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## Evaluation of the Release of Microcapsulated Vanillin under Simulated Oral Conditions

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### Abstract

In this study, vanillin release from multilayered microcapsules consisting of isolated soy protein, modified starch, and chitosan produced by spray dryer was investigated. Vanillin release was studied using a mouth simulator. The parameters included oral variable such as saliva and the tension on vanillin release. To examine the release of vanillin, the release of these microcapsules (one and two-layers) at 37 °C and pH=6.8, as well as frequent chewing (0, 30 and 55 rpm) were investigated. The results of the release of single (isolated soy protein) and two-layers (modified starch) microcapsules according to the Korsmeyer-Peppas equation showed that the two-layer microcapsules were less diffusion coefficient than the one-layer microcapsules. Mean dissolution time for two-layer and one-layer were calculated 1.24 and 1.08 h at 37 °C, respectively. Therefore, at the same conditions (adding saliva and shear stress), the release rate of vanillin from the two-layer microcapsules was lower than the one-layer microcapsules due to the modified starch coating around the shells and the increase in the thickness of the shell of the two-layered microcapsule, which reduces the release velocity of vanillin from the two-layer microcapsules.

**Keywords:** Modeling, Mouth Simulator, Release, Vanillin

### Introduction

In the microencapsulation method, the solid, liquid or gas components used as the core material are covered by various materials. Usually, the wall can be designed in such a way that the microcapsules release their contents at a controlled rate and under certain conditions (Jafari, Assadpoor, He, & Bhandari, 2008). Controlled release is a method in which one or more components are available at a specified time and location at the desired speed. Using

this technology, it is possible to use some temperature and pH sensitive additives, such as flavoring compounds in food systems (Pothakamury & Barbosa-Cánovas, 1995). For determining the mass transfer properties of the microcapsules, release predicting, and fitting of experimental data, we need to produce the valid mathematical models (Zuidam & Nedovic, 2010).

Flavor release during eating is largely dependent on oral parameters. These parameters include the temperature of the mouth, the composition and amount of saliva, the frictional forces, the amount of mixing, pH, and many other factors; therefore, a simple analysis of the headspace analysis (for aromatic compounds) or extraction fluid (for taste compounds) is simple. In which the conditions and dynamic phenomena in the mouth are not considered. For this reason, construction and use of a simulator palate and mouth can make the results available closer to reality (Salles *et al.*, 2007).

Therefore, the overall aim of this study was to evaluate the release of vanillin encapsulated by soybean protein isolate, modified starch and chitosan using spray drying method, physicochemical properties, and oral conditions in order to measure the effects of oral parameters such as saliva and the stress range imposed on its release rate.

## Material and methods

### Solution preparation

The stock solution of SPI (0-3%) was prepared by methods of Huang, Sun, Xiao, & Yang (2012). As for the OSA starch stock solution (0-3%) was prepared by methods of Nilsson & Bergenstahl (2007). As for the chitosan solution (0-1.5%) was prepared using the methods of Chuah, Kuroiwa, Kobayashi, & Nakajima (2009).

### Emulsions preparation

The primary emulsion of the optimum SPI, secondary emulsion of optimum OSA starch and the tertiary emulsion of optimum chitosan concentration was prepared by the method of Noshad, Mohebbi, Shahidi, & Koocheki (2015).

### Microcapsule production

to prepare the Microcapsules, a semi-industrial spray dryer was used. 184 °C, drying temperature, concentration of 8.5% (w/w) maltodextrin and 0.36% (w/w) vanillin were used, and two- and three-layer microcapsules were also prepared in the same conditions (Noshad *et al.*, 2015).

The moisture content, Encapsulation efficiency, Solubility, Hygroscopicity, and Release of vanillin were evaluated and finally by first order, Hixson and Crowell, Higuchi, and Korsmeyer-peppas models were used to the fitting of experimental data.

### Vanillin release

To investigate the release of the encapsulated vanillin, the release of these microcapsules (one and double) at 37 °C and pH=6.8, as well as frequent chewing (0, 30 and 55 rpm) were examined. For the apply of temperature and shear stress, an oral simulator was designed and developed by the Department of Food Science and Technology of Ferdowsi University of Mashhad (Fig. 1).



**Fig. 1.** Oral simulator

### Results and discussion

Table (1) shows the values obtained for moisture content, solubility, hygroscopicity and encapsulation efficiency of the powders. The moisture content of the microcapsules varied from 2.32 to 3.64% being within the range expected for spray-dried products (Rocha-Selmi, Bozza, Thomazini, Bolini, & Fávaro-Trindade, 2013). The addition of OSA starch to primary emulsion had no significant effect on the moisture content of microcapsules while incorporation of chitosan to secondary emulsion and formation of tertiary emulsion leads to the reduction of moisture content in the microcapsules. The solubility parameters, there was no significant difference between values obtained for microcapsules. The hygroscopicity of the microcapsules varied over the range of 8.7-11.3 g water absorbed/100 g sample for three formulations of study microcapsules, with no significant differences between. For the encapsulation efficiency, there were significant differences between three-layer microcapsules with two and one-layer microcapsules.

**Table 1.** Measurement of moisture content, hygroscopicity, solubility and encapsulation efficiency of the various microcapsule formulations

Treatments	Moisture content (%)	Solubility (%)	Hygroscopicity (g/100 g of powder)	Encapsulation efficiency (%)
One layer	3.61±0.04 <sup>a</sup>	11.42±2.55 <sup>a</sup>	11.30±0.02 <sup>a</sup>	51.91±2.53 <sup>a</sup>
Two layer	3.09±0.06 <sup>a</sup>	12.28±0.31 <sup>a</sup>	8.70±0.03 <sup>a</sup>	44.59±3.33 <sup>a</sup>
Three layer	2.32±0.30 <sup>b</sup>	10.16±0.69 <sup>a</sup>	10.20±0.02 <sup>a</sup>	27.59±2.67 <sup>b</sup>

There were no significant differences among the samples with the same letters in the same column ( $P < 0.05$ ).

To find out the mechanism of vanillin release, first 60% vanillin release data was fitted in Korsmeyer-Peppas model Eq. (1), (Arifin, Lee, & Wang, 2006).

$$\frac{Q_t}{Q_e} = Kt^n \quad (1)$$

Where  $Q_t/Q_e$  is the fraction of the drug released at time  $t$ ,  $K$  is a constant corresponding to the structural and geometric characteristics of the device and  $n$  is the release exponent that is indicative of the mechanism of the vanillin release. The mean dissolution time (MDT) used for comparison of release profiles of different formulations because it shows the vanillin release-retarding efficacy of the polymers used in a formulation (Balcerzak & Mucha, 2010; Dash, Narasimha Murthy, Nath, & Chowdhury, 2010). A higher value of MDT indicates a higher flavor retarding ability of the polymer and vice versa. Mean Dissolution Time (MDT) was calculated from the following Eq. (2), the value of  $K$  and  $n$  were calculated according to Korsmeyer-Peppas equation (Arifin *et al.*, 2006; Balcerzak & Mucha, 2010).

$$MDT = \left[ \frac{n}{(n+1)} \right] \cdot K^{-1/n} \tag{2}$$

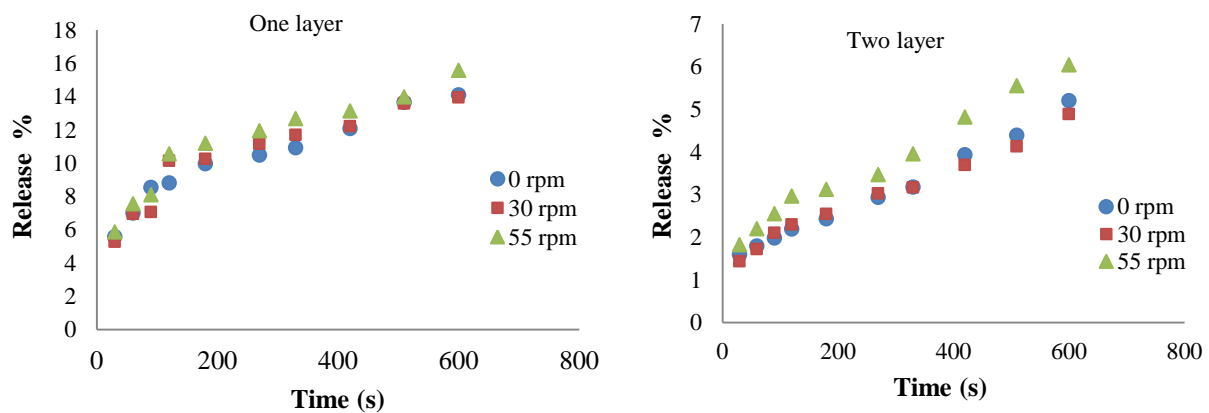
The Korsmeyer-Peppas model best explained the vanillin release as its value of R<sup>2</sup> was greater than 0.86 for all formulations. The value of “n” from 0.24 to 0.4 indicated that vanillin release mechanism from all microcapsules was diffusion controlled (Balcerzak & Mucha, 2010; Dash *et al.*, 2010). MDT values of one-layer, two-layer, and three-layer microcapsules were found to be 1.08, 1.24 and 1.78 h at 37 °C, which showed that three-layer microcapsules had more release retarding efficacy (Table 2).

It could be seen that an increase in temperature leads to reductions in the MDT of one-layer and two-layer microcapsules, resulting in a facilitating volatiles release. In addition, analyzing the two tested temperatures showed the three-layer microcapsules were relatively resistant to high temperature (80 °C) and increasing the temperature did not lead to decrease in the MDT of the three-layer microcapsule (Table 2).

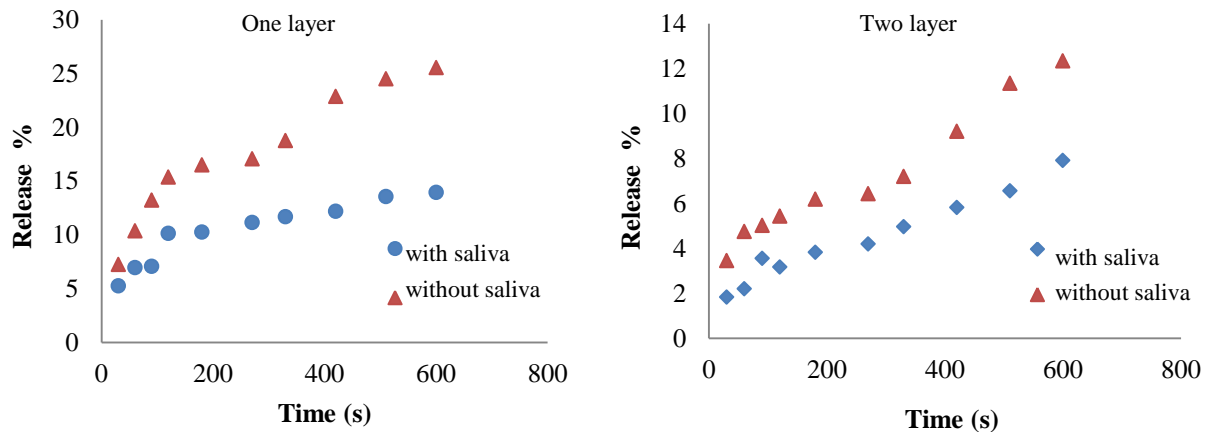
**Table 2.** Kinetic parameters of microcapsules according to Korsmeyer-Peppas model

Temperature (°C)	One-layer			Two-layer			Three-layer		
	n	MDT (h)	R <sup>2</sup>	n	MDT (h)	R <sup>2</sup>	n	MDT (h)	R <sup>2</sup>
37	0.24	1.08	0.88	0.37	1.24	0.86	0.25	1.78	0.93
80	0.32	0.86	0.96	0.40	0.98	0.97	0.26	1.77	0.96

Fig. (2) Shows the vanillin release profile of one and two-layer capsules as a function of shear stress. The results show that applying shear stress can increase its release rate. It seems that the shear stresses, like a compressive force, increase the output of vanillin from the pores of the microsphere. In addition, during some shear stresses, some of the microcapsules are degraded, resulting in an increase in the release of vanillin into the environment. It seems that the shear stresses, like a compressive force, increase the output of vanillin from the pores of the microcapsules. In addition, some of the microcapsules are degraded, resulting in an increase in the release of vanillin into the environment. As shown in Fig. (3), adding saliva can increase the release rate of vanillin. This is probably due to the change in the ionic strength of the environment, which causes the instability of the microcapsules. Similar results were observed by (Ansarifar, Mohebbi, Shahidi, Koocheki, & Ramezani, 2017) about the use of various saliva ratios for the release of limonene encapsulated with soybean and pectin isolate protein fibril layers.



**Fig. 2.** Effect of shear stress on vanillin releasing profile of one and two layer microcapsules



**Fig. 3.** Vanillin release profile of one and two layer capsules in the presence and absence of saliva in shear stress 30 rpm

### Conclusion

According to the proposed objectives and the obtained results, this study showed that the use of multilayer technique prior to spray drying of vanillin microcapsules made it possible to protect it and control its release. All microcapsules were not very water-soluble or hygroscopic while three-layer microcapsules compared to one and two layer microcapsules had lower moisture content and predominantly shriveled surfaces. In addition, although three-layer microcapsules compared to one and two-layer microcapsules had a less encapsulation efficiency, but three-layer microcapsules had more release-retarding efficacy.

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