Stability of spray-dried microencapsulated citral

Loksuwan J.
Thammasat university - Pathumthani, Thailand
jarunee@alpha.tu.ac.th

INTRODUCTION

Citral (3,7-dimethyl-2,6-octadienal) is an important flavor component that present in essential oils of several plants such as citronella (lemongrass), lemon myrtle, lime, lemon and orange. Natural citral consists of two isomers, neral and geranial. It is widely used as flavoring or fragrance agents in foods and cosmetics for its strong lemon odor. However, citral is highly susceptible to oxidation and volatilizes rapidly leading to the loss of aroma and formation of off-flavor. These are important problems for the application of citral in food. To overcome these problems, microencapsulation of citral in protective matrix could be employed.

In food industry, microencapsulation technique is widely used to protect food ingredients against deterioration or volatile losses (Shahidi F. 1993). The protective mechanism is to build a specific matrix (wall system) to entrap sensitive ingredients resulting in improved stability and shelf life. Among several microencapsulation techniques available, spray drying is the most commonly used in the food industry due to its low cost and easiness. The wall materials most frequently used are gum, starch or modified starch and proteins. Among these, starch or modified starch has advantages due to its low cost and availability in large quantities (Shahidi F. 1993). Numerous studies have been conducted to evaluate the influence of the composition of wall materials on the stability of spray-dried flavors. (Inglett G.E.1988; Trubiano P.C. 1988; Kim Y. D. 1996; Krishnan S. 2005).

The objective of this study was to investigate the ability of acid modifies starch prepared from rice flour and tapioca starch as wall materials for microencapsulation of citral by spray drying, in comparison with gum arabic and maltodextrin.

MATERIALS AND METHODS

M**aterials :** Native rice flour and tapioca starch was purchased from local market (Thailand). Maltodextrin 18DE and Gum arabic were gifts from Burley Jucker Specialties Ltd.Thailand and Jumbo Trader, Thaland, respectively. Citral was purchased from Sigma Chemical Company, St. Louis, USA. All chemicals were analytical or HPLC grades and were purchased from Merck Darmstadt, Germany.

Preparation of acid modified rice flour or tapioca starch : Acid modified rice flour (R/60C/3.0N)or tapioca starch (T/60C/3.0N)were prepared by acid hydrolysis of native rice flour or native tapioca starch according to the procedure of Loksuwan (2005, 2007). Briefly, starches were suspended in H_2SO_4 in a ratio of 1:5 w/v and the hydrolysis was allowed to proceed for 3 hr at 60°C. The starches were neutralized, filtered and washed with distilled water, followed by ethanol and dried in hot air oven at 70°C for 16 hr. Table 1 shows the selected hydrolysis conditions.

Preparation of microencapsuled citral : For microencapsulation, an emulsion (feed liquid) used for spray-drying was prepared according to the procedure of Loksuwan J (2007) as follows: The

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emulsions were prepared using acid modified starch, maltodextrin, or gum arabic, at 29 % w/w. Acid modified starch was dispersed in distilled water and heated under steam pressure for 5 min to obtain gelatinized starch paste. Maltodextrin or gum arabic was dissolved in distilled water at room temperature. Citral was added to each solution in a ratio of 0.05:1 w/w, on dry starch basis. The mixture was homogenized using an Ultra-turax probe mixer to obtain an emulsion and maintained under slow stirring during spray-drying. The spray-dryer (Armfield FT 30, Armfield Technical Education Co., England) was operated at an inlet temperature of 190$\pm$5°C, blower at 4 and pump at 25.

Retention of citral during homogenization : The ability of wall materials to retain citral during homogenization was conducted. Mixture of wall material and citral was homogenized using an Ultra-turax probe mixer operated at 19600 rpm for 1, 5, and 10 min. The resulting emulsions were then spray-dried. The spray-dried powders were collected and analyzed for total and surface citral using HPLC.

Storage stability of microencapsulated citral : Spray-dried encapsulated citral with acid modified starch, maltodextrin, or gum arabic were stored at -18°C (in freezer), 37°C and 45°C (in controlled temperature oven)
Samples (each about 0.5 g) of the spray-dried powder were transferred to 10-ml glass test tubes closed tightly with screw caps. Changes in total citral during storage were determined by HPLC.

Analysis of total and surface citral of spray-dried powder : Total citral was determined by dissolving 50 mg of spray-dried powder with 2.5 ml distilled water in a screw cap-test tube (2.5x15 cm), followed by extraction with 20 ml hexane using a shaker set at 315 rpm for 30 min at room temperature. The hexane fraction was collected and analyzed for citral.

Surface citral was determined by direct extraction of 50 mg spray-dried powder with 10 ml hexane in a screw cap-test tube (2.5x15 cm) using a shaker set at 65 rpm for 20 sec, followed by centrifugation at 1000 g for 1 min. The clear solution was collected and analyzed for citral.

HPLC analysis : Citral contents were determined according to the method of Rauber, C. et al. (2005) using HPLC with some modification. The extracted sample was injected into a Thermo Separation PC 1000 equipped with a Spectral System UV 1000 detector set at 233 nm, 20 µl sample loop and a Spherisorb® CN column (250 mm x 4.6 mm, 5 um). An isocratic mobile phase of N-hexane: Ethanol (85:15, v/v) was used at a flow rate 0.7 ml/min.

RESULTS AND DISCUSSION

Retention of citral during homogenization
The effect of homogenization time on the total and surface citral of spray-dried powder is shown in Figure 1. Results showed that increasing homogenization time from 1 to 10 min had no significant ($p>0.05$) effect on retention of citral with acid modified rice flour (R/60C/3.0N), acid modified tapioca (T/60C/3.0N) and maltodextin, but had significant ($p\leq 0.05$) effect with gum arabic. The increase in retention with gum arabic as the homogenization time increased is due to its emulsion property. Increasing homogenization time may allow gum arabic to adsorb more to the citral droplet and produce smaller droplet resulting in stable emulsion.

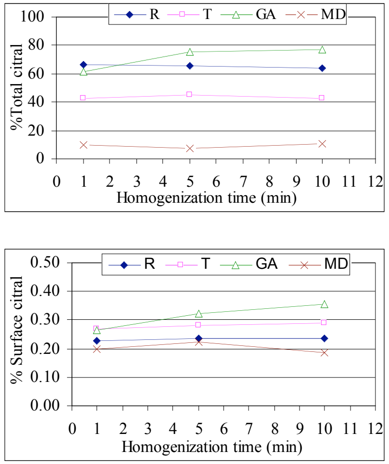

Figure 1: Effect of Homogenization time on total and surface citral of microencapsulated citral with acid modified rice (R) and tapioca (T), gum arabic (GA), and maltodextrin (MD)

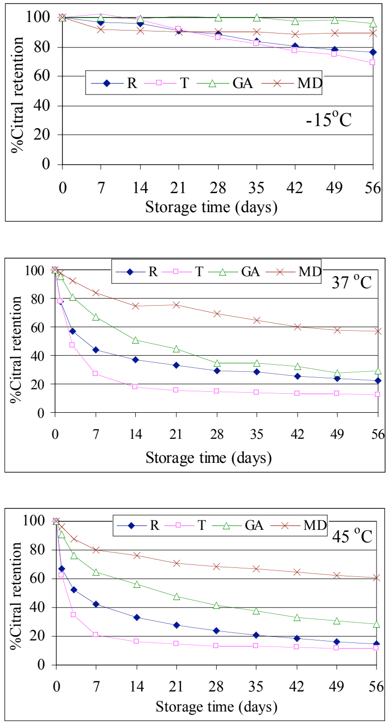

Figure 2: Retention of microencapsulated citral with various wall materials during storage for 56 days at -18, 37, 45°C

The results also showed that retention of citral with R/60C/3.0N immediately after spray-drying was better than T/60C/3.0N and maltodextin. This is probable due to composition of wall materials. R/60C/3.0N contains more protein about 5.5% proteins, while T/60C/3.0N contains only 0.02%, thus R/60C/3.0N may formed more stable emulsion. Maltodextrin gave the lowest retention during spray-drying can be explained by its lacks of emulsifying capacity.

Retention of spray dried citral during storage : Because the concentrations of citral in various wall materials after spray-drying differed due to loss during process, storage stability was expressed as relative to the concentration of citral in each powder after spray-drying (day 0). Figure 2 shows the retention of spray dried citral in various type of wall materials during storage at -18, 37, and

45°C. Results showed that the degradation rate increased with increasing storage temperature. For all samples, degradation rate was initial fast followed by a slower rate. At 37 and 45°C, maltodextrin gave better retention than other wall materials, while at -18°C gum arabic showed a better retention. T/60C/3.0N gave the lowest retention at all temperatures. The better retention with maltodextrin may be due to its ability to form effective barrier.

CONCLUSIONS

Homogenization time had effect on retention of citral in gum arabic but no effect on other wall materials. The retention of citral is strongly influenced by wall material. Maltodextrin gave poor retention of citral during spray-drying, but had the best retention during storage. Acid modified rice flour had better retention of citral than acid modified tapioca.

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