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Research Article

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Application of Microencapsulation of Black Plum Peel Extract in the Production of Hard Candy and its Effect on Chemical Properties, Antioxidant Capacity, and Phenolic Compounds

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Abstract

Microencapsulation in the food industry is a relatively new technique that plays a role in increasing the shelf life of active compounds during processing, as well as storing and delivering active agents to target sites in the body. The aim of this study was to use microencapsulated black plum peel (BPP) extract, rich in antioxidant compounds, to produce a functional hard candy and examine its chemical properties. For this purpose, maltodextrin, starch, pectin, and gelatin were used to produce the coating. An increase in the percentage and type of hydrocolloids, along with an increase in dextrin content, showed a significant effect on the antioxidant capacity and total phenolic compounds (TPC) of the microencapsulated coating. The antioxidant capacity and PC of the samples ranged between 40.75% - 4.48% and 53.46 mg gallic acid equivalents (GAE)/100 g fresh weight (FW) - 6.92 mg GAE/100 g FW, respectively. Increasing the temperature did not have a significant effect on the antioxidant capacity or TPC. However, increasing the pH resulted in an increase in antioxidant compounds and a decrease in TPC. Based on the results, the antioxidant capacity and PC of the core ranged between 88.50% - 74.33% and 873.55 mg GAE/100 mg FW - 666.54 mg GAE/100 g FW, respectively. Hard candy was produced with 25% dextrin and 1.5% of each hydrocolloid (Gelatin and pectin) at a temperature of 50 °C and a pH of 2. Chemical analysis results showed no significant difference between the various samples containing different types of hydrocolloids in terms of moisture content (MC), ash content (AC), antioxidant capacity, and PC. Therefore, any of the hydrocolloids can be used for producing hard candy. The production of candy with encapsulated antioxidant compounds can result in a new functional product. Additionally, due to the optimal use of BP processing waste, it has the potential to gain a favorable position in the industry in terms of exports and the growth of the agricultural economy and environmental protection.

Keywords

Black plum peel, Hard candy, Processing waste, Microencapsulation

Introduction

Candy is a tasty and delightful product favored by all. The word "candy" is derived from the Arabic word "Gandi", which originates from the Persian word "Gand", meaning sugar. Hard candy is a common type of sugar candy that contains water, sugar, coloring, and flavoring [1]. Hard candy is classified as non-crystalline and is cooked at high temperatures (140 - 150 °C), resulting in a hard texture and glossy appearance. The main ingredients of hard candy are sucrose, water, and glucose syrup or inverted sugar [1]. Hard candy production has expanded due to its simplicity, long shelf life, and the pleasant sensation it creates [2]. Natural or artificial flavorings can be used to create a desirable taste in food products. Today, due to the harmful effects of artificial flavorings, it is recommended to use natural flavorings in food products [3]. The food industry is seeking alternatives rich in natural antioxidants, which play an important role in preventing cardiovascular diseases, cancer, and neurogenic disorders [4]. Artificial colors can make food, especially for children, more appealing. However, studies conducted in recent decades show that these artificial colors or their contaminants can be carcinogenic or neurotoxic to humans [5].

One of these natural materials is plum peel, which, based on preliminary tests, can be used to produce natural flavor and color. A significant amount of plum peel remains during the drying process of plums. So far, no application has been proposed for this valuable food material, although it contains valuable compounds such as anthocyanin and PC. Given the large volume of this product and its nutritional and therapeutic properties, it can be used to produce high-value-added products [6].

Microencapsulation is a relatively new technique in the food industry that plays a role in extending the shelf life of active compounds during processing, as well as storing and delivering active agents to target sites in the body. A microcapsule consists of a core and a shell. The shell's function is to protect sensitive food components, such as flavors, vitamins, or salts, from water, oxygen, and light, transform liquids that are difficult to transport into free-flowing powders, and separate certain food components from others during storage [7]. Choosing the right encapsulating material is crucial as it affects the efficiency and stability of the encapsulation process. Some materials used for encapsulation walls include carbohydrates (starch, modified starch, dextrin, sucrose, cellulose, and chitosan), gums (Arabic gum, alginate, and carrageenan), lipids (wax compounds, paraffin, monoglycerides, hydrogenated oils, and fats), minerals (calcium sulfate and silicate), and proteins (gluten, casein, gelatin, and albumin) [8].

De Moura et al. [9] used encapsulated Hibiscus extract in candy production. The aim of their study was to evaluate the release of anthocyanin under simulated gastrointestinal conditions and to use these microcapsules in pectin-based candy production. The use of encapsulated anthocyanin in jelly candies showed that up to 73% of the compounds could be preserved. Akib et al. [2] used red beet for flavor and rheological properties in candy. The study aimed to find the best hard candy formula through a hedonic test, including organoleptic (sensory) properties, water content, AC, and hardness. Four formulas were tested: K (without red ginger extract), A (1% extract), B (1.75% extract), and C (2.5% extract). Among the samples, sample A had the highest acceptance. Charoen et al. [10] used crude extract from *Psidium guajava* in jelly candy production. The aim of their research was to expand the production of jelly candies based on gum using this plant. The study tested three temperature levels (50, 60, 70 °C) for drying the plant, and the antioxidant capacity of the dried samples was measured at three pH levels (4, 6.5, and 8.5) using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) method. The results showed that 50 °C and a pH of 4 yielded the highest antioxidant levels. In 2015, Hooda [5] developed a plant-based candy to minimize the side effects of high-altitude conditions and reduce oxygen deficiency [5].

The purpose of this research was to use microencapsulated BPP extract, which is rich in antioxidant compounds, to produce functional hard candy and examine its chemical properties. Starch, pectin, and gelatin were used for the coating. After determining the antioxidant capacity and PC in the shell, the best combination of hydrocolloids was identified. Then, the appropriate temperature and pH for producing the core were identified, and finally, candy with the highest antioxidant and PC content was produced. In the next step, the chemical properties (acidity, pH, moisture, and AC), total phenolic content, and antioxidant capacity of the BPP candy were determined. Producing candy with encapsulated antioxidant compounds can result in a new functional product, and by making optimal use of plum processing waste, it could gain a favorable position in the industry in terms of exports and contribute to the growth of the agricultural economy.

Materials and Methods

Raw materials

BPP was obtained from Neyshabur, located in Razavi Khorasan province, and stored in a freezer at -18 °C until use. Maltodextrin (with DE = 4 - 7), starch, pectin, and gelatin were sourced from Sahel Borj Company in Neyshabur. Citric acid was purchased from Jovin Sabzevar Company. Salt, oil, tartaric acid, glucose syrup, and sugar were bought from local supermarkets.

Preparation of BPP for microencapsulation process

First, the BPP was dried at three temperatures: 50 °C, 60 °C, and 70 °C. The extraction of plum peel was done by dissolving the dried peel in a 5% citric acid solution and placing it on a shaker (ROTOMIX BEHDAD, Model 101 - Iran) for 2 hr. The solution was then subjected to ultrasonic treatment for 15 min using an ultrasonic bath, followed by filtration and centrifugation at 6000 rpm for 15 min (EBA 200 ILETTICH, AR1800EN, Germany). To study the effects of pH changes on antioxidant compounds and total phenolic content of the core, pH levels of 2, 4, and 6 were used.

Microencapsulation

For microencapsulation, a combination of maltodextrin with starch, gelatin, and pectin in a ratio of 1:3 was used as the wall material. To form microcapsules, two phases organic and aqueous were prepared. To prepare the organic phase, a specific amount of maltodextrin was placed in a beaker, mixed with a magnetic stirrer, and then a few milliliters of the plum extract were added. If insoluble particles were observed, a short ultrasonic homogenization was performed using an ultrasonic bath (SONICOR - model TS - 901/16/6HC, USA). For the aqueous phase, specific amounts of pectin, starch, and gelatin (1.5%) were separately dissolved in 15 ml of distilled water and stirred with a magnetic stirrer for 1 hr until completely dissolved. The organic phase was slowly added drop wise to the aqueous phase while stirring rapidly with a magnetic stirrer. The sample was then placed in an ultrasonic bath for a specific time [2]. Finally, the encapsulated extract was used to produce hard candy.

Hard candy production

For the production of hard candy, glucose syrup, sugar, and water were mixed and heated. When the temperature reached 116 ˚C, tartaric acid and salt were added, and after 3 min, a small amount of oil was added, followed by further heating. The Brix level was adjusted between 68 and 75 by adding water. In the next step, the mixture was removed from the heat, and the encapsulated extract was added. Before cooling, the mixture was poured into molds [11]. The formulation of the produced candy was as follows: 0.5% salt, 40% sugar, 40% glucose syrup, 10% plum peel extract, 8.5% water, and 1% tartaric acid. A control sample of hard candy without encapsulated extract was also produced. Figure 1 shows images of BP, BPP, and the produced hard candy.

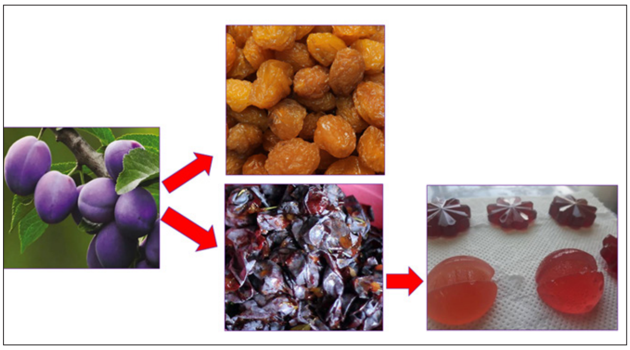

Figure 1: BP, BPP, and the produced hard candy.

Tests

PH

PH was measured using a pH meter (Dana, China).

Acidity

Five grams of each sample were taken, and 100 ml of freshly boiled water was added in a 250 ml beaker. The solution was then transferred to a 250 ml volumetric flask and brought to volume. After homogenizing, the solution was titrated with 0.1 N sodium hydroxide in the presence of phenolphthalein until a stable pink color appeared. The total titration amount was used to calculate the acidity percentage using the following formula [12, 13].

MC

The MC of the samples was measured by oven drying method. An empty porcelain crucible was dried in an oven at 105 ˚C for 30 min, then placed in a desiccator containing silica gel for 15 min and weighed to a constant weight. Two grams of hard candy (W1) were placed in the crucible, dried in an oven for 6 hr at 100 - 102 ˚C, and weighed until constant weight (W2) was reached. Additionally, the sample was placed in a decorator for 15 min to cool at room temperature and was then weighed [12].

$$MC\ (\%) = \frac{xW_1 - W_2}{W_1} \times 100$$

AC

AC refers to inorganic residue left after the complete combustion of organic matter in a food sample. It presents the total mineral content of the food. Measurement of AC is important in assessing the quality and nutritional value of food products. Steps for measuring assets are as follows:

- Weighing the sample: To this purpose, a known weight of sample took (W1).
- Burning the sample: The sample placed in a pre-weighted, dry crucible. After that, ignite the sample in a muffle furnace at 500 - 600 ˚C for 4 - 6 hr, or until a white or gray ash is obtained 9 indicating complete combustion of the organic material.
- Cooling and weighing: After the combustion process was completed, the crucible removed and let it cool in a desiccator. Then, the crucible weighed with the ash residue (W2).
- AC calculation: The AC was calculated using the following formula:

$$AC\ (\%) = \frac{W_1 - W_2}{W_1} \times 100$$

Determination of antioxidant capacity

The percentage of free radical scavenging was used as a measure of antioxidant activity of the extracted samples using the DPPH method. The basis of this method is the ability of the samples to reduce free radicals. The stable organic radical DPPH has a deep purple color, which diminishes as it is reduced. APPH solution was prepared at 1M concentration. Three milliliters of the DPPH solution and 100 µl of the extracted sample were mixed in a sample container. For the control solution, the extraction solvent (Ethanol) was added instead of the sample. After 60 min of reaction in the dark, the absorbance at 517 nm was measured using a spectrophotometer, and the percentage of DPPH scavenging by the samples was calculated using equation (1) [14].

$$\%\ Inhibition = \left(\frac{Ac - As}{Ac}\right) \times 100 \qquad (1)$$

Where: Ac = Control sample solution absorbance; and As = Sample solution absorbance.

Total phenolic content measurement

Total phenolic content was measured using the Folin-Ciocalteu reagent (FC). The method involved adding 20 µl of FC reagent to 2 µl of the extracted sample, which was mixed with 1.58 ml of distilled water. After 5 min, 300 µl of saturated sodium carbonate was added, and the mixture was well stirred. After 1 hr, the absorbance of the sample was recorded at 765 nm. Total phenolic content was calculated using a GA calibration curve. Absorbance was measured using a UV-Vis Spectrophotometer (JENWAY 7315, SA 5039-Australia) [12, 14].

Statistical analysis

Data was analyzed using a completely randomized design with a factorial arrangement. Chemical tests and determination of antioxidant compounds were conducted in two replications. Calculations were performed using Excel software. For variance analysis, Minitab software was used. Mean comparisons were performed using Turkey's test at a 5% significance level. Graphs were drawn using Graph Pad Prism 6 software.

Results and Discussion

TPC and antioxidant capacity of the coating

Statistical analysis showed that increasing the percentage and type of hydrocolloids, as well as the amount of dextrin, had a significant effect on the antioxidant capacity and PC of the microencapsulated coating [15]. As seen in figure 2, increasing the percentage of hydrocolloids increased the antioxidant capacity and PC of the samples, while increasing the dextrin percentage led to a reduction in both antioxidant capacity and PC [16]. Increasing the hydrocolloid percentage appears to enhance the preservation of phenolic and antioxidant compounds. Additionally, it was observed that there was no significant difference in the antioxidant compounds of the coating containing pectin, starch, and gelatin. However, figure 2 shows that there was no significant difference between the TPC of pectin and gelatin, but the number of PC in the coating containing starch was higher than the others. The antioxidant capacity and PC of the samples ranged from 40.75% - 4.48% and mg GAE 100/g FW 53.46 - 6.92, respectively. Based on the results, the coating containing 25% dextrin and 1.5% of each hydrocolloid was selected for final microencapsulation.

This result was similar to other researchers' findings. Kramer [17] studied the effect of starch-based edible films and coatings in protection of antioxidant compounds. His research showed coated candies could protect antioxidants. Antioxidant compounds amount in the coated candies had not changed after process [17]. Klinjapo and Krasaekoopt [15] studied microencapsulation of color and flavor in confectionary products. Their research demonstrated one of the most effective methods for preserving color and flavor in hard candies and other confectioneries [15]. Rizk et al. [18] studied microencapsulation of calendula carotenoid pigment and its antioxidant capacity before and after the hard candy production. They found that microencapsulation helped the antioxidant protection during the hard candy production process [18].

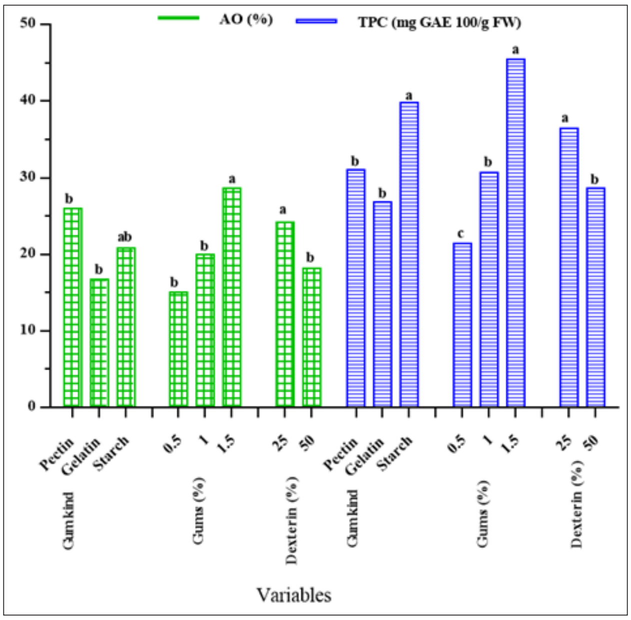

Figure 2: Dextrin and different hydrocolloids effects on antioxidant capacity and PC of microencapsulated cover.

Antioxidant capacity and PC of the core

Figure 3 shows the effect of increasing temperature and pH on the antioxidant capacity and PC of the core. As can be seen, increasing the temperature did not significantly affect the antioxidant capacity or TPC. However, increasing the pH led to an increase in antioxidant compounds and a decrease in TPC. Based on the results, the antioxidant capacity and PC of the core ranged from 88.50% - 74.33% and mg GAE 100/g FW 873.55 - 666.54, respectively.

Although an increase in pH led to higher antioxidant capacity, considering that sodium hydroxide was used to increase the pH and due to the acidic nature and low pH of BPP extract, a pH of 2 was chosen for candy production. Furthermore, given the lack of significant effects of temperature and pH on antioxidant properties and PC, a temperature of 50 °C was used for candy production. Charoen et al. [10] and Kanthain et al. [19] in their study on the effect of crude *P. guajava* extract for jelly candy production, used a temperature of 50 °C and a pH of 4 as the conditions with the highest antioxidant properties for candy production.

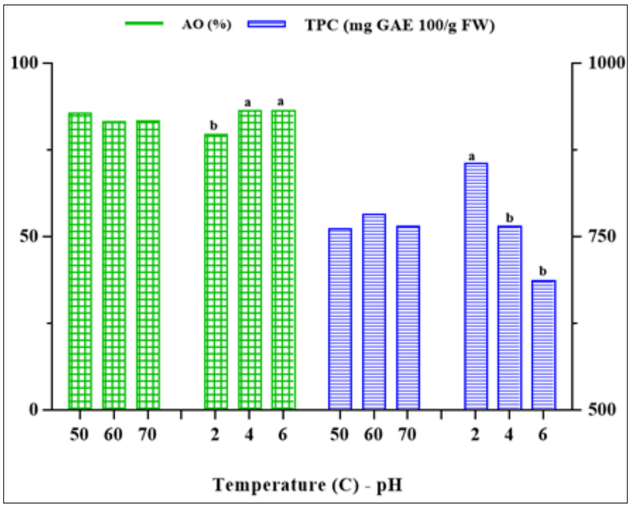

Figure 3: Temperature and pH effects on antioxidant capacity and core PC.

Kanthain et al. [19] studied formulation of hard candy containing spray-dried Veronia Cinerea extract. They used spectrophotometry for measuring DPPH like this study and resulted TPC were significantly decreased after the process [19].

Chemical properties of hard candy

Based on the results, the hard candy was produced with 25% dextrin and 1.5% of each hydrocolloid at 50 °C and a pH of 2. Figure 3 shows the chemical properties, antioxidant capacity, and PC of the hard candy produced under optimal conditions. As can be seen, there were no significant differences in MC, ash, antioxidant capacity, and PC between the different hydrocolloid-containing samples.

In terms of acidity, there was a significant difference between the samples. The highest acidity was observed in the sample without hydrocolloids, and the lowest acidity was found in the sample containing pectin. The lack of hydrocolloids likely led to the absence of gel network formation and the failure to encapsulate acidic compounds, resulting in higher acidity in the sample without hydrocolloids.

Encapsulation represents a class of delivery systems that can provide powerful impact. This method can protect flavors, antioxidants, antimicrobials, and even nutraceutical ingredients [20]. In this study encapsulation caused lower acidity and the result was similar to Lakkis [20] discovery.

Solano et al. [21] used encapsulation in hard candy production and monitored color and anthocyanin degradation during storage. They resulted in encapsulated hard candies; their colors had not changed significantly during storage [21]. This study result was similar.

Conclusion

In this study, the effect of the microencapsulation process on the production of candy, while preserving antioxidant compounds and total phenolic content, was examined. By increasing the percentage of hydrocolloids, the antioxidant capacity and PC of the coating increased, while an increase in dextrin percentage resulted in a decrease in these properties. The number of TPC in the starch-containing coating was higher than in other coatings. Increasing the pH resulted in higher antioxidant compounds but lower TPC. Based on the results, the optimal conditions for producing hard candy were determined to be 25% dextrin and 1.5% of each hydrocolloid at a temperature of 50 °C and a pH of 2. No significant differences were observed between the different hard candy samples in terms of MC, ash, antioxidant compounds, and PC. The acidity of the sample without hydrocolloids was higher than the others.

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Conflict of Interest

None.

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