FlowCam®

FlowCam for Microencapsulation Process Analysis

OVERVIEW

Microencapsulation is a process by which individual particles can be stored within a shell, surrounded or coated with a continuous film of material to provide protection from the external environment. Beyond providing protection, the encapsulated particles benefit from improved stability, increased shelf life, and the potential for controlled release. A wide range of relevant applications includes cosmetics, fragrances, food and beverage, phamaceuticals, textiles, and more.

Flow Imaging Microscopy (FIM) using FlowCam provides the ability to gain unique insights into the microencapsulation process by dynamically monitoring capsule formation over time, while tracking the effects of temperature, concentration pH, and other variables that can affect the process.

Complex coacervation, whereby microcapsules are formed by combining two hydrocolloids to produce a shell around droplets of an active ingredient (usually in an emulsion), is a common technique which can greatly benefit from FlowCam monitoring.

METHOD

FlowCam was used to monitor coacervate formation in a test vat over a period of time as the sample cooled under constant agitation. The sample was continuously pumped into the FlowCam sample port directly from the reaction vessel. During this process, the view of the flow cell was monitored by eye, while data runs were collected every 15-30 minutes (or if a significant change was observed by eye). The expected size range for the coacervates was in the 80 µm to 140 µm diameter range. Thus the FlowCam instrument was configured with a 4X objective (approximately 50X overall magnification) and a 300 µm-depth flow cell. Measurements were also made during each run to correlate temperature and pH data to the particle images.

Figures 1 through 4 show particle images obtained with the FlowCam during different stages of the experiment. From a visual examination alone, it seems apparent that the run completed at t_0 + 39 minutes shows the cleanest coacervate formation. After this time, the particles began to agglomerate as more gelatin attached itself to the capsules.

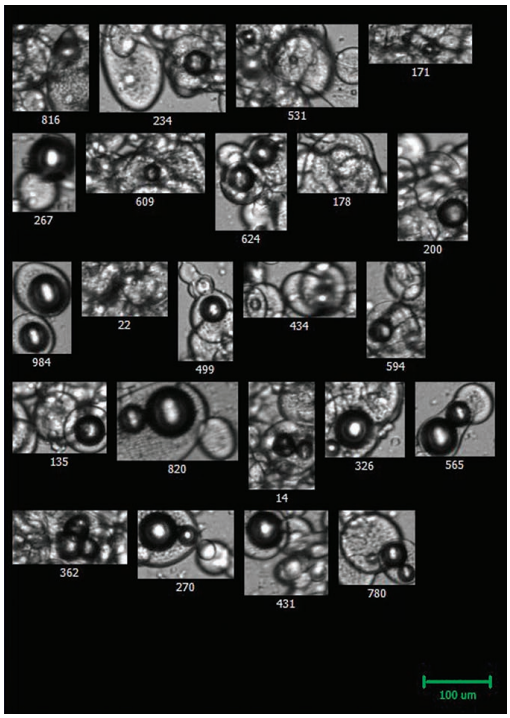

Figure 1. Coacervate Images, T = t_0 + 9 minutes. Active ingredients are the dark circles.

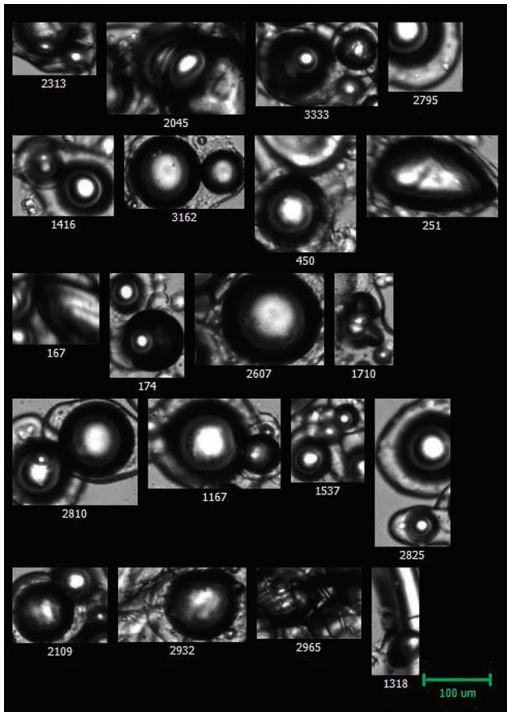

Figure 2. Coacervate Images, T = t_0 + 30 minutes. Gelatin casing begins to form around the active ingredient.

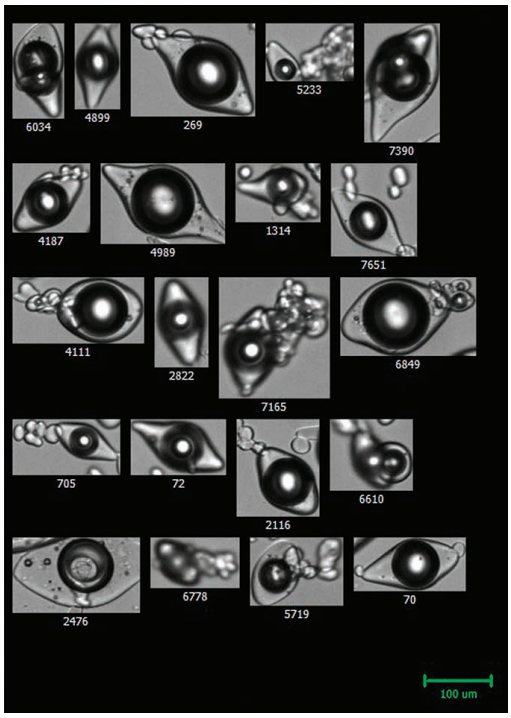

Figure 3. Coacervate Images, T = t_0 + 39 minutes. Coacervates are fully formed.

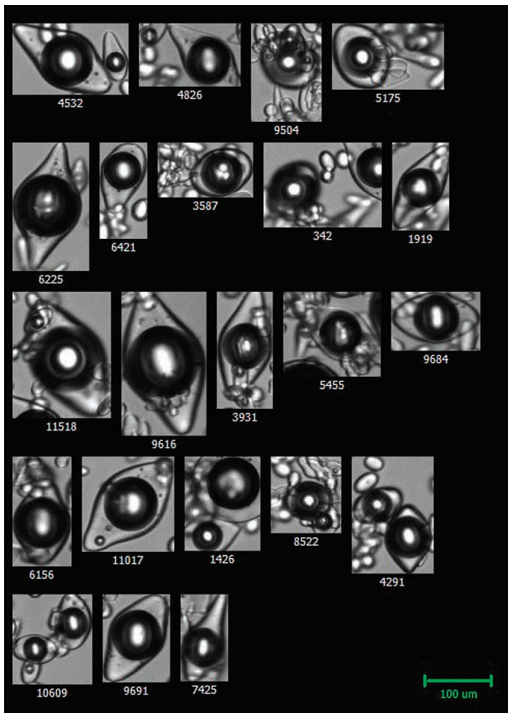

Figure 4. Coacervate Images, T = t_0 + 58 minutes. Coacervates still visible, but agglomeration beginning to occur.

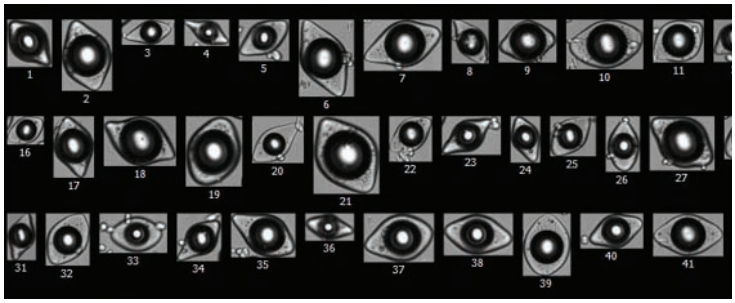

Figure 5. Coacervate images stored in library for statistical pattern recognition

In order to verify the qualitative observations, VisualSpreadsheet® software was used to perform a statistical pattern recognition on each run to quantify the actual number of coacervates. To accomplish this, an image library was created to display all coacervate particles (with specific particle properties) of varying size and orientation. This library is shown in Figure 5.

The results of the statistical pattern recognition operation are shown in a table in Figure 6. These results provide quantitative confirmation of the earlier subjective results: namely that the highest concentration of clean coacervates were formed at t_0 + 39 minutes. After that time point, the gelatin began to attach itself to the capsule walls, causing agglomeration and eventually disintegration of the coacervates.

Time	Number of particles matched to Library	Percent Matched
t_0	0	0%
t_0 + 9 min	0	0%
t_0 + 30 min	0	0%
t_0 + 39 min	1,199	15%
t_0 + 58 min	895	8%
t_0 + 83 min	572	7%

Figure 6. Table showing matching statistics after statistical pattern recognition is run against coacervate library

CONCLUSIONS

FlowCam provides detailed insight into the various stages of coacervate formation and has the potential to be an indispensable tool for microencapsulation applications.

