P-074 **Encapsulation of ascorbic acid: impact of loading rates**

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INTRODUCTION AND OBJECTIVES

Recent studies have confirmed that soluble vitamins including ascorbic acid (AA), thiamin, riboflavin, vitamin B6 and folic acid are subject to loss during food processing (Bui & Small, 2008, 2009; Hau Fung Cheung, *et al*., 2009). Collaborative research between the Defence Science and Technology Organization (DSTO) Scottsdale, Tasmania and RMIT University has sought to overcome the problems arising from inherent instability of AA during processing and storage of foods (Wijaya, *et al*., 2011). The results have demonstrated the potential of microencapsulation by spray drying as a means to enhance retention. The purpose of this current study has been focused on preparation and morphology of microcapsules incorporating various loading rates of AA.

MATERIALS AND METHODS

Food grade ingredients used in microencapsulation were AA as core material and a combination of hydrocolloid agents as wall materials. These were maltodextrin (Fieldose 30) and pregelatinised, modified waxy maize starch (Instant MAPS), both supplied by Penford Australia Ltd, Melbourne, Australia. In the trials the rates of loading of the active ingredient incorporated were 6, 9, 18, 36, 54 and 72% of the dry weight of capsular material.

Solution and microcapsule preparation by spray drying

The combination of maltodextrin and modified maize starch were prepared by dissolution in distilled water in the ratio of 8.3:1. The solution was heated to approximately 50°C to ensure hydration. After cooling to 35°C AA was added and stirred until fully dissolved. The pH of each solution was then adjusted to 4.0 using NaOH (2M). A Niro Atomiser (Copenhagen, Denmark) laboratory spray drier with a vane–type rotary atomiser was used for all trials and the conditions were based upon those described by Uddin *et al*. (2001).

Environmental scanning electron microscopy (ESEM) of microcapsules

ESEM was used to observe the outer structure of microcapsules with an FEI Quanta 200 instrument. The settings were: accelerating voltage of 30 kV; pressure of 0.5 Torr; spot size of 4.0; and working distance of 10 mm.

Particle size distribution analysis

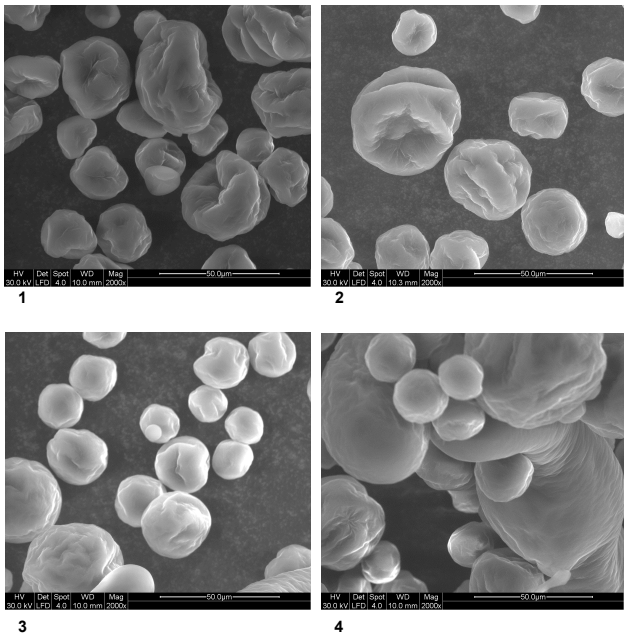
For particle size analysis, small amounts of the microcapsular material were dispersed in *iso*-butanol with continuous stirring. Laser beam scattering (Malvern Mastersizer X, Model MSX025A) was used following the procedure described by Cornell *et al*. (1994). Malvern software was used for data analysis**.**

AA analysis using capillary electrophoresis

AA was analysed by capillary electrophoresis using a procedure based upon that of Thompson *et al*. (1995). An Applied Biosystems instrument (model 270 A-HT) was used with a fused-silica capillary (undeactivated, 75µm internal diameter, Agilent Technologies). The buffer was sodium orthophosphate-sodium tetraborate (0.02 M, pH 8.6) containing sodium deoxycholate. D-Erythorbic acid (D-*iso*-AA) was used as internal standard and the operating conditions were: +15 kV applied voltage, 28°C temperature and 254 nm for detection.

RESULTS AND DISCUSSION

All capsules prepared by spray drying were evaluated by ESEM particularly in order to assess overall shape and the surface appearance as well as the integrity of the capsules and any evidence of damage or breakage. Typical appearance of capsules is shown in Figures 1-4. Those prepared with 18 and 36% AA were similar in appearance to those with 9 and 54% (Figures 2 and 3).



Figures. 1-4. Morphology of spray dried microcapsules obtained using ESEM. Capsules were prepared with AA incorporated at rates of 6% (1, top left) 9% (2, top right) 54% (3, lower left) and 72% (4, lower right).

The images obtained by ESEM demonstrate that the microcapsules are basically spherical in shape. The surfaces appear to be continuous and show indentations to varying extents. The microcapsules retain good structure and integrity for loading rates of 6, 9, 18, 36 and even as high as 54% (Figures 1-3). At a level beyond this, capsules appeared to lack coherence (Figure 4).

Particle size distribution of microcapsules was measured by laser diffraction and the results are summarised in Table 1. The microcapsules prepared by spray drying with different loading rate consist predominantly of particles having diameters in the range of 19 to 234 μm.

Table 1. Microcapsule size of encapsulated AA with varying loading rates

Core loading rate	Diameter of microcapsules (μm)		
	Mean of peak	Mean of 10%	Mean of 90%
6%	51.2	19.0	86.2
9%	47.0	25.0	73.2
18%	36.5	17.5	58.4
36%	46.6	21.0	80.4
54%	90.8	32.7	162.5
72%	130.8	45.2	234.4

Furthermore, the results show that for core loadings in the range of 6-36%, there is no significant effect in terms of diameter. However, when the core loading rate is increased to 54 and 72%, there are increases in size. These confirm the observations from ESEM images showing that higher rates resulted in larger particles having less regular shapes.

The impact of varying loading levels on loss of AA

A further aspect of microencapsulation which has been investigated was the impact of varying levels of incorporation on the loss of AA (Figure 5).

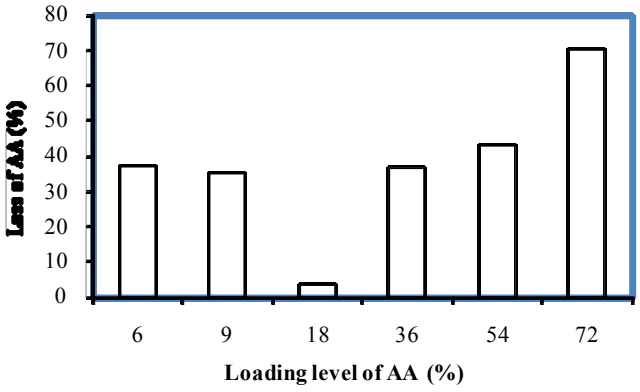


Figure 5. The impact of storage of microcapsules on the retention of AA for capsules with varying levels of incorporation of AA. Experimental conditions involved storage of samples in air-tight containers under dark conditions at ambient temperature (approx 22°C) for six months.

There was a significant impact of loading levels on loss of AA with particularly low retention at the highest level of AA incorporation.

CONCLUSIONS

It can be concluded that microcapsules have good structure and integrity when loading rates were within the range of 6-54%. At the level beyond this, capsules appeared to lack coherence. Loading rates also had a significant effect on loss of AA. Loading rates may be selected to ensure uniformity of distribution within the food being fortified and further studies of retention are recommended.

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