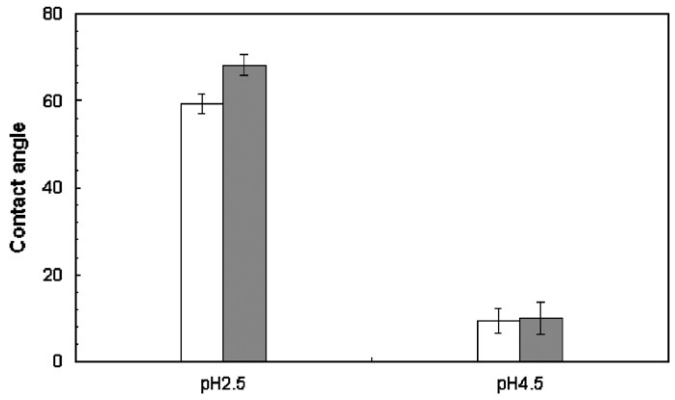

Fig. 13. Effect of Ca^{2+} on contact angle of lipid surface at two different pH. Liquid phase are 50 g/L citrate buffer with 0.8 g/L β-lactoglobulin and 0.2 g/L α-lactalbumin. □ 0.1 g/L $CaCl_2$; ■ 0 g/L $CaCl_2$.

the hardness of water. Measurements were made at pH 2.5 and pH 4.5 as shown in Fig. 13. The contact angle at pH 4.5 is much lower than that at pH 2.5. The contact angles of the lipid treated with the mixture of β-lactoglobulin and α-lactalbumin were similar to those treated with whey proteins. Addition of $CaCl_2$ did not significantly affect the contact angle.

4.8. Discussion—free energy of dispersion

Adhesion of particles onto the air–water interface will depend on the hydrophobicity of the particle surface. When the food suspension consisting of the suspension of particles is mixed, the particles can either (i) be transported to the air–water interface at the top or (ii) the particles can collide with the air bubbles formed by mixing. Particles of size much smaller than the air bubbles will anchor at the bubble interface and the extent of penetration of the particle into the air bubble will depend on the contact angle.

For lipid coated particles much smaller than the bubble size, the curvature of the air bubble can be neglected and the air–water interface can be assumed to be planar. In the absence of gravitational effect, the interface will be planar without the formation of any meniscus. The area of contact between the particle and air is $2\pi R^2(1-\cos\theta)$. The flat area of water surface covered by the particle is $\pi R^2(1-\cos^2\theta)$. Therefore, the free energy E to remove the particle from the water surface into water is

$$E = 2\pi R^2(1-\cos\theta)(\gamma_{SL}-\gamma_{SA}) + \pi R^2(1-\cos^2\theta)\gamma_{LA}, \quad (1)$$

where γ_{SA} and γ_{SL} refer to the solid air and solid liquid surface free energies respectively and γ_{LA} is the surface tension of the liquid. Young's equation for equilibrium at the three phase contact line is given by

$$\gamma_{SA} - \gamma_{SL} = \gamma_{LA}\cos\theta. \quad (2)$$

Combining Eqs. (1) and (2), one obtains,

$$E = \pi R^2\gamma_{LA}(1-\cos\theta)^2. \quad (3)$$

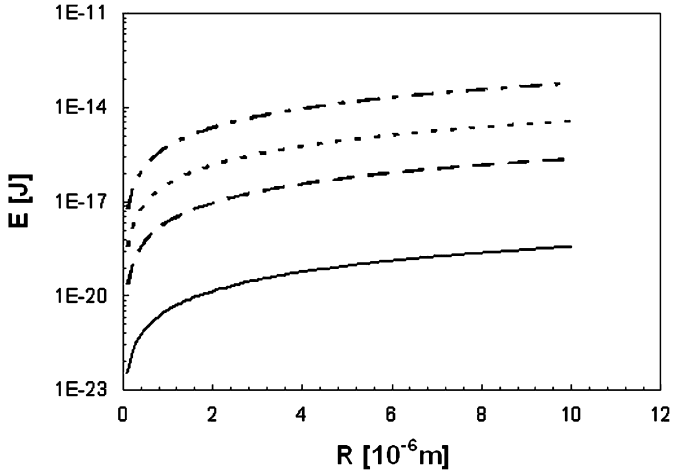

Fig. 14. Free energy of moving a particle from the surface to the bulk versus particle radius for different contact angles. ___1°; – – – – 5°; - - - - - 10°; –·–·– 20°.

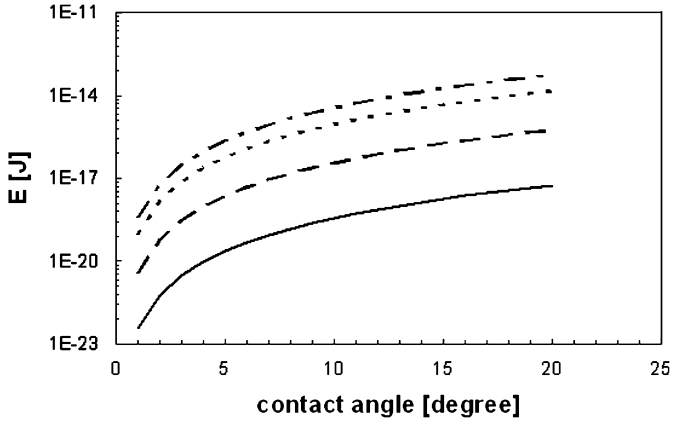

Fig. 15. Free energy of moving a particle from the surface to the bulk versus contact angle for different particle radius. —— 0.1 μm ; – – – – 1 μm; - - - - - 5 μm ; –·–·– 10 μm.

The change in free energy for moving a particle from the air–water interface to the bulk for different particle sizes and contact angles are shown in Figs. 14 and 15. It clearly shows that decreasing the particle size, in addition to decreasing contact angle, is also important and effective in decreasing the free energy needed to disperse the particles.

5. Conclusion

Micronutrient fortified foods, including beverages, are becoming increasingly important in many countries. The contribution to micronutrient intakes from fortified foods in the US ranged from 6% for vitamin B6 and folic acid to 24% for iron and vitamin. Iron fortification could increase nutritive value and consumer acceptance of dairy and other food products. Success of iron fortification depends on encapsulation to prevent unacceptable color and flavor change as well as enable uniform suspension of microparticles in the food system. However, without proper treatment, the inert surface would adhere to the air-aqueous phase interface thus compromising the texture of food suspension. To study the wettability of lipid coated particles in food suspension, contact angle of lipid surface at different pH, salt concentration, protein concentration, surfactant type and concentration was measured by a goniometer. Contact angle of the treated lipid surface was characterized by viewing a 2 mm diameter droplet of surfactant solution with a Ramehart 50-00 goniometer. Contact angle decreased with an increase in the surfactant concentration when the concentration was low. At higher concentration, the contact angle was fairly constant. Whey protein was the most efficient compared to other surfactants and decreased the contact angle from 100° to 40° at a concentration of about 0.5 g/L or higher.Tween 20 was more efficient than other tween surfactants and it decreased the contact angle from 100° to 67° at a concentration of 1 g/L. Dispersibility of 150-80SV soybean oil coated particles in aqueous solution was characterized by the measurement of bulk particle concentration upon suspension. Most of the lipid coated particles were lighter than the solution thus resulting in considerable loss from the bulk. Particles do not wet the buffer solution and therefore reside at the air–water interface. However, particles wet whey protein solution because of which they tend to form a cream layer at the top. Stirring was found to decrease particle loss due to creaming. Π–A isotherm of 150-80SV lipid coated particle at air–water interface was obtained using Langmuir trough. Contact angle of the particles at the air–liquid interface was inferred from the critical surface pressure in the isotherm. The contact angle from this technique agreed well with the contact angle of planar surface that was measured using goniometer. Wettability of solutions was characterized by the measurement of bulk particle concentration upon suspension. At pH 2.5, 5 g/L whey protein in the presence of 0.5 mol/L NaCl in 50 g/L citrate buffer showed highest dispersibility, while 1 g/L whey protein solution with 0.5 mol/L NaCl and Tween-20 solution showed much poorer dispersibility.

Acknowledgements

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