P-077

Fundamentals of dripping for microencapsulation applications

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INTRODUCTION

Many encapsulation methods consist in extruding drop per drop a liquid in an other liquid. The contact of the droplets with the receipting bath lead to a solidification or membrane formation at the interface of the droplets. The process of dripping is generally analyzed from the observation of the resulting capsules. We propose a more direct observation using a high speed camera.

Encapsulation by dripping has been divided in four steps:
• Droplet formation at end of the needle,
• Fall of the droplets in air,
• Droplet penetration in the liquid,
• Microcapsule formation it-self

In this first study, alginate bead production was considered as simple and fast way to solidify the droplets. The observations will concern the 3 first steps of simple drop per drop method under gravity.

MATERIAL AND METHODS

Alginate bead production

Alginate solution (20 g/L) was dropped in a 20 g/L $CaCl_2$ solution (Panreac, Spain) using a syringe pump and 30 ml syringe connected to tronconique tip (EFD, France). Glycerol (Labogros, France) was added into $CaCl_2$ solution to modify the viscosity and Tween 20 (Sigma Aldrich, USA) was added into $CaCl_2$ solution to modify the surface tension. Beads were let curing for 30 min, filtered and observed under microscope to determine the shape and the size.

High speed video observations

Images of the droplet formation, fall and bath penetration was recorded with a Phantom v7.3 camera (Vision Research, USA) at 400 images per second.

RESULT AND DISCUSSION

Droplet formation at the tip

Figure 1 presents different steps of the droplet formation
• Liquid flows forming a suspended pear shape droplet
• The droplet is maintained on tip through a neck,
• The neck elongates until it breaks,
• Droplet falls and move to a spherical form.

Neck diameter is mainly equal to the external tip diameter (Figure 2) and the size of the droplet is linked to the external tip diameter but also to the liquid flow. The last effect may be due to a longer neck at breaking time for

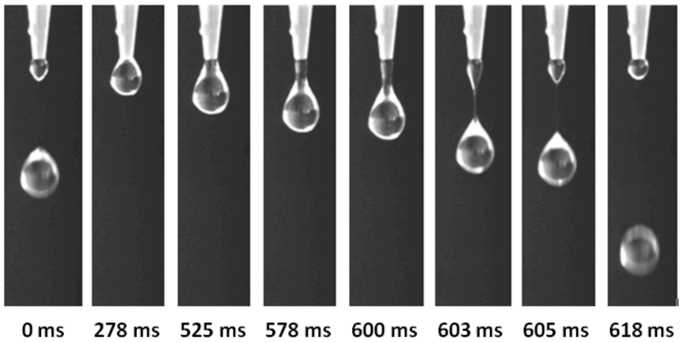

Figure 1. Drop formation sequence

higher flow (data not shown). Part of the neck being incorporated in the droplets.

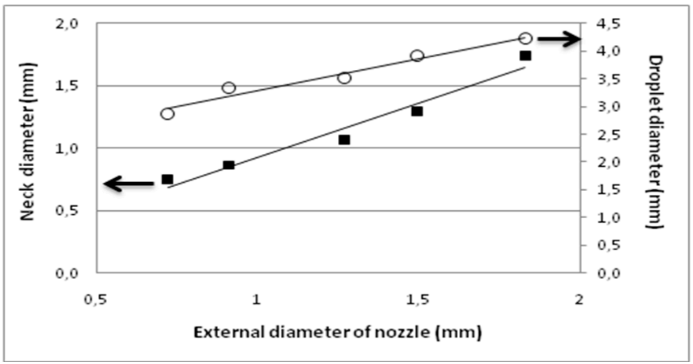

Figure 2. Diameter of neck and droplet with different nozzles

Conditions to get spherical droplets

With help of the video recording, it was also possible to define the distance and period to get spherical droplets (Figure 3 and 4) in function of the nozzle diameter and alginate flow.

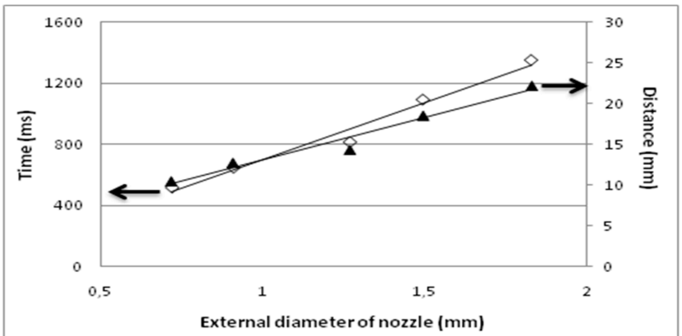

Figure 3. Time and distance to obtain spherical droplet for different nozzles

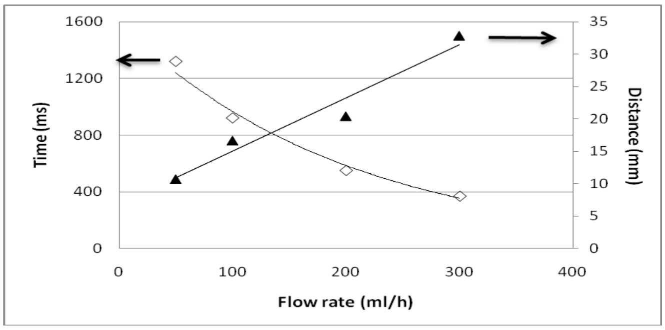

Figure 4. Time and distance to obtain spherical droplet for different flow rates

Even with large tip diameter and high flow the distance to get spherical droplet was limited to 4 cm and the time less than 1 s.

Droplet falling velocity and kinetics energy

By measuring the distance covered by the droplets during a certain time (certain number of frame), it was possible to draw the droplet velocity and the kinetics energy ($E = ½ m v^2$) profiles (Figure 5). Terminal velocity was calculated as 8,4 m/s (Chan et al.,2009). In most laboratory experimental set-up, the droplet would penetrate the solidifying bath at a speed between 25 and 33 % of the terminal.

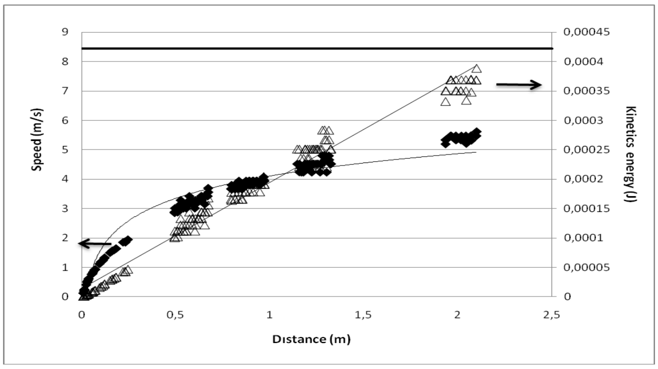

Figure 5. Droplet velocity and kinetics energy (—— line indicates terminal velocity)

Droplet penetration into liquid

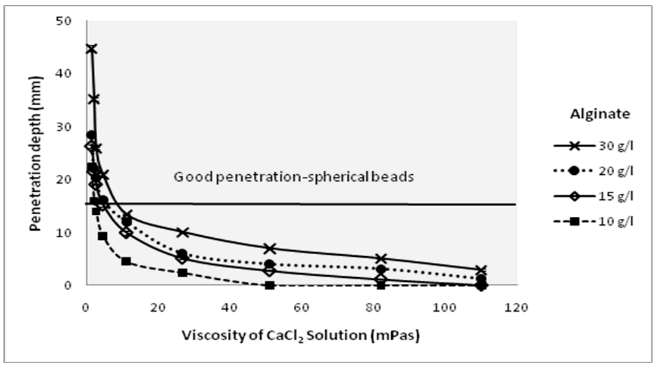

Figure 6. Effect of viscosity on penetration

The droplet (or bead) deforms the surface of the receipting bath until it breaks it allowing full penetration. If their kinetics energy is too low the droplet is rejected. The penetration depth to get incorporation is mainly function of the viscosity and surface tension of the receipting bath (Figures 6 and 7) but generally around 1,5 cm.

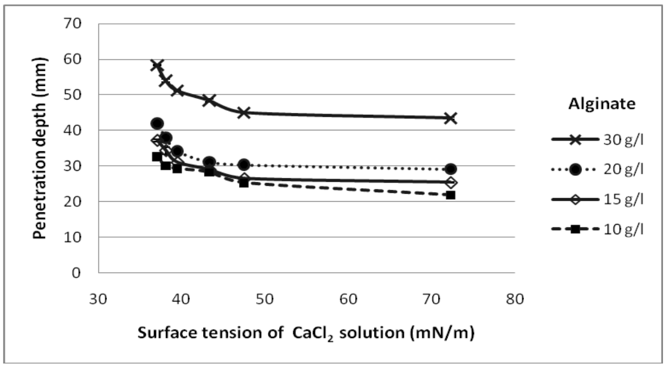

Figure 7. Effect of surface tension on penetration

Even the droplet penetrates correctly the receipting bath, resulting beads may not be spherical. The viscosity of the droplet, and at some extend of the receipting bath determine the sphericity of the beads (Pregent et al.,2009). (Figures 8 and 9)

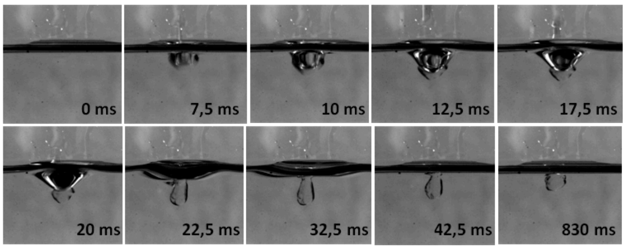

Figure 8. Shape deformation of droplet (μ_{CaCl2}**:110 mPas)**

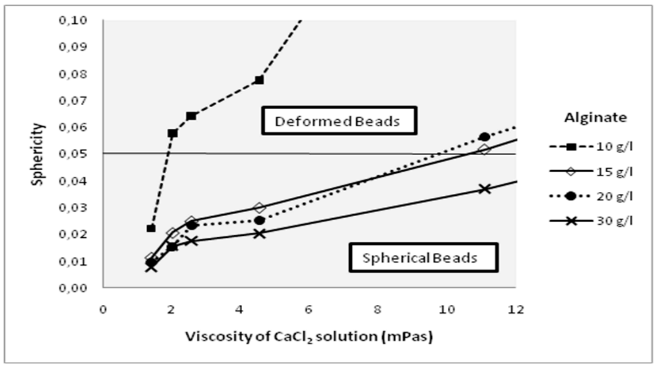

Figure 9. Sphericity factor of beads

CONCLUSIONS

Even preliminary experiments, this data show that using high-speed camera may really help defining optimum conditions to realize a good dripping techniques. Future work plan to analyze more deeply the process and extend the study to more complex process like electrodripping or even co-extrusion with nozzle resonance.

REFERENCES

- Chan, E.-S. et al., 2009. *Prediction models for shape and size of ca-alginate macrobeads produced through extrusion-dripping method*, Journal of Colloid and Interface Science 338 (2009) 63-72.
- Pregent, S. et al., 2009. *The impact and deformation of a viscoelastic drop at the air-liquid interface*, Journal of Colloid and Interface Science 331 (2009) 163-173.