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FULL PAPER

Characterization of freeze-dried ginger slices with carboxymethyl cellulose as potential coating material prior to osmotic dehydration

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Abstract

Edible coating prior to osmotic dehydration has been studied extensively to enhance the performance of drying in food products. In this study, the effectiveness of carboxymethyl cellulose (CMC) coating before osmotic dehydration of ginger slices on the mass transfer was studied. The samples were then dried using a freeze dryer, and the changes in colour, structural, and chemical components were determined. Coated ginger showed a minimised solute gain without affecting the water loss performance. The mass transfer kinetics were fitted successfully using the Magee model. After the freeze-drying process, the combination of CMC and osmotic dehydration as a pre-treatment influenced the colour properties and the microstructure without altering the functional groups. From these findings, it shows that CMC coated ginger slices and osmotic dehydration prior to freeze-drying can be an alternative to meet the demands of current consumers, and thus be a promising route for obtaining dried ginger.

1. Introduction

Ginger (*Zingiber officinale*) has been grown widely in many tropical and subtropical countries and is often used not only for culinary purposes but also valued for its medical properties. Total world ginger production in 2019 is 4,081,374 tonnes and India, Nigeria, China and Nepal are the major producers in the world (FAOSTAT, 2021). The growing demand for ginger was expected to increase especially in major importing countries, the United States and Europe (Nair, 2019).

The moisture content of ginger ranged from 85 to 95%, which is prone to rapid deterioration (Osae *et al.*, 2019; Muhamaruesa *et al.*, 2020). Thus, dehydration is one of the essential methods in food preservation, which reduces water activity and thus limits bacterial growth and extends their availability on the market. Nowadays, there are various dehydration methods spurred for the development of dried products since the requirement of each product varies. Commonly, the selection was based on the material to be dried, the cost and also the operating conditions that affect the final quality of the dried product (Sabarez, 2016).

Over the past decades, there has been sustained research activity recorded in the osmotic dehydration process for various food materials. Osmotic dehydration is often used as a pre-treatment step prior to drying as the process offers partial water removal by immersion of food material in the hypertonic solution for a definite period of time. During the process, there are also solute inflows from solution to product concurrently, which is one known challenge in this dehydration method. Excessive solute uptake during the osmotic process can impede the surface layer of the product and this additional resistance can lower the water diffusion rate during the finish drying process (Phisut, 2012). On top of that, increasing consumer awareness of the health effects of sugar leads to increased demand for low sugar-product (Wilson, 2016). Thus, the approaches of the edible coating prior to osmotic dehydration as a potential method for lowering the solute gain without affecting the performance of water removal have been studied extensively.

The coating serves as an extra barrier to mass transfer during the osmotic dehydration process. Various edible coatings are available nowadays and lots of them were prepared from natural products such as polysaccharides, proteins, lipids and their derivatives

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(Dhall, 2013). The edible coatings possess some characteristics features such as good mechanical strength, desirable sensory properties, the ability to quickly form film, non-toxic, high-water diffusivity and also high stability (Shit and Shah, 2014; Osman and Barazi, 2018; Batista *et al.*, 2020; Chhikara and Kumar, 2021; Panahirad *et al.*, 2021). Carboxymethyl cellulose (CMC) is one of the polysaccharide edible coatings that had been implemented in the osmotic dehydration process as a pre-treatment before the drying process to improve the quality of the final products.

Lyophilization, also known as freeze-drying has been widely used as a finish drying method, in which the process involved removing water by freezing the product and sublimating the ice to vapour. This method allows the retaining of food quality and structural properties such as high porosity, good rehydration, superior taste and aroma and nutritional retention compared to another drying system (Rajkumar *et al.*, 2017; Xu *et al.*, 2019). In recent years there has been an increased interest in exploring the combination of pre-treatment methods prior to freeze-drying with aims to enhance the shelf life and quality of the food products (Mothibe *et al.*, 2014; Prosapio and Norton, 2017; Alipoorfard, Jouki and Tavakolipour, 2020).

Thus, the key idea in this paper is to study the influence of CMC coating on the mass transfer during osmotic dehydration and evaluate the changes in colour, microstructure and chemical components of the freeze-dried ginger slices.

2. Materials and methods

2.1 Sample preparation

The fresh ginger (*Zingiber officinale*) was purchased from a local market in Kuala Nerus, Terengganu, Malaysia. The sample was then washed and sliced into a uniform shape with a diameter of 15 mm and a thickness of 4 mm. The initial moisture content of ginger slices was determined by drying in a hot air oven (Memmert, Beschickung loading, model 100–800, Germany) at 105°C for until constant weight is reached (AOAC, 2000). Commercial sucrose was used for preparing the osmotic solution with a concentration of 50% (w/w).

2.2 Coating and osmotic dehydration process

Coating solution at a concentration of 1% (w/w) of CMC (Acros Organics, Geel, Belgium) was prepared using the method described by Rahimi *et al.* (2013). CMC-coated ginger slices were prepared by dipping the samples in a CMC solution at room temperature and then dried in a drier at 70°C for 10 mins. The coated and non-coated samples were then immersed in the sucrose solution with a ratio of 1:10. In each experiment, a fresh sucrose solution was used. After 120 mins of immersion time, the sample was removed from the osmotic solution and rinsed immediately in flowing water, drained on tissue paper to remove surface moisture.

2.3 Freeze-drying

For finish drying, the samples were dried in a freeze dryer (Labconco, FreeZone, Kansas City, MO, USA) for 24 hrs. Prior to drying, all samples were frozen in a freezer at -18°C of temperature for 24 hrs. All the samples were kept in a desiccator until further analysis.

2.4 Water loss and solute gain determination

The terminology used to evaluate the mass exchange of sample and solution between ginger slices and sucrose solution during osmotic dehydration are the water loss (WL) and solute gain (SG). The WL and SG were calculated using Equation (1) and (2), respectively.

$$WL = \frac{w_{wo}-(w_t-w_{st})}{w_{wo}+w_{so}} \times 100 \quad (1)$$

$$SG = \frac{(w_{st}-w_{so})}{w_{wo}+w_{so}} \times 100 \quad (2)$$

Where w_{wo} is the mass of water in the sample before dehydration (g), w_t is the mass of the sample after dehydration (g), w_{so} is the mass of the solids the in sample before dehydration (g) and w_{st} is the mass of the solids in the sample after dehydration (g).

The kinetics during osmotic dehydration was modelled using the Magee model as shown in Equation 3. This model has been reported as an adequate model for predicting the mass transfer kinetics of various food products such as plum (Ibitwar *et al.*, 2008), beetroot (Kaur and Singh, 2013) and pomegranate (Allahdad *et al.*, 2019).

$$WL \text{ or } SG = A + k\sqrt{t} \quad (3)$$

Where A and k are constants and t is time (mins).

The coefficient of determination (R^2), chi-square (χ^2) and root mean square error ($RMSE$) as shown in Equation (4), (5) and (6), respectively were determined to evaluate the fitness quality of the model (Md Salim *et al.*, 2016).

$$R^2 = 1 - \frac{\sum_{i-1}^{i=n}(X_i^{exp}-X_i^{pred})^2}{\sum_{i=n}^{i=n}(X_i^{pred}-\bar{X}^{exp})^2} \quad (4)$$

$$\chi = \sum_{i-1}^{i=n}\frac{(X_i^{pred}-X_i^{exp})^2}{n-m} \quad (5)$$

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(X_i^{pred}-X_i^{exp})^2} \quad (6)$$

Where n is the number of observations, X_i^{pred} is the ith predicted value of WL or SG, X_i^{exp} is the ith experimental value, $\bar{X}^{exp}$

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value of WL or SG, m is the number of variables and is the mean of the experimental value of WL or SG.

2.5 Colour measurement

The colour of fresh and freeze-dried samples under different conditions was measured using CR-400 Chroma Meter (Konica Minolta Co. Ltd., Japan). The measured parameters are L^* (lightness), α^* (green–red coordinate) and b^* (blue–yellow coordinate). The total colour changes were calculated using the Hunter-Scotfield equation (Equation 7); where $\Delta\alpha = \alpha^* - \alpha^*_0$; $\Delta b = b^* - b^*_0$ and $\Delta L = L^* - L^*_0$, subscript 0 represents the fresh sample.

$$\Delta E = \sqrt{(\Delta\alpha)^2 + (\Delta b)^2 + (\Delta L)^2} \quad (7)$$

2.6 Microstructure characterization

Changes in the microstructure of freeze-dried ginger under different conditions were observed using scanning electron microscope (SEM) (JEOL, JSM 6360LA, Japan). The samples were cut along its cross-sectional axis and then coated with gold particles using gold coater to form a thin layer thickness, which enhanced the conductivity of electrons into the sample material during the SEM observation (Zulkifli *et al.*, 2021). The samples were photographed at 300 magnifications.

2.7 Fourier transform infrared spectroscopy

The Fourier transform infrared spectroscopy (FTIR) spectra of freeze-dried ginger under different conditions were obtained using a Thermo Nicolet 380 spectrometer (Thermo-Scientific, USA). The data were recorded at room temperature in the wavelength range of 4000 – 400 cm^{-1} (Kamarudin and Isa, 2013).

2.8 Statistical analysis

All experiments were conducted in triplicate and the means were analyzed using analysis of variance (ANOVA) to establish mean differences between treatments. Duncan's multiple range test was used to compare the difference between means at a probability level of $p < 0.05$.

3. Results and discussion

3.1 Mass transfer

The kinetics of WL and SG of the coated and uncoated sample of osmotic dehydration of ginger slices were shown in Figure 1. Interestingly, coating ginger can minimize the SG while maintaining the WL performance compared to uncoated ginger. During the immersion process, osmotic pressure from the sucrose solution is the main driving force for the mass transfer to occur. Thus, once coating material is applied to the sample, the barriers have been formed, which has led to a decrease in the penetration of the solute into the sample. These findings are in line with previous studies on pumpkin (Jansrimanee and Lertworasirikul, 2017) and apple (Jalaee *et al.*, 2011). The fitted semi-empirical Magee model with the experimental data is shown in Figure 1 and the statistical analysis for this kinetics fitting is presented in Table 1. From the analysis, the model is well-fitted for both conditions.

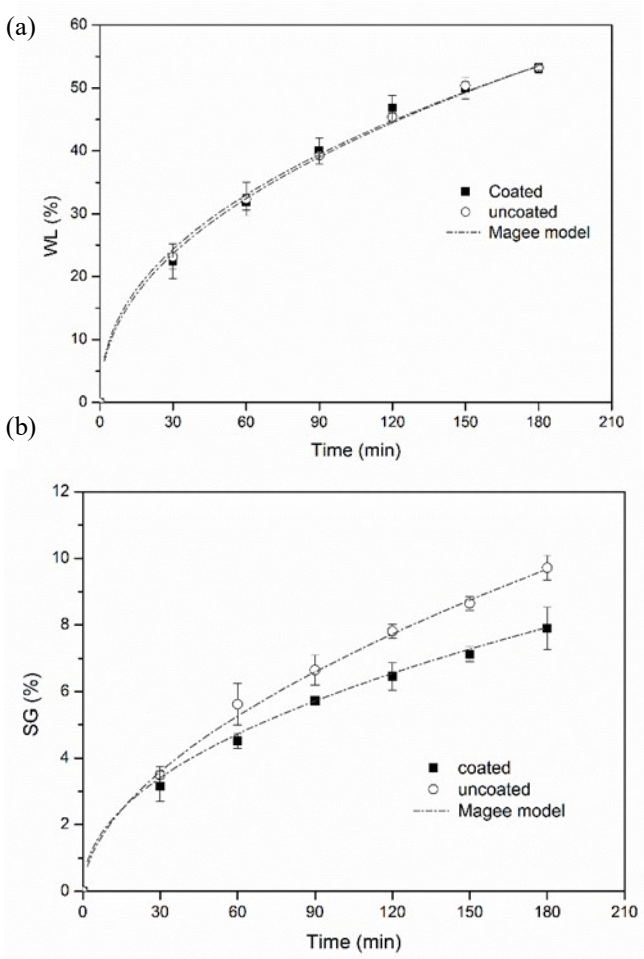

Figure 1. Kinetics of osmotic dehydration of uncoated and coated ginger slices on (a) Water loss (WL) and (b) Solute gain (SG).

3.2 Colour measurement

Colour is a crucial factor in the quality assessment of

Table 1. Value of Magee model parameter for WL and SG of uncoated and CMC coated osmotically dehydrated ginger slices.

Condition	WL (%)					SG (%)				
	A	*k*	χ^2	*RMSE*	R^2	*A*	*k*	χ^2	*RMSE*	R^2
Uncoated	5.0073	0.4563	0.6037	0.7770	0.99	0.5451	0.5539	0.2236	0.4728	0.99
Coated	5.4692	0.4392	0.9685	0.9841	0.99	0.6840	0.4718	0.4183	0.6467	0.99

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dehydrated products (Lyu *et al.*, 2015; Md Salim, Gariépy and Raghavan, 2017; Wang *et al.*, 2019). Fresh ginger has a light yellow in colour, and its colour density was depending on the cultivar, growing area and maturation stage (Iijima and Joh, 2014). In this study, the values for the L^*, α^* and b^* coordinates of the fresh ginger slices were 76.59, −3.59 and 26.31, respectively. The freeze-drying process with different pre-treatment conditions on ginger slices results in some changes in colour parameters as shown in Table 2. It was noted that lyophilization process prevents enzymatic browning, resulting in increased lightening of the sample surface. This has also been observed in kale (Dziki *et al.*, 2018), plum (Muñoz-López *et al.*, 2018) and other fruits such as bananas, apples and kiwis (Valentina *et al.*, 2016). Meanwhile, the infusion of sucrose results in less change in colour compared to the use of CMC edible coating and untreated freeze-dried sample. This also accords with previous studies (Chauhan *et al.*, 2011; Bozkir *et al.*, 2019), which showed that osmotic dehydration as pre-treatment inhibits enzymatic and oxidative browning, allowing for high colour retention. The clear film formed when CMC is used as a coating prior to osmotic dehydration makes the sample lighter than the osmotic dehydration sample. Ghanbarzadeh, Almasi and Entezami (2010) reported that the L^* values of the film were significantly increased by the addition of the CMC.

3.3 Microstructure

The microstructure changes of freeze-dried ginger under different conditions are shown in Figure 2. It is clearly seen that ginger starch granules are spherical to ellipsoidal in shape, as previously reported (Moreschi *et al.*, 2006; Kuk *et al.*, 2017). For freeze-dried ginger as shown in Figure 2(a), the cell wall has a honeycomb-like structure in which small starch granules are embedded. While, in Figure 2(b), ginger treated with osmotic dehydration pre-treatment results in cell wall disarrangement as the result of osmotic pressure imposed by the sucrose solution during the immersion process. At the same time, the starch granules still remained intact but with swell. According to a previous study (Hedayati *et al.*, 2016), penetration of sucrose into the starch granules can results in higher water absorption and thus expanding the granule size. Starch swelling was also observed as CMC coating was applied prior to the osmotic dehydration process and the cell wall was severely preserved, as shown in Figure 2(c). The coating helped to reduce the amount of solute uptake within the cell wall, which was in accord with the decrease in SG. This observation has also been reported previously on pumpkins (Jansrimanee and Lertworasirikul, 2017).

3.4 FTIR

In order to confirm the effect of CMC coating and osmotic dehydration on the chemical components of ginger, Figure 3 depicts a comparison of the FTIR spectra of freeze-dried ginger slices. From the figure, it shows that similar functional groups have been observed for all conditions as no new peaks have been formed. Detailed peak and band assignment of freeze-dried ginger samples match those observed by previous studies on ginger (Zhao *et al.*, 2015; Hussein *et al.*, 2017). The broad bands at 3323 cm^{-1} confirm the involvement of O–H stretching of hydrogen-bonded hydroxyl groups. Meanwhile, the band of 2895 cm^{-1} was due to the asymmetric sketching vibration of methyl groups (C–H) which represents the aliphatic compound in the ginger. At spectral band detected at 1635 and 1388 cm^{-1} corresponded to the amide I was attributed to coupled

Table 2. Colour parameter of fresh and freeze-dried ginger with different pre-treatment conditions.

Condition	L^*	α^*	b^*	ΔE
Fresh	76.59±1.52^{c}	-3.59±0.09^{c}	26.31±0.11^{a}	-
Freeze-dried	89.48±0.42^{a}	-4.27±0.11^{b}	26.53±0.70^{a}	12.91±0.43^{a}
Osmo-freeze-dried	86.16±0.03^{b}	-2.03±0.06^{a}	24.95±0.16^{b}	9.80±0.06^{c}
CMC coated osmo-freeze-dried	87.44±0.04^{a}	-2.61±0.12^{a}	23.33±0.23^{c}	11.30±0.11^{b}

Values are presented as mean±SD. Values with different superscripts within the same column are statistically significantly different ($p<0.05$).

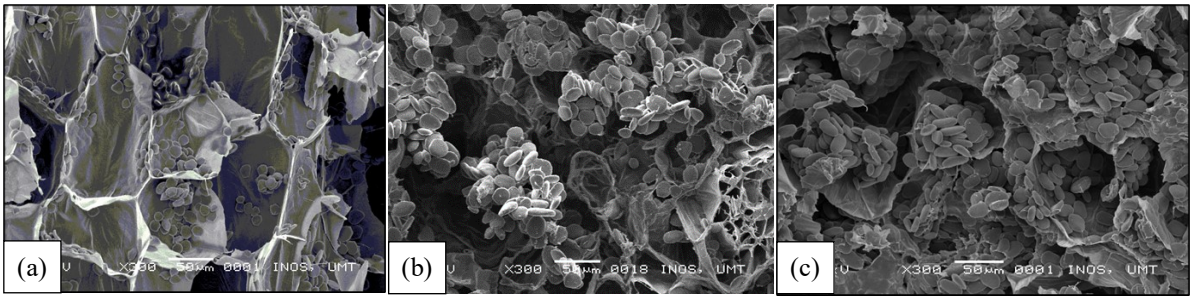

Figure 2. The scanning electron microscope (SEM) micrographs for (a) freeze-dried ginger, (b) osmo-freeze-dried ginger, and (c) CMC coated osmo-freeze-dried ginger.

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C=O stretching and aromatic skeletal combined with C-H in-plane deforming and stretching, respectively. The strong band at 1197 and 1022 cm^{-1} observed in the spectra were assigned mainly to C–O–C stretching and the stretching of C-H and C-C. The weak band at 511 cm^{-1} might result from pyrrole fold motions. Pretreated samples exhibited slight band shifting when compared to freeze-dried, indicating that the presence of CMC and sucrose had some effect on the intensity of the

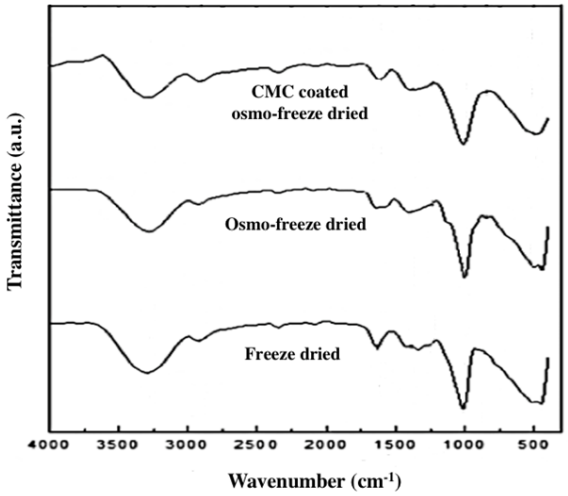

Figure 3. The FTIR spectrum of freeze-dried ginger, osmo-freeze-dried ginger and CMC coated osmo-freeze-dried ginger.

vibrations, which could be attributed to structural changes (Chandra *et al.*, 2021).

4. Conclusion

The application of the CMC coating process improved the mass transfer during the osmotic dehydration process of ginger slices by minimizing the solute gain without affecting the performance of water loss. The Magee model can be used to describe the mass transfer kinetics during the osmotic dehydration process of ginger. The combination of CMC coating and osmotic dehydration process results in higher colour retention compared to untreated freeze-dried ginger. Meanwhile, the structure of the freeze-dried product was also improved with both pre-treatment combinations. This study also confirmed that there is no major change in the ginger functional groups for all treatment processes.

Conflict of interest

The authors declare no conflict of interest.

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