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Use of Scanning Electron Microscopy and Molecular/Atomic Spectroscopy to Characterize Beverage Flocculant

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As part of a large food company, a role of our laboratory is to assist in solving customer issues. Problems may arise while products are in the manufacturing process or in many cases from the end product. One cause of customer concern is finding flocculant, haze or sediment in their beverage. Flocculant found in beverages are usually a combination of substances not limited to what is seen on the ingredient label or a foreign material that was introduced by unknown sources. Additionally, ingredients found in the beverage are provided by different manufacturers, including flavor components which are at times proprietary. It is beneficial to use several techniques to provide a clearer and more complete solution to the problem. In this case, chemical, molecular, and morphological information were gathered from the combination of both microscopy and spectroscopy techniques to accurately assess the components of a precipitate.

Advantages of using these complementary techniques was proven in the success of solving a customer sediment issue as they attempted to launch a new vitamin enriched low calorie beverage. A delay in going to market was caused by this issue and a key ingredient provided by our company was suspect. This beverage flocculant had very fine particles and easily dissipated back into solution when disturbed. After several weeks the precipitate aggregated to a manageable size that was carefully separated from the liquid matrix. From the FT-IR spectra, amorphous and crystalline silica and fatty acid ester were discovered as the main components of the flocculant (Figure 1). Although fatty acid ester was a component of the beverage [1], this technique did not fully define the interaction between components that caused this problem. SEM images of the agglomeration reveal the presence of a matrix of circular droplets, submicron spheres and diatoms (Figures 2A and 2B). Elemental analysis through EDS provided further information with the identification of silicon, oxygen, and carbon elements within the matrix. (Figure 3). The diatoms can be associated with crystalline silica while the submicron spheres identify the amorphous silica from FT-IR data. Elemental information of the circular droplets found carbon and oxygen and can be associated to the fatty ester component of the spectra.

In using these complementary techniques we were able to deduce the gradual agglomeration of fatty acid ester with the small silica particles. Closer inspection of ingredients identified a possible source of the silica particles from another ingredient supplier. By applying several technologies to gather morphological, elemental, and chemical information through scanning electron microscope (SEM), x-ray spectroscopy (EDS), and Fourier-transform (FT-IR) respectively, we were able to fully characterize a beverage precipitate. This broad technical approach assisted in solving our customer's problem and enabled a successful product launch.

Reference

[1]"Create extremely hydrophilic polyglycerol fatty acid esters for beverages" The Free Library 01 March 2002. 19 February 2010 <http://www.thefreelibrary.com/Create extremely hydrophilic polyglycerol fatty acid esters for...- a083564934>.

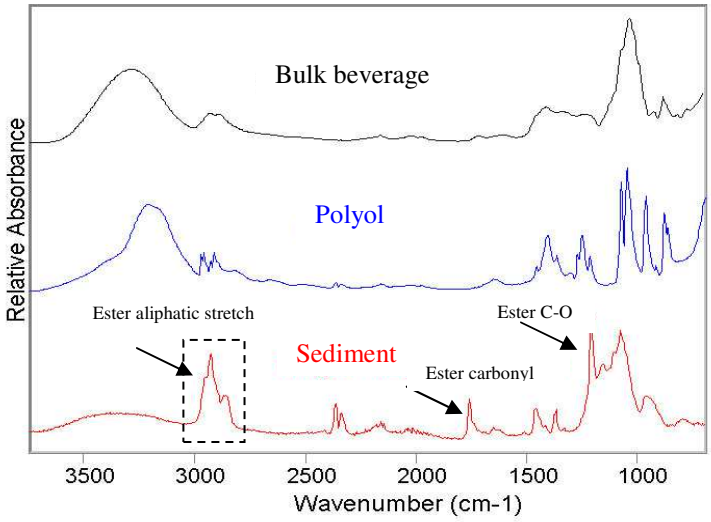

Figure 1. FT-IR spectra comparing the bulk beverage, polyol ingredient and sediment. The presence of several representative peaks within the spectra confirms the presence of an ester fatty acid.

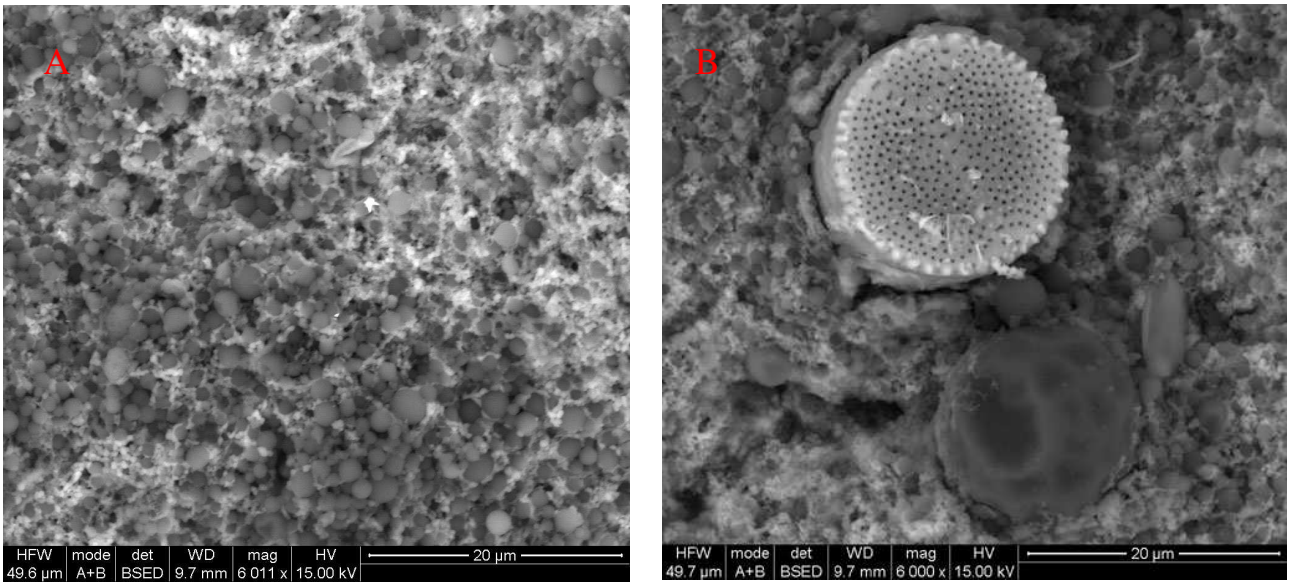

Figure 2A and B. A) Backscattered SEM images of the isolated sediment shows a matrix of circular droplets and submicron spheres. B) The recognizable shape of a diatom is seen embedded within the surrounding matrix of spherical components.

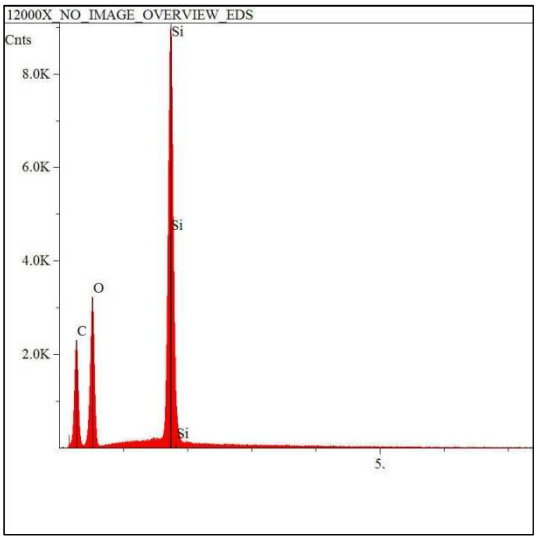

Figure 3. A representative elemental spectrum showing the presence of carbon, oxygen, and silicon within the sediment.

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