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Microwave-Osmo-Dehydro-Freezing and Storage of Pineapple Titbits—Quality Advantage

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Abstract: Osmotic dehydration is a pre-treatment given prior to finish drying or freezing preservation to improve quality and/or minimize the damaging effects on quality parameters, by partially reducing the moisture content of the sample. Pineapple titbits were partially dried using microwave assisted osmotic dehydration under continuous flow medium spray condition (MWODS) and then frozen with or without a sodium alginate–calcium chloride-based edible coating. The effects of MWODS pre-treatment and edible coating on the quality parameters of pineapple titbits frozen and stored at −20 °C for 10 and 50 days were evaluated after thawing. Both treatments (MWODS and alginate) resulted in superior quality products as compared to the control sample. MWODS, with its advantages over the conventional osmotic dehydration (COD) of rapid and higher moisture removal (16% in 10 min vs. 4 h in COD), while limiting solids gain (2.5% MWODS vs. 4.5% in COD), resulted in improved quality over the control during the frozen storage. The sodium alginate–calcium chloride edible coating treatment further reduced the drip loss in MW-osmo-dehydro-frozen pineapple titbits, possibly due to the effect of the calcium cross linked firming of the fruit texture. Both resulted in enhanced appearance, color and textural properties.

Keywords: pineapple; edible coating; dehydro-freezing; sodium alginate; microwave; osmotic; dehydration

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1. Introduction

Freezing is a commonly used method in the food industry for long-term food preservation, due the increased demand for non-seasonal products. Frozen foods rank next to fresh and refrigerated products due to the consumer perception and the advantage of low temperature preservation (protection from thermal damage) and long-term storage possibilities at lower temperatures. However, freezing can have damaging effects on the textural quality of food products due to ice crystal formation and growth, which is affected by the freezing rates, storage time, storage temperatures and thawing methods. In order to minimize the damaging effects of freezing on textural quality parameters, rapid freezing conditions are usually employed, which result in the formation of smaller ice crystals and cause less damage [1]. However, temperature fluctuations during storage can still lead to partial melting and recrystallization, resulting in the same damage. The results of such damage to cellular structure usually leads to texture collapse during thawing and causes the cellular fluids to be removed as drip loss and the final quality loss in the thawed product. The partial removal of water prior to freezing reduces the amount of water that goes through crystallization during the freezing process and reduces the resulting damage. This technique is referred to as dehydro-freezing and is recognized as a better freezing method than conventional freezing for fruits and vegetables [2]. This pre-treatment, which has been considered especially beneficial in several studies, helps to remove a portion of water from the fruit, reduces the amount of ice formation, increases its cryoprotectancy, and reduces discoloration, energy consumption and drip loss [3]. Furthermore, dehydro-freezing allows the preservation of fruits and vegetables which are highly susceptible to

freeze damage and large ice crystals formation due to their cell wall structure [4]. The dehydro-freezing process has been explored in a number of studies [5–7], and specifically for fruits: tomato [8], strawberry [9], quince [10], apple [11], kiwi [12], peach [13], and mango [14]. Ramallo and Mascheroni (2010) found dehydro-freezing to have a significant quality advantage over the conventional freezing and storage of pineapples [15]. Moreover, James et al. (2015) [7] and Schudel et al. (2021) [16] have provided overviews comparing conventional freezing and dehydro-freezing of vegetables and demonstrated an advantage of the latter for reducing cell damage.

Combined methods of preservation are preferred due to the consumer preference for minimally processed fruits and vegetables [17]. Any method of drying can be used for the partial removal of moisture; however, osmotic dehydration (OD) can bring in additional advantages of energy saving, flavor balancing (moderating the sugar to acid ratio) and other advantages. When osmotic dehydration is used for pre-treatment, then the whole process is known as osmo-dehydro-freezing (ODF) [16]. Moreover, ODF enhances the quality characteristics of the frozen products, such as the color, flavor and texture, by reducing the amount of free water in the fresh produce that will freeze later on, form ice crystals and cause cell wall rupture and tissue damage [18]. Economic advantages can also be achieved through ODF; the partial removal of moisture from fresh produce prior to freezing allows a reduction in the amount of moisture to be frozen, and a reduction in the weight and size of food products [19]. Furthermore, ODF reduces nutrient destruction and enzymatic browning [16]. Several studies on ODF have been carried out with different fruits and vegetables [4,16,17]. While osmotic dehydration pre-treatment has been credited with several benefits, the process is inherently slow and very time consuming. Therefore, several new technologies have been introduced to enhance the OD process, including vacuum, ultrasound, high pressure and microwave drying [20].

A microwave (MW) assisted osmotic dehydration (OD) (MW + OD = MWOD) was designed by Li and Ramaswamy (2006) [21] and it is an excellent alternative for the enhancement of conventional osmotic dehydration (COD). MWOD was designed by placing an osmotic dehydration chamber within the microwave cavity, where fruits are fully immersed in an osmotic solution. The heat generated by the microwave (MW) field is absorbed by the water molecules in the syrup and fruit pieces, which creates an internal pressure gradient that enhances the water movement to the surface of the produce. Moreover, the MW enhances the outflux of water/water vapour, and together with the external osmotic pressure it enhances the dehydration process and reduces processing time. Using MWOD, fewer solutes will be also gained by the fruit samples due to the high-water outflow rate that can cause a resistance for the solids inflow. As a result, the final products were reported to have a better natural flavor and enhanced color, texture and nutritional properties. This MWOD process was modified later on to MWODS, which is microwave osmotic drying with a continuously moving osmotic solution in a spray mode [22]. This technique has been successfully evaluated for many products, such as cranberries [23] and mango fruits [24,25], to demonstrate the quality advantage.

The pineapple (*Ananas comosus*) is a tropical fruit belonging to the family *Bromeliaceae*. Pineapple is used in both its fresh cut form and after pressing it to produce pineapple juice. In many tropical countries, chunks of pineapple are commonly used in desserts such as fruit salad, as well as for toppings in specialty pizza. Crushed pineapple puree is used in several product formulations such as yogurt, jam, sweets, and ice cream. Commercially, pineapples are preserved as slices or titbits by thermal processing, freezing or dehydration processing. The processing procedures used have a large influence of the product quality.

Therefore, it is clear that microwave assisted osmotic dehydration under continuous medium flow spray condition (MWODS) can effectively be used to partially but quickly reduce moisture content in pineapple titbits prior to freezing. A sodium alginate–calcium chloride based edible coating can be applied on cut fruits to firm the fruit structure. Finally, freezing has been a successful method of food preservation. The goal of this study was to evaluate the combination of the above three steps, namely MWODS, alginate coating

and freezing (never done before), into one study, leading to the successful dehydrofreezing of pineapple titbits. Specifically, therefore, the objective of the study was to first employ MWODS pre-treatment to partially dry pineapple titbits, then coat the dried titbits with sodium alginate–calcium chloride edible film, freeze and evaluate the quality change during frozen storage at −20 °C for 10 and 50 days.

2. Materials and Methods

2.1. Sample Preparation

Fresh pineapple fruits were purchased fresh from the market and stored at 4 °C overnight until use the next day to prepare the fruit samples by cutting the pineapple fruits into 3 cm thickness slices in triangular shapes.

2.2. Coating Solution Preparation

Sodium alginate–calcium chloride salts were used in this study as coating materials due to the combined effect as a firming agent and high-water solubility. Distilled water was used to prepare the coating solutions of 2% (*w*/*w*) sodium alginate (Sigma, Oakville, ON, Canada) and 2% (*w*/*w*) calcium chloride (Sigma, Oakville, ON, Canada). To prepare the sodium alginate coating solution, sodium alginate powder was added to distilled water and the beaker was placed on a magnetic stirring rod at 300 rpm with no heat until the sodium alginate powder was completely dissolved at room temperature. To prepare the calcium chloride solution, calcium chloride salts were added in a volumetric flask with distilled water and shaken to completely dissolve the salt [26].

2.3. Conventional Osmotic Dehydration-Samples Preparation

COD was performed with pineapple titbits, using the method of Ferrari et al. (2013) [27] with a few modifications. During COD, 40 °Brix sucrose solution was used, and the product/solution mass ratio was 1:10 (5 pieces/flask). Osmotic dehydration was carried out with continuous stirring using a paddle stirrer at 120 rpm for 4 h at 40 °C.

2.4. MWODS Sample Preparation

The experimental setup has been detailed in Azarpazhooh and Ramaswamy (2010) [22], Wray and Ramaswamy (2015) [23] and Shinde and Ramaswamy (2019; 2021) [24,25]. Briefly, the MWODS setup consisted of a commercial spray head (Waterpik, Model RPB-173C, 12.5 cm diameter, Waterpik Technology Inc., Markham, ON, Canada) placed on a custom-made glass sample chamber (12.5 cm diameter) located inside a domestic microwave (Danby DMW1153BL 0.031 m^3, Guelph, ON, Canada) with a nominal output power of 1100 W at 2450 MHz (Figure 1). The prepared fruit pieces were placed in a nylon mesh bag on the perforated acrylic plate "stage" positioned inside the glass chamber. The acrylic stage allowed the osmotic solution to be collected at the bottom of the glass chamber and then pumped at the required flow rate using a peristaltic pump (Model 75211-30 Digital gear pump, Barnant Company, Barrington, IL, USA through the spray head to shower the fruit samples. Prior to bringing back to the showerhead, the osmotic solution was pumped through a long coil inside a temperature-controlled water bath (Model TDB/4 Groen division, Dover Corp, Downers Grove, IL, USA) to maintain the targeted inlet temperature for the osmotic solution. The temperature of the osmotic solution was monitored using a pair of in-line Type-T thermocouples connected to the digital thermometer (Omega DP-462, Omega Technology, Laval, QC, Canada). The thermocouples were placed externally to the MW oven immediately before admitting the solution in and immediately after exiting the microwave cavity, to measure the temperature of the osmotic solution at inlet and exit locations. The schematic of the experimental setup is illustrated in Figure 1.

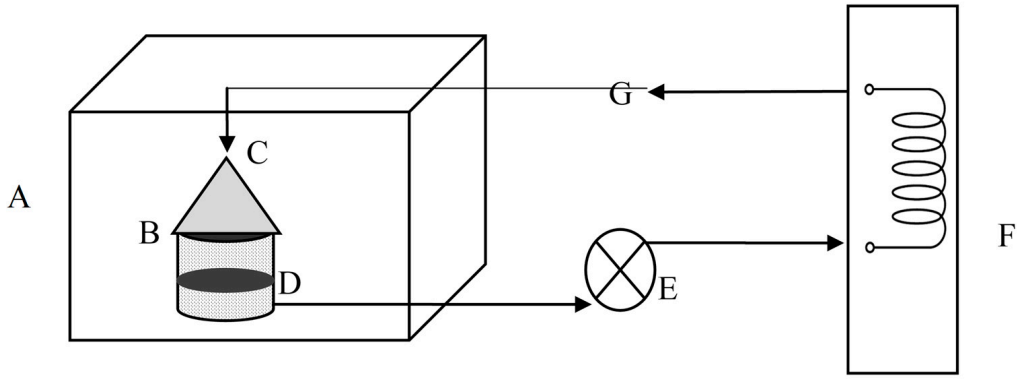


Figure 1. Schematic diagram of MWODS assembly. A: microwave oven cavity, B: microwave transparent sample chamber, C: spray head, D: digital gear pump, E: water bath (containing heat exchanging coils, not pictured), and F and G are thermocouple measuring points immediately before and after the solution enters and leaves the microwave cavity, respectively.

Experiments were carried out using a 40 °Brix sucrose solution. Pineapple titbits were placed in a small plastic mesh bag and transferred into the osmotic dehydration chamber inside the MW oven. The sugar solution was showered on the fruit samples placed on the perforated acrylic plate inside the MW compatible glass container (100 g/run) at 40 °C for 10 min with a continuous medium flow rate of 1500 mL/min. Temperature was monitored, and the syrup circulation system was maintained in continuous mode from the bottom of the chamber up to the spray head. The experiment was carried out for 10 min only since it was a pre-treatment for freezing [23,24].

2.5. MWODS Coated Samples Preparation

Sodium alginate and calcium chloride solutions were poured into plastic containers and the osmotically dehydrated pineapple titbits using MWODS were placed in a plastic mesh and dipped completely into the sodium alginate solution for 5 min, removed and drained for 1 min, then dipped in the calcium chloride solution for 5 min, removed, drained and blotted with a wet filter paper for 10 min at room temperature (22 °C) to remove the surface excess of the coating solution [26].

2.6. Frozen Storage

Uncoated and coated samples were stored in plastic containers, packaged in zip-sealed polyethylene bags and frozen stored in the freezer at (−20 ± 2 °C) for 10 and 50 days. Each container had several pineapple pieces for subsequent analysis. Finally, for analysis, the samples were thawed overnight in a refrigerator at 10 °C.

2.7. Weight Reduction, Moisture Loss and Solid Gain

A digital balance (Denver instrument, APX-323, NY, USA) was used to measure the weight of the pineapple fruit samples before and after osmotic dehydration (both COD and MWODS) to calculate the weight reduction percentage after osmotic dehydration. The oven method was used to determine moisture content in both fresh and osmotically treated fruit samples using COD and MWODS. Fruit samples were kept overnight in a hot air oven at 105 °C. The following equations were used to calculate Weight Reduction (WR) (%), Moisture Loss (ML) (%) and Solid Gain (SG) (%):

$$\bullet \ \text{WR}\ (\%) = 100\ \frac{(M_0 - M_t)}{M_0} \tag{1}$$

$$\bullet \ \text{SG}\ (\%) = 100\ \frac{(M_t \cdot s_t - M_0 \cdot s_0)}{M_0} \tag{2}$$

$$\bullet \text{ ML } (\%) = 100 \frac{(M_0 \cdot x_0 - M_t \cdot x_t)}{M_0} \quad (3)$$

where M_0 is the mass of the sample at time zero and M_t is the mass of the sample at time t; s_t is the solid fraction (dry matter) at time t and s_0 is the solid fraction at time 0; x_0 is the moisture fraction at time 0, while x_t is the moisture fraction at time t [23].

2.8. Ratio of Moisture Loss over Solids Gain

The ratio of ML/SG was used to describe osmotic dehydration efficiency and calculated with ML over SG [22].

$$\text{Ratio} = \frac{\text{ML}}{\text{SG}} \quad (4)$$

2.9. Drip Loss

Drip loss was calculated by taking the weight of the thawed samples every 10 min for 1 h at room temperature, until constant weight was reached. The difference between the weight measured at any time (t) and the initial weight of the sample was described as the drip loss (%) [18].

$$\text{Drip Loss (t) } (\%) = \frac{\text{Weight before thawing } - \text{ weight after thawing (t)}}{\text{Weight before thawing}} \times 100 \quad (5)$$

2.10. Color

The color characteristics of pineapple titbits were measured using a calorimeter, which was the tristimulus Minolta Chroma Meter (Minolta Corp, Ramsey, NJ, USA), to determine the L* value (lightness), a* (green–red chromaticity) and b* (yellow–blue chromaticity), Chroma (C value) and Hue angle (H value). The calorimeter was calibrated using a white standard. The color parameters of pineapple titbits before and after various treatments were measured. Readings were carried out at room temperature on five to six samples of control and coated samples [28].

2.11. Texture

Seven to ten samples from each lot (control and coated) were subjected to a puncture test using a TA XT plus Texture Analyzer (Texture Technologies Corporation, Hamilton, MA, USA/ Stable Micro Systems, Godalming, Surrey, UK) fitted with a 25 mm diameter round tipped puncture probe with a speed of 10 mm/s. The force deformation and firmness of the fruit samples were measured based on the force-deformation curve. Measurements are in Newton (N) [28].

2.12. Statistical Analysis

Commercial statistical analysis software (Analysis ToolPak in Excel, 2007 Version) was used to conduct one-way or two-way ANOVA at 95% level of confidence and 5% level of significance ($p < 0.05$). The testing conditions were different for the two parts, as pre-treatment in the first part was used to demonstrate the appropriateness of MWODS and then in the second part for dehydro-freezing/storage studies. A 5% significance ($p < 0.05$) level was used for differences. Experiments were performed in triplicate.

3. Results and Discussion

3.1. Weight Reduction

Weight reductions in the pineapple titbits after MWODS and conventional osmotic dehydration are compared in Figure 2a. WR with MWODS was on average 14.8% after only 10 min of treatment. However, with COD, the WR reached 14.4% only after almost 4 h of treatment. MWODS was, therefore, highly effective in reducing the treatment time. One-way ANOVA statistical analysis showed a significant difference between MWODS and COD ($p < 0.05$).

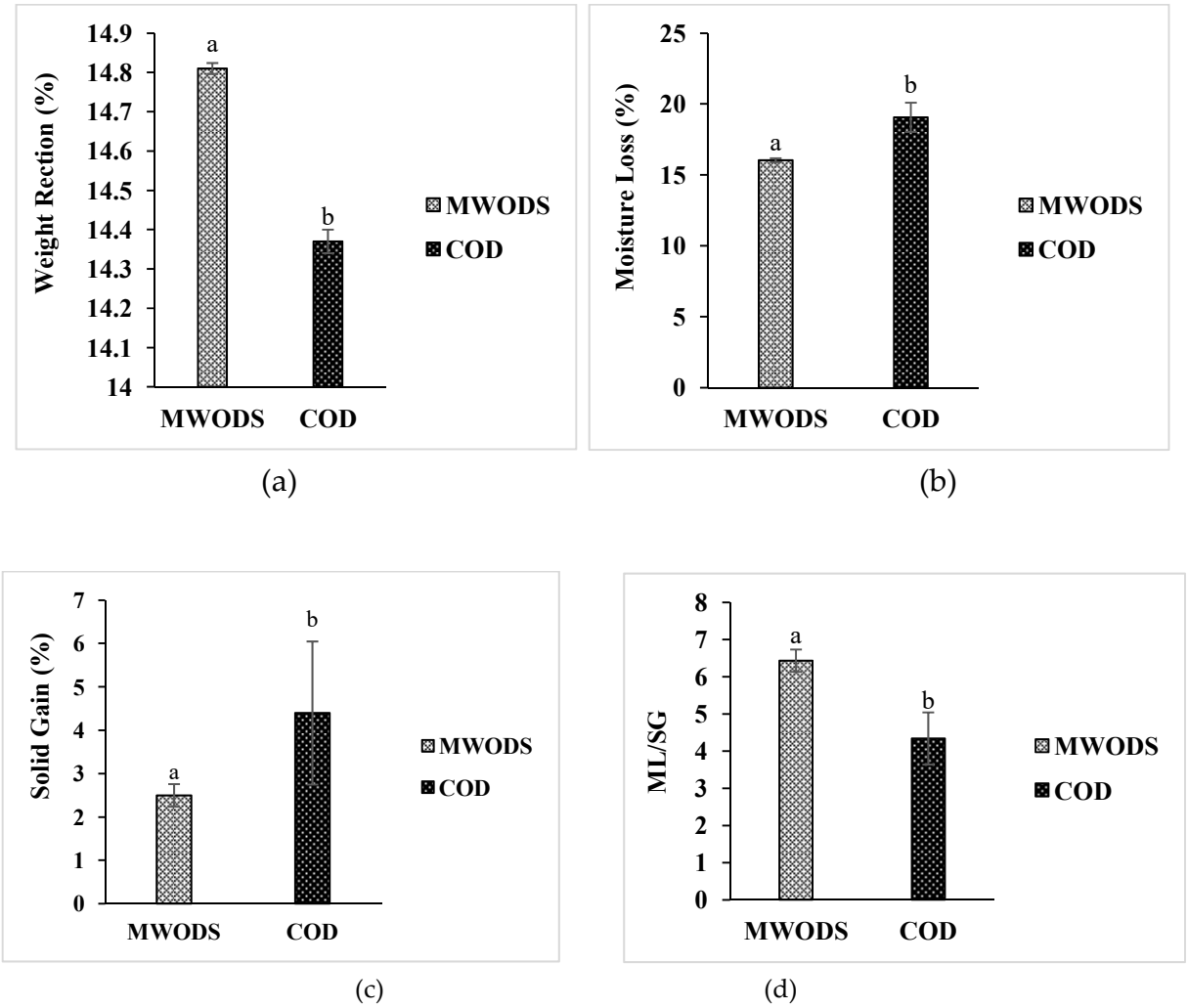


Figure 2. Weight reduction (%) (**a**), Moisture loss (%) (**b**), Solid gain (%) (**c**) and ML/SG ratio (**d**) in osmotically dehydrated pineapple titbits using conventional osmotic dehydration (COD) and Microwave Assisted Osmotic Dehydration in Spray Mode (MWODS). Different lowercase letters indicate a significant difference between MWODS and COD ($p < 0.05$).

Similar results were reported by Azarpazhooh and Ramaswamy (2010) [22], using MWODS under spray conditions at 40 °C using 40 °Brix sucrose solution, as used in this study, with an average 20% weight reduction in the initial weight of apples in 20 min. However, 210 min were required to achieve the same WR (%) under similar conditions using COD. Several previous works have also reported very long treatment times associated with COD. For example, Silva et al. (2014) [29] reported that, using COD with 40% and 60% sucrose solutions at 27 °C, the time required to reach a weight reduction of 24% to 40% was approximately 6 h.

3.2. Moisture Loss

After 10 min of MWODS, the moisture loss (ML) in pineapple titbits was 16.0%. After the subsequent dipping of the MWODS treated samples in the sodium alginate and calcium chloride coating solutions, ML increased further to 19.1% as a result of the coating treatment. Again, the similar ML was reached in COD only after 4 h of treatment. OD in a microwave environment helped to increase the speed and extent of moisture transport diffusing through the tissues of the fresh fruits. One-way ANOVA statistical analysis showed a significant difference in ML between the MWODS and COD ($p < 0.05$) (Figure 2b).

Similar behavior was also observed in cranberries [23], and in mango fruits [24] under similar conditions. Up to 40% ML was reported after 30 min MWODS [24]. It was also found that the moisture loss in apple cylinders after 30 min was 10% and reached 30% after 3 h of MWODI (immersion), in comparison with 18% moisture loss after 3 h of COD with the osmosis carried out at 40 °C and 40 °Brix [21]. A much higher ML of 44% was obtained by Azarpazhooh and Ramaswamy (2010) [22] in 30 min using MWODS, due to a better penetration of the microwave in the apple tissue in the spray mode as compared to microwave assisted osmotic dehydration by immersion (MWODI) and COD. The spray mode (MWODS) also extended the ML further to 50% after 2 h of treatment. The enhanced microwave heating effect was reported to be behind the higher moisture loss in the microwave and medium circulation in the spray mode compared to the conventional osmosis. Unfortunately, there are no other reports on MWODS to compare with other than results from our own laboratory.

3.3. Solid Gain

The solid gain (SG) in the fruit samples in MWODS was only 2.5% after 10 min, in comparison to 4.4% using COD after 4 h. One-way ANOVA statistical analysis showed a significant difference between the MWODS and COD ($p < 0.05$) (Figure 2c). MWODS treatment has been reported earlier to reduce the SG (%) in several fruits. Using MWODS on apple fruits, solids gain has been reported between 2.5% and 3% in the first 30 min of treatment and gradually increased to 3 to 4.5% after 120 min of treatment [22]. It was also reported a decrease in solids gain in apple cylinders, using MWODI process in comparison with COD; however, the reported values were, relatively, higher. At 40 °C and 40 °Brix, a 16% decrease in solid gain was reported using MWODI [21]. With mango pieces, a 5.2% solid gain after 30 min of MWODS was reported by Shinde and Ramaswamy (2019) [24]. In cranberries, also, a decrease in the solid gain was reported with an increase of sucrose concentration due to the formation of a dense layer on the fruit surface that reduced the uptake of solutes [23].

3.4. ML/SG Ratio

The ratio of moisture loss to solids gain has been considered an important factor and an indicator for the optimization of the osmotic dehydration [21–23]. ML/SG ratio was significantly higher in samples treated under MWODS in comparison with conventional osmotic dehydration (6+ in MWODS vs. ~4 COD). One-way ANOVA statistical analysis showed a significant difference between the MWODS and COD ($p < 0.05$) (Figure 2d). It is desirable to have a higher moisture loss and a low solid gain, which results in a higher ML/SG. Similar results have been reported in earlier studies for different fruits with differences ranging between twelve and five between MWODS and COD Azarpazhooh and Ramaswamy, 2010 (apple) [22]; Li and Ramaswamy, 2006 (apple) [21]; Wray and Ramaswamy, 2015 (cranberry) [23] and Shinde and Ramaswamy, 2019 (mango) [24].

3.5. Drip Loss

Osmotically dehydrated samples using conventional osmosis (COD) showed discoloration and extremely soft texture following 4 h of treatment (poor quality), and for this purpose samples treated with COD were not frozen and stored. Only untreated samples (as control), and MWODS samples with and without sodium alginate–calcium chloride treatment were frozen and stored at −20 °C.

Drip loss results mainly from the texture damage to fruits due to the formation of ice crystals during freezing [1,2]. The increase in the drip loss (%) indicates slow crystallization or high recrystallization rates, resulting in the growth of ice crystals during freezing and/or during the frozen storage. The drip loss was measured every 10 min for up to 60 min holding at room temperature for the untreated samples (control samples) and osmotically dehydrated samples under MWODS (MWODS samples and MWODS-coated samples), following storage at −20 °C for 10 and 50 days.

3.5.1. Effect of MWODS on Drip Loss

After 10 days and 50 days of storage, a lower drip loss (%) was observed in MWODS samples when compared with the untreated control samples (COD samples were not frozen and stored, as mentioned earlier). MWODS treatment reduced the drip loss after thawing, probably due the protection offered because of dehydro-freezing, as detailed in the introduction section; it helps to remove a portion of water from the fruit, reduces the amount of ice formation, increases its cryoprotectancy, and reduces discoloration, energy consumption and drip loss [2,3]. These observations observed with convective dehydro-freezing will gain an additional advantage with MWODS because of the extra benefits resulting from the influence of MW and osmotic dehydration, resulting in more effective ML and a better ML/SG ratio. Two-way ANOVA statistical analysis was used to show the significant difference in the drip loss (%) between the untreated control samples and MWODS samples ($p < 0.05$) after 10 days and 50 days of storage (Table 1).

Table 1. Drip loss (%) in untreated control, MWODS samples and MWODS-coated samples after 10 and after 50 days of storage at −20 °C.

Storage Time Days	Samples	Thawing Time 0 min	Thawing Time 10 min	Thawing Time 20 min	Thawing Time 30 min	Thawing Time 40 min	Thawing Time 50 min	Thawing Time 60 min
10	Control	$0^{a,A}$	$2.03 \pm 1.5^{d,A}$	$5.98 \pm 1.7^{c,A}$	$8.33 \pm 2.1^{e,A}$	$9.63 \pm 2.3^{g,A}$	$11.45 \pm 3.04^{m,A}$	$13.26 \pm 3.6^{q,A}$
	MWODS samples	$0^{a,A}$	$0.16 \pm 0.1^{b,C}$	$1.59 \pm 0.2^{d,C}$	$4.001 \pm 0.7^{f,C}$	$6.74 \pm 0.3^{k,B}$	$9.47 \pm 0.9^{t,C}$	$11.65 \pm 1.07^{n,C}$
	MWODS-coated	$0^{a,A}$	$0.06 \pm 0.6^{c,C}$	$0.72 \pm 0.6^{e,C}$	$2.07 \pm 0.1^{b,C}$	$3.3 \pm 0.1^{b,C}$	$4.48 \pm 1.06^{c,C}$	$6.1 \pm 1.6^{c,C}$
50	Control	$0^{a,A}$	$1.37 \pm 0.8^{c,B}$	$4.87 \pm 1.2^{e,B}$	$7.73 \pm 1.2^{b,B}$	$9.87 \pm 1.6^{c,A}$	$11.32 \pm 1.7^{c,A}$	$14.36 \pm 2.06^{c,B}$
	MWODS samples	$0^{a,A}$	$0.67 \pm 0.2^{d,D}$	$2.88 \pm 0.4^{f,D}$	$5.91 \pm 1.3^{c,D}$	$9.04 \pm 1.1^{c,C}$	$11.27 \pm 1.2^{c,D}$	$14.36 \pm 0.3^{c,D}$
	MWODS-coated	$0^{a,A}$	$0.07 \pm 0.1^{e,C}$	$0.85 \pm 0.1^{d,C}$	$2.31 \pm 0.2^{d,C}$	$4.10 \pm 0.1^{d,D}$	$5.52 \pm 0.9^{d,D}$	$8.64 \pm 1.1^{d,D}$

Different lowercase letters indicate a significant difference ($p < 0.05$) between untreated control, MWODS samples and MWODS-coated samples at similar times of storage to study the effect of MWODS treatment and sodium alginate-based edible coating. Different uppercase letters indicate a significant difference ($p < 0.05$) between untreated control after 10 days and 50 days of storage, MWODS samples after 10 days and 50 days of storage, and MWODS-coated samples after 10 days and 50 days of storage to study the effect of storage time. Thawing time 50–60 min until a constant drip loss.

Some results have been reported with osmo-dehydro-freezing (ODF) using different sugar solutions, suggesting, for example, reduced drip loss (%) in frozen rambutan after thawing [30]. Additionally, a reduction in drip loss was observed in frozen–thawed strawberries due to the osmotic dehydration treatment prior to freezing [20], in frozen apples [2], and in frozen Kiwi fruits after thawing [31]. More recently, Ramallo and Mascheroni (2010) [15] reported some reduction in the drip loss in ODF pineapple cut fruits after thawing. However, all these reports involved COD of up to 240 min, as compared to a 10 min MWODS treatment as carried out in this study, with a similar magnitude of ML. The higher drip loss in normal frozen–thawed samples is caused by the increased ice crystal damage in the tissue [31]. The drip loss has been generally reported to be lower following some form of OD, and the extent of reduction has been reported to increases with the OD treatment time [2,15].

3.5.2. Effect of the Coating on Drip Loss

It has been generally accepted that freezing results in changes in the microstructure of plant tissues, resulting in texture damage, shrinkage and drip loss. For fruit and vegetable processing, calcium salts (calcium chloride) have been also frequently used for texture firming since calcium can bridge with low methoxyl pectin present to form a three-dimensional network. Some studies make use of the addition of exogenous pectin methylesterase (PME) to catalyze the decomposition of pectin to form pectic acid, which can react with Ca^{2+} to form calcium pectate, a molecular gel [32]. Some studies also proved that sodium alginate dispensed to a plant matrix and then treated with calcium chloride can create a strong three-dimensional network that can yield textures ranging from a soft gel to a hard structure [33]. The soluble sodium alginate will transform to insoluble calcium alginate during this treatment, causing the texture firming. That is why the combination of sodium alginate and calcium chloride treatment was evaluated

in this study. Sodium alginate–calcium chloride treatment has been traditionally used to firm fruits and vegetables. The sodium alginate–calcium chloride coating helped to reduce the drip loss in pineapple titbits by 47% after 10 days of freezing storage at −20 °C, and 40% after 50 days of freezing storage at −20 °C, thus acting as a natural barrier. However, the duration of the frozen storage negatively affected the drip loss in both control and coated samples, with a 24% and 41% increase in drip loss, respectively, after 50 days of storage. Two-way ANOVA analysis showed a significant difference in the drip loss (%) between the MWODS samples and MWODS-coated samples ($p < 0.05$), both after 10 days and 50 days of storage at −20 °C (Table 1).

Clearly, an effect of the recrystallization phenomena that occurs in the frozen foods during storage leads to an increase in the structural breakdown. During frozen storage, the development of extracellular ice crystals will cause cell separation in the middle lamella region, cell-wall rupture and cell shrinkage, which will result in quality loss, structure collapse, water dislocation and exudate production, in addition, to multiple deteriorative biochemical reactions. The deterioration of the structural integrity of the plant membranes will also prevent the retention of the hydrostatic pressure within the cells and causes drip loss and tissue softening [34]. This explains the increase in drip loss (%) over frozen storage time in untreated samples, MWODS samples and MWODS-coated samples (Table 1).

Some similar general results have been observed in different studies with slightly different context (freezing or dehydro-freezing) or fruits. Sodium alginate-based edible coating was reported to reduce the drip loss in the osmotically dehydrated and frozen strawberries after thawing [20]. Whey protein edible coating with and without the incorporation of beeswax was reported to reduce drip loss (%) in frozen strawberries after thawing [35]. In another study, chitosan-based edible coating was found to reduce drip loss in frozen strawberries after thawing [18]. It can be reasonably assumed that less cellular damage occurs during freezing and/or storage, and that more cells survive in the dehydro-frozen samples [7]. Furthermore, the increasing drip loss was also in agreement with the decreasing texture values [3], as will be detailed in the next section.

3.6. Texture

3.6.1. Effect of MWODS on the Texture

Before freezing and after using MWODS treatment, a decrease in the firmness of the pineapple titbits from 61.1 (N) to 55.2 (N) was observed. This is the effect of MWODS treatment on the texture of pineapple fruit pieces. MWODS resulted in some texture degradation, as most dehydration processes will do. The magnitude of this reduction is about 10%, which is not unusual after osmotic dehydration treatment due to removing as much as 19% of the moisture.

The evaluated texture after frozen thawed MWODS demonstrated that the MWODS process improved the texture of the pineapple titbits. The firmness was 20% higher in MWODS samples as compared with untreated (control) samples after 10 days of storage, and 14% higher after 50 days of storage. During the frozen storage, there was a progressive loss in texture in both control and MWODS samples, with the 10 day stored samples showing better texture values than the 50-day frozen stored sample (Figure 3). ANOVA results showed a significant difference between the untreated control and MWODS samples before freezing and after thawing ($p < 0.05$). The improvement in the firmness of the pre-treated strawberries was reported to be caused by the protective effect on the cell's integrity induced by the osmotic dehydration process [3]. Similar results were observed in pre-dehydrated frozen-thawed apples [5]. In this study, pre-dehydrated samples showed better texture after freezing and thawing. Cell structure deterioration was caused by the high moisture content in the fruit samples, which caused cell wall and texture damage during freezing/thawing. Moreover, osmotic dehydration of strawberries for 4 h prior to freezing showed much better tissue organization and a better texture after thawing when compared with the strawberry frozen without pre-treatment [36]. Osmo-dehydro-freezing using conventional osmotic dehydration has been credited with a positive effect

on the mechanical properties of kiwi after freezing and thawing when compared with untreated samples [31]. An improvement in the firmness of dehydro-frozen carrot and bell pepper was also reported by Schudel et al. (2021) [7], as it increased by 35% and 52% after dehydration with a moisture loss of 27% and 16%, respectively. Thus, ODF in general is superior to conventional frozen thawed fruits and MWODS-frozen fruits are even better than ODF samples.

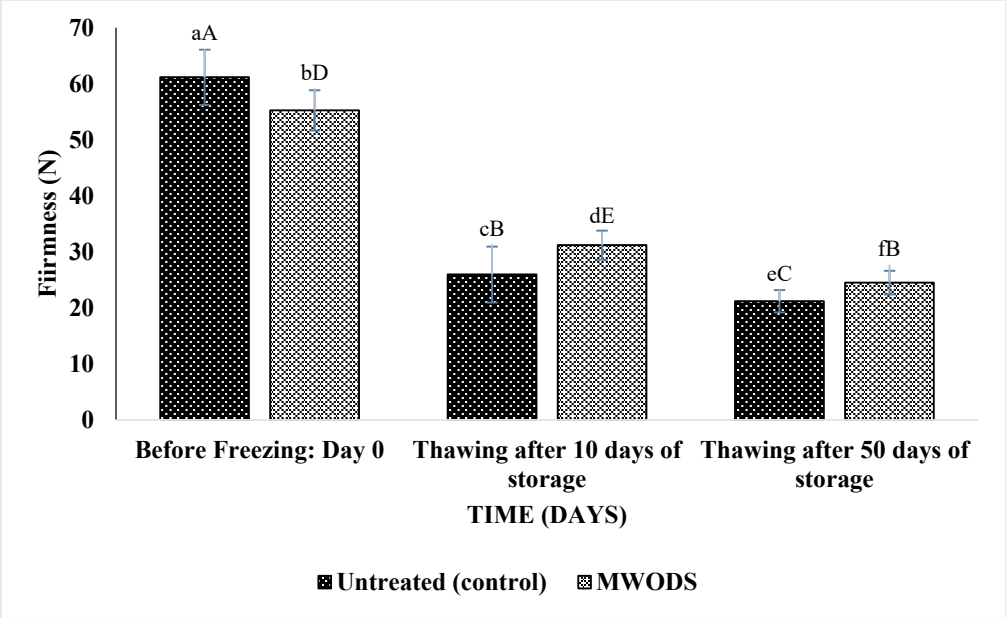


Figure 3. Firmness (N) of untreated fresh samples (control) and MWODS samples prior to freezing and after thawing following 10 and 50 days of frozen storage. Different lowercase letters indicate a significant difference between untreated control and MWODS samples at similar times ($p < 0.05$) to study the effect of MWODS treatment. Different uppercase letters indicate a significant difference between untreated control samples at different times and MWODS samples at different times to study the effect of storage time.

3.6.2. Effect of the Edible Coating on Texture

The edible coating generally helps to preserve the texture of the fruit samples by reducing the moisture loss and firmness after thawing. The firmness was 16% higher in the MWODS-coated samples as compared with the MWODS control samples after 10 days of storage, and 18% higher after 50 days of storage. ANOVA statistical analysis showed a significant difference between the MWODS control samples and MWODS-coated samples, and before freezing and after thawing ($p < 0.05$) (Figure 4). Han et al., (2004) [37] reported some beneficial effects of chitosan coating with and without the incorporation of calcium salts on the texture preservation of strawberries after thawing. The addition of calcium chloride salts further aided in the texture protection due to their role as firming agents. Calcium chloride salts can also interact with the pectic acids found in the plant's cell walls and form calcium pectate that can maintain the structure of the fruits [35].

3.7. Color

The L* value, a* value, b* value, C value and H value were measured and compared. The a* and H values did not show any significant difference between untreated control samples, MWODS samples and MWODS-coated samples ($p > 0.05$).

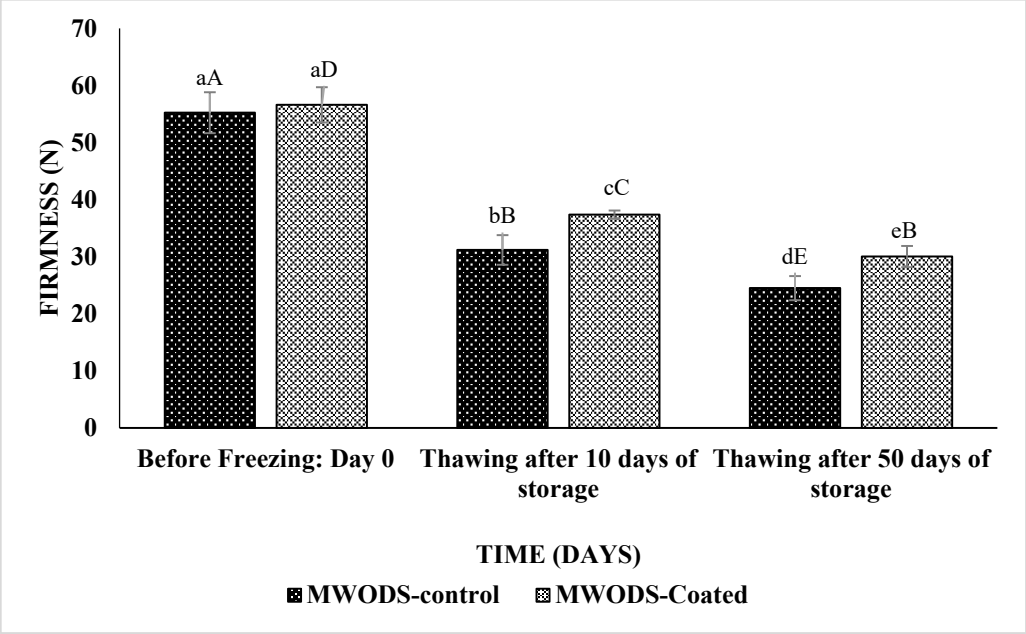


Figure 4. Firmness (N) of MWODS control samples and MWODS-coated samples prior to freezing and after thawing. Different lowercase letters indicate a significant difference ($p < 0.05$) between MWODS control samples and MWODS-coated samples at similar times to study the effect of sodium alginate-based edible coating. Different uppercase letters indicate a significant difference ($p < 0.05$) between MWODS control at different times and MWODS-coated at different times to study the effect of storage time.

3.7.1. L* Value

The MWODS treatment caused some surface dullness or darkening, resulting in a decrease in the lightness value of the pineapple titbits (Table 2). Storage time further affected the lightness of the uncoated samples, resulting in further decreases in L values after 10 and 50 days of storage. However, in MWODS-coated samples, the L* value was almost the same after 10 and 50 days of storage. The sodium alginate–calcium chloride edible coating therefore helped to better maintain the surface lightness of the fruit samples during freezing and prevented surface darkening. The L* value in MWODS samples was lower than in MWODS-coated samples after 10 and 50 days of storage. The L* values were, respectively, 25% to 38% lower in MWODS samples compared with MWODS-coated samples. ANOVA statistical analysis showed a significant difference in the L* value between the untreated samples (control) and MWODS samples after thawing following 50 days of storage, while a significant difference between MWODS samples and MWODS-coated samples was observed after thawing following 10 and 50 days of storage ($p < 0.05$) (Table 3).

Silva et al. (2014) [29] reported that osmotically pretreated frozen pineapples showed a decrease in the L value after thawing, and a decrease in the luminosity was observed in osmotically dehydrated samples in comparison with fresh samples.

Table 2. L* values, b* values and C values in pineapple titbits before and after MWODS treatment.

	Untreated Control Samples	MWODS Samples
L* value	61.95 ± 1.4^{a}	42.01 ± 0.7^{b}
b* value	34.91 ± 1.2^{a}	33.19 ± 1.6^{a}
C value	34.95 ± 1.2^{a}	33.21 ± 1.6^{a}

Different lowercase letters indicate a significant difference between untreated control samples and MWOD samples to study the effect of MWODS treatment ($p < 0.05$).

Table 3. L* values, b* values and C values in untreated control samples, MWODS samples and MWODS-coated samples after thawing.

	Thawing after 10 Days of Storage at −20 °C			Thawing after 50 Days of Storage at −20 °C		
	Untreated Control Samples	**MWODS Samples**	**MWODS-Coated Samples**	**Untreated Control Samples**	**MWODS Samples**	**MWODS-Coated Samples**
L* value	33.37 ± 2.7^{aA}	31.52 ± 1.04^{aA}	41.59 ± 0.75^{cC}	28.8 ± 2.4^{cB}	25.83 ± 2^{aB}	41.2 ± 1.34^{bC}
b* value	29.42 ± 2.1^{bA}	25.04 ± 1.04^{cA}	27.02 ± 0.73^{cB}	23.94 ± 1.9^{cE}	21.99 ± 1.7^{cC}	25.59 ± 3.1^{dB}
C value	29.47 ± 2.1^{bA}	25.31 ± 1.05^{dA}	27.44 ± 0.72^{dB}	24.00 ± 1.2^{eE}	22.29 ± 1.7^{eC}	25.65 ± 3.1^{fB}

Different lowercase letters indicate a significant difference between untreated control, MWODS samples and MWODS-coated samples at similar times of storage to study the effect of MWODS treatment and sodium alginate-based edible coating. Different uppercase letters indicate a significant difference ($p < 0.05$) between untreated control after 10 days and 50 days of storage, MWODS samples after 10 days and 50 days of storage, and MWODS-coated samples after 10 days and 50 days of storage to study the effect of storage time. Thawing time 50–60 min until a constant drip loss.

3.7.2. b* Value

The b* value, which is the green/yellow indicator, was measured in untreated control samples, MWODS samples (uncoated) and MWODS-coated samples. No significant difference in the b* values was observed after MODS treatment (Table 2). After thawing, the b* value was also measured. The b* value decreased in all the samples after thawing. However, the b* values in MWODS samples were lower than in MWODS-coated samples. Moreover, the freezing storage negatively affected the yellow color of the pineapple titbits, although the coated samples showed better results. The sodium alginate–calcium chloride edible coating preserved the yellowness of the fruit samples during freezing and storage for 10 and 50 days at −20 °C. One-way ANOVA statistical analysis showed a significant difference in the b* value in untreated control samples, MWODS samples and MWODS-coated samples after thawing following the two different storage periods ($p < 0.05$) (Table 3).

It was shown that Dermesonlouoglou et al. (2016) [3] after the thawing of osmotically pretreated strawberry fruits stored at low temperatures (above −12 °C) and very low freezing temperatures (below −12 °C), a retention in the color was observed in the osmotically dehydrated samples when compared with the untreated samples prior to freezing.

3.7.3. C Value

After thawing, the C value was measured in untreated control samples, MWODS samples (uncoated) and MWODS-coated samples. After thawing, the C value decreased in untreated control samples, MWODS samples and MWODS-coated samples. However, lower values were obtained in MWODS samples. Additionally, the increase in storage time negatively affected the color Chroma in all the samples. The sodium alginate–calcium chloride edible coating somewhat reduced the loss of color chromaticity during storage.

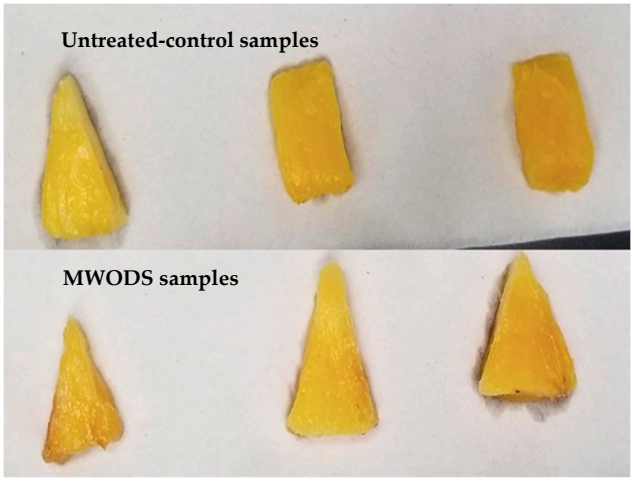
Using one-way ANOVA statistical analysis, a significant difference in the C value in untreated control samples, MWODS samples and MWODS-coated samples was observed after thawing following 10 days and 50 days of storage ($p < 0.05$) (Table 3).

Based on the literature, an increase in the C value is normally observed in osmotically pretreated pineapples prior to freezing. The increase in the sugar concentrations caused by the higher moisture loss, which also increases the concentration of the color pigments in the plant's tissue, might resulted in an increase in the chromaticity values. Such results have been observed using papaya and guava [29].

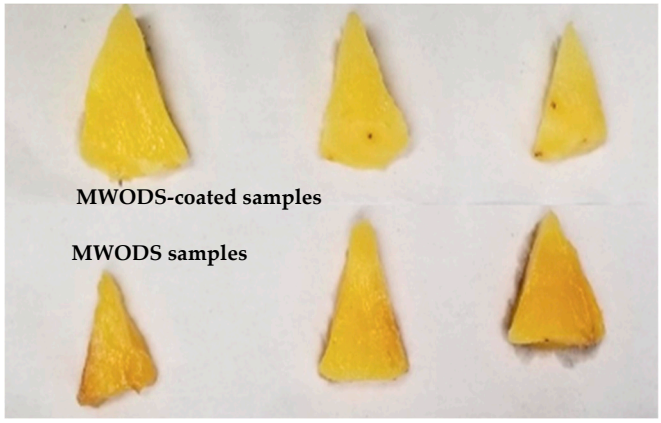
3.8. Appearance

A slight browning was observed in the pineapple titbits after MWODS treatment, and a change in the appearance was observed in the untreated control samples and MWODS samples after thawing following 10 days of storage at −20 °C (Figure 5a). The thawing of the pineapple titbits after 10 days of frozen storage at −20 °C caused a change in the appearance and surface darkening in uncoated samples. The MWODS-coated samples showed no

surface browning, and the appearance of coated pineapple titbits was fully maintained after thawing (Figure 5b). Similar results were observed after thawing following 50 days of storage at −20 °C (Figure 5c). The sodium alginate–calcium chloride edible coating maintained the appearance of the pineapple titbits and prevented surface darkening.



(**a**)



(**b**)

(**c**)

Figure 5. The appearance of untreated control and MWODS samples (in triplicate) after thawing for 1 h at room temperature following 10 days of storage at −20 °C (**a**). The appearance of MWODS samples and MWODS-coated samples after thawing for 1 h at room temperature following 10 days of storage at −20 °C (**b**) and 50 days of storage at −20 °C (**c**).

4. Conclusions

MWODS was used as a pre-treatment to reduce the moisture content in pineapple titbits prior to freezing. In comparison with the COD, a significant enhancement in ML and a significant reduction in the treatment time and solid gain (%) were also observed in pineapple titbits treated with MWODS. This combination resulted in almost doubling the ML/SG ratio, a parameter that has been closely related to the quality of osmotically dehydrated products. Additionally, an enhancement in the texture and a reduction in drip loss after the thawing of the pineapple titbits was clearly observed. Similar results have been demonstrated earlier in mango fruits, using MWODS with the incorporation of maltodextrin (MD). The MWODS process helped to restrict the solids uptake, and enhanced ML and the ML/SG ratio. Furthermore, the special sodium alginate–calcium chloride edible coating applied to the osmotically dehydration pineapple titbits after the MWODS technique helped to significantly reduce the drip loss and enhance the sensory properties after thawing. Based on the results analysis, the sodium alginate–calcium chloride coating was recommended as an excellent pre-treatment for enhancing the quality parameters of frozen pineapple titbits by preventing surface darkening, reducing drip loss and maintaining the tissue texture.

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