

# SEM Studies of Surfactant-Assisted Micro-Mixing of Melamine and Wax Particles

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**ABSTRACT:** The validity of the micro-mixing phenomenon was investigated by mixing a melamine dispersion, stabilized by low molecular mass poly(acrylic acid) (~5000 Daltons) and a wax emulsion, stabilized by an ethoxylated alkyl phenol surfactant. The wax particles were observed to be attached to the melamine surface because of the interaction between the poly(acrylic acid) dispersant and the ethoxylate group of the wax surfactant, resulting in the

sedimentation of mixed particles. This phenomenon has potential application in the mixing of solid particles that require intimate contact and homogeneous mixing. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 99: 2554–2557, 2006

**Key words:** micro-mixing; surfactant; melamine dispersion; wax emulsion; poly(acrylic acid)

## INTRODUCTION

Polymeric surfactants have been known to interact with particle surfaces via various mechanisms (electrostatic forces, van der Waals forces, and hydrogen bonding), and thus, influence the dispersion stability of the solid particles.<sup>1</sup> Applications have included the use of nonionic surfactants at different concentrations to modify the stability of coal suspensions;<sup>2</sup> high molecular mass hydroxyethylcellulose to induce depletion flocculation of polymerically stabilized polystyrene particles;<sup>3</sup> and nonionic surfactant to agglomerate ink particles during paper deinking.<sup>4</sup>

A novel application for polymeric surfactants is surfactant-assisted micro-mixing. Inherently, dry mixing of two solid particles does not allow effective deagglomeration or homogeneous mixing, as is required for example in pyrotechnic delay elements. However, if it is attempted to first disperse the particles in individual dispersions and subsequently mix the two dispersions, the interaction between the suitably chosen dispersants should result in the flocculation of a homogeneous mixture. This concept was investigated using melamine particles, stabilized by low molecular mass poly(acrylic acid), and wax particles, stabilized by an ethoxylated alkyl phenol surfactant. The poly(acrylic acid) and the ethoxylated surfactant interact by hydrogen bonding, thus facilitating the formation

of melamine-wax agglomerates that flocculate out. Figures 1 and 2 show the proposed interaction between the particles.

## EXPERIMENTAL

### Materials

The melamine (Degussa, 99%) and water-based wax emulsion (Atebin A1 Micro, Böhme Africa, 50% solids) were both used as received. The poly(acrylic acid) (Goodrite K732, Carst and Walker) was diluted to a 10% (w/w) aqueous solution.

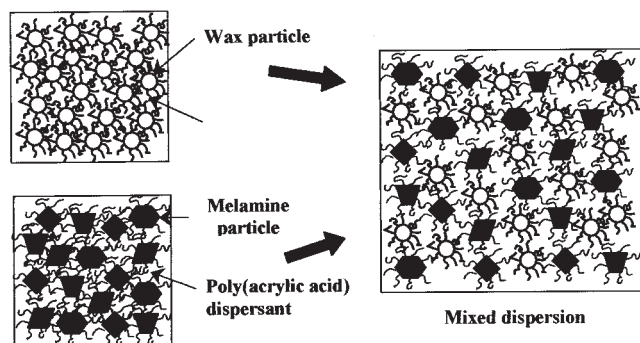
### Melamine dispersion

10% (w/w) Melamine dispersions were prepared by using an increasing amount of poly(acrylic acid) dispersant so as to determine the point at which a stable dispersion is obtained. The prepared dispersions were gently mixed in an end-over-end tumbler for 48 h to ensure reaction of the melamine with the poly(acrylic acid) dispersant. In addition, a control was prepared without the presence of the poly(acrylic acid) dispersant so as to ensure that the melamine and wax do not inherently interact.

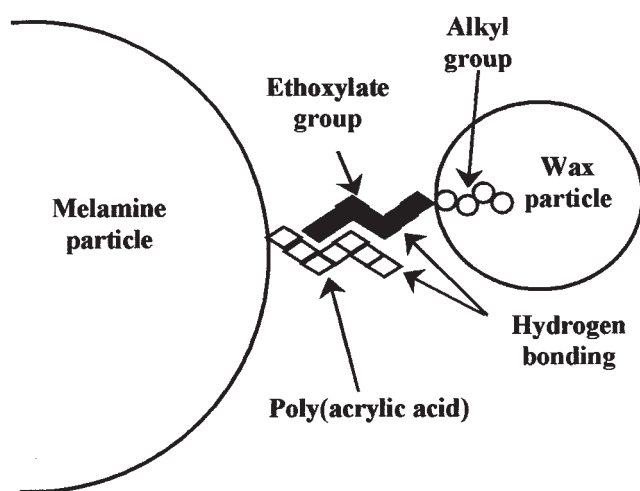
### Micro-mixing

The micro-mixing was based on a 1:1 volume melamine: volume wax. The wax emulsion was added to the melamine dispersion and the mixture was centrifuged for 20 min at 3000 rpm at ambient conditions.

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**Figure 1** Schematic diagram of surfactant-assisted micro-mixing.



**Figure 2** Schematic representation of hydrogen bonding between the poly(acrylic acid) and the ethoxylate group of the alkyl phenol ethoxylate surfactant.

The supernatant was removed and the sediment obtained was prepared for analysis as explained below.

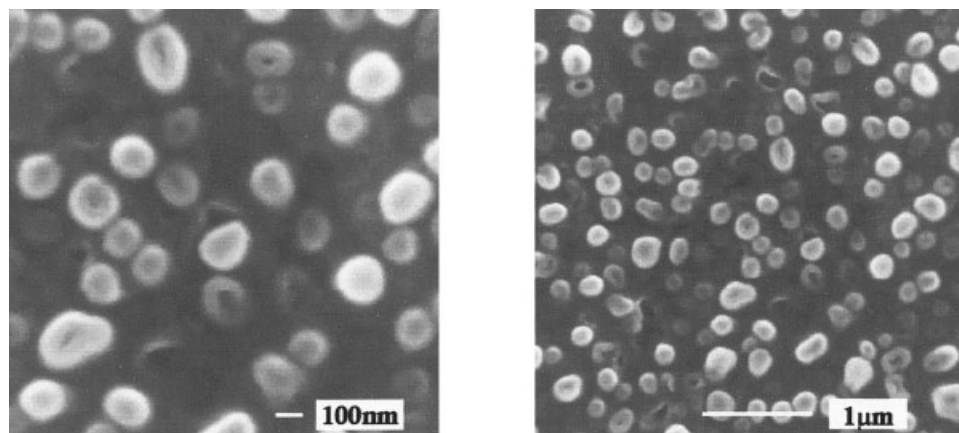
### Analysis techniques

The wax, melamine, and sediment particles were viewed using scanning electron microscopy (SEM).

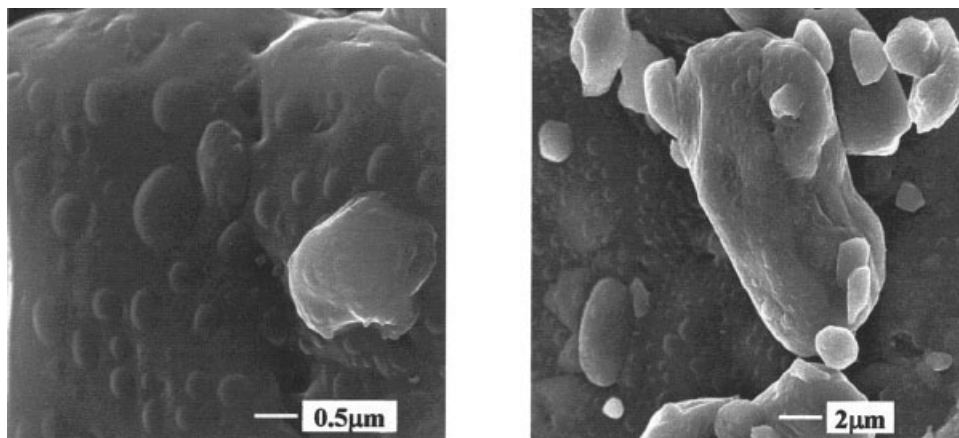
Samples of the wax emulsion were placed on graphite stubs that were subsequently freeze dried in liquid propane at  $-180^{\circ}\text{C}$  with a Reichert KF 80 freeze plunger. The frozen samples were transferred under liquid nitrogen into slots in a copper block ( $63 \times 63 \times 15 \text{ mm}^3$ ). The copper block was kept completely immersed in liquid nitrogen in a plastic container, thus keeping the material under nitrogen atmosphere and preventing the condensation of moisture onto the sample. The copper block was thereafter transferred to a Fisons high vacuum unit where the evacuation started immediately. Freeze-drying was carried out over 2 days, over which the temperature steadily increased back to room temperature. The samples were coated with chromium in a Gatan Ion beam coater, model 681. The melamine and sediment particles were placed on the polished side of graphite stubs, using double-sided carbon tape, and the excess particles were removed. Samples were coated with chromium; however, charging of the particles was observed and the sample was thereafter coated with ruthenium tetroxide. All the particles were viewed with a JEOL 6000F in-lens field emission scanning electron microscope.

### RESULTS AND DISCUSSION

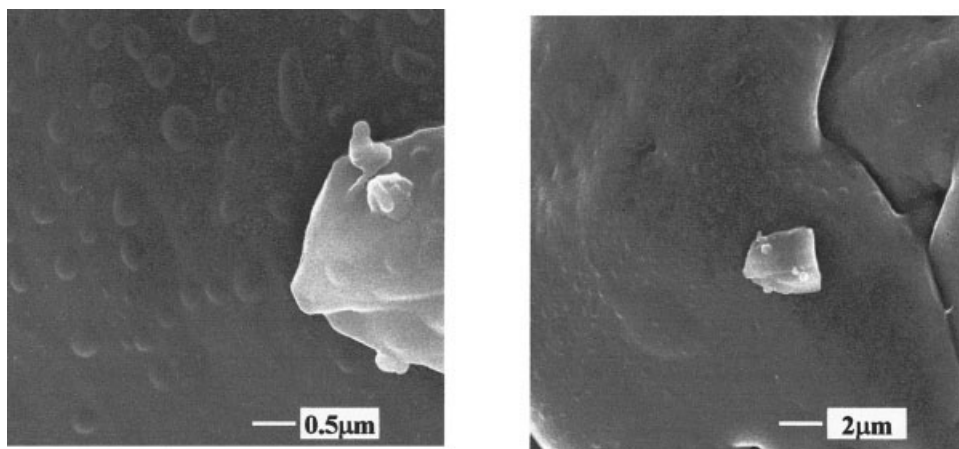
SEM figures for the wax, melamine, and sediment particles reflect their internal structure in the melamine-wax micro-mixing experiments. Figures 3(a) and 3(b) show the wax particles (100–300 nm) in Atebin A1 Micro wax emulsion. Wax particles are observed to occur in a matrix of continuous phase though their size seems to be different. Figures 4(a) and 4(b) show the surface of the melamine particles prior to use in the micro-mixing experiments. The surface of the melamine particles has characteristic “humps,” which are also observed on the surface of the melamine particles in the control experiment (Figs. 5(a) and 5(b)). This shows that although the melamine and wax particles were in contact in the control exper-



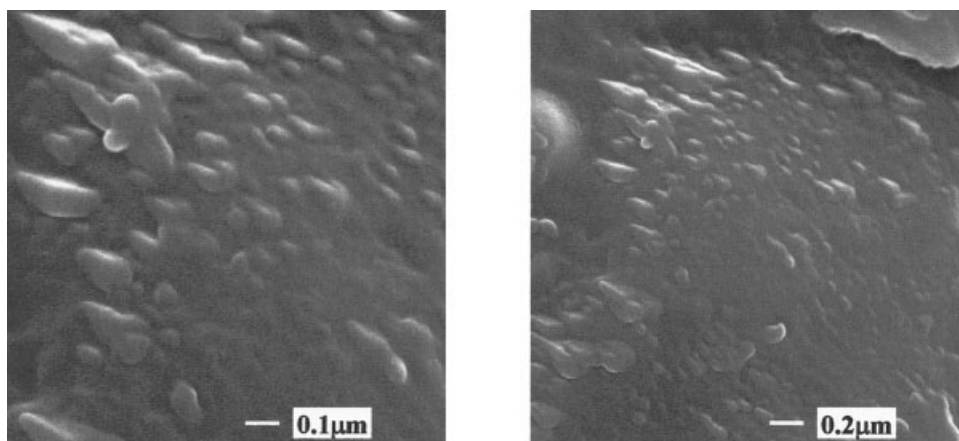
**Figure 3** (a), (b) Wax particles stabilized by alkyl phenol ethoxylate surfactant (Atebin A1 Micro, Böhme Africa).



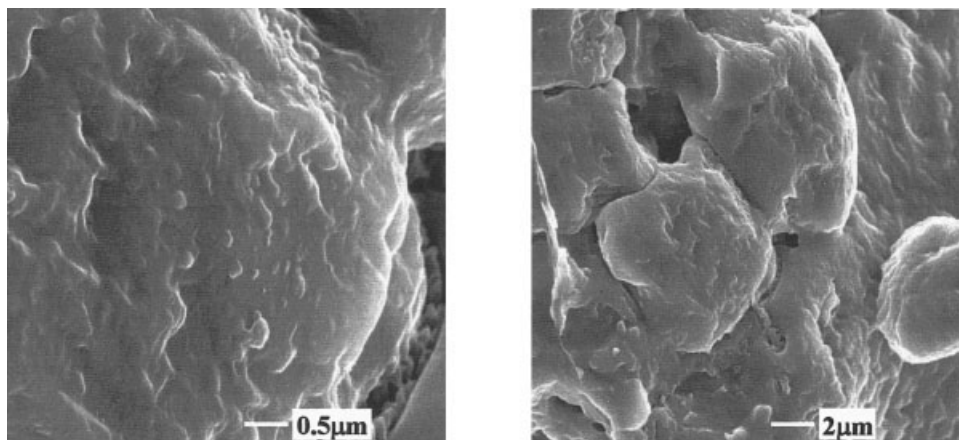
**Figure 4** (a), (b) Melamine particles prior to use in micro-mixing experiments.



**Figure 5** (a), (b) Melamine particle surface of melamine dispersion not stabilized by poly(acrylic acid).



**Figure 6** (a), (b) Surface of melamine particle treated with poly(acrylic acid) and subsequently micro-mixed with wax emulsion. Wax particles on melamine particle surface.



**Figure 7** (a), (b) Surface of melamine particle treated with poly(acrylic acid) and subsequently micro-mixed with wax emulsion. No wax particles on melamine particle surface.

iment, no interaction occurs between them in the absence of the poly (acrylic acid) stabilizer.

Figures 6(a) and 6(b) show the surface of a melamine particle treated with 8% poly(acrylic acid) relative to melamine on a mass basis. The presence of the wax particles on the treated surface indicates that there exists an interaction between the poly(acrylic acid) and the ethoxylate groups of the wax emulsion surfactant (hydrogen bonding) such that the wax particles become attached to the melamine surface and cause particle sedimentation. Figures 7(a) and 7(b) show an area of a treated melamine particle surface with no observable wax particles. However, on comparison of Figures 5(a) and 5(b) with Figures 7(a) and 7(b), the melamine surface shows distinct differences with respect to surface texture. The "humps" observed in Figures 5(a) and 5(b) are not discernable in Figures 7(a) and 7(b). This is possibly attributed to the interaction of free ethoxylated alkyl phenol surfactant

found in the matrix of continuous phase as was observed in Figures 3(a) and 3(b). The interaction of the poly(acrylic acid)-treated melamine with the matrix film results in a change in melamine particle surface texture.

The evidence of surfactant-assisted micro-mixing opens the possibility of using polymeric surfactants such as amphiphilic block copolymers in this novel application, especially in situations that require good particle-particle interactions.

## References

1. Shimabayashi, S.; Uno, T.; Nakagaki, M. *Colloid Surface Physicochem Eng Aspect* 1997, 123–124, 283.
2. Taylor, P.; Liang, W.; Bognolo, G.; Tadros, Th. F. *Colloid Surface* 1991, 61, 147.
3. Kiratzis, N.; Faers, M.; Luckham, PF. *Colloid Surface Physicochem Eng Aspect* 1999, 151, 461.
4. Johansson, B.; Johansson, M. *Nord Pulp Pap Res J* 2000, 15, 243.