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Bilayer edible coating of figs (*Ficus carica*) using gelatin and guar gum incorporated with grapefruit seed extract

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Abstract

Figs (*Ficus carica*) is a nutritious fruit that has perishable behaviour during storage period. The application of edible coating could be an alternative to preserve the quality and extend the fruit's shelf life. However, there is currently a lack of knowledge regarding the use of post-harvest technology to increase the shelf life of fig fruits. Therefore, in the present study the effect of four different coatings, namely Gelatin, Guar gum, Guar gum – Gelatin bilayer coating and Gelatin – Guar gum bilayer coating incorporated with Grapefruit seed extract (GSE) on the post-harvest quality of fig fruits were studied. Fig fruits dripped in distilled water was used as control. The changes in weight loss, pH, total soluble solids (TSS), and titratable acidity (TA), firmness and total polyphenol contents of fig fruits stored at 4 °C for 15 days were monitored. The findings indicated that gelatin – guar gum bilayer coating incorporated with GSE had a more potent preservation effect than other coatings. In comparison to other treatments, figs coated fruits with Gelatin – Guar gum bilayer coating showed significantly (p<0.05) less weight loss, pH, TSS, and TSS/TA ratio. Furthermore, compared to other treatments, figs coated with Gelatin – Guar gum bilayer coating showed noticeably higher firmness, TA, and polyphenol content. Therefore, the bilayer coating made of Gelatin – Guar gum incorporated with GSE can act as an efficient edible coating for maintaining the post-harvest shelf life of fig fruits.

Keywords: Bilayer edible packaging, food grade polymers, food spoilage, shelf-life, active coating

1. Introduction

The fig (*Ficus carica*) fruit is well-known for its taste and several health benefits. The fruit contains large number of antioxidants and vitamins, which makes them to eliminate various diseases. Furthermore, figs are a good source of fatty acids, fibre, amino acids, and minerals including calcium, potassium, iron, phosphorus, manganese and so on (Ghosh & Dash, 2020)^[11]. Fresh figs have a short postharvest shelf life due to its sensitive epidermal tissue and also due to the growth of yeast and mould. The ostiole, is a small opening in the fresh fig fruit, that allows small insects to enter and promotes the growth of a variety of microorganisms (Waghmare & Annapure, 2018)^[44]. One of the greatest ways to solve this problem is the usage of edible coating which has the ability to preserve the fruit against microbial contamination and to increase the shelf life of the fruit (Kumar & Neeraj, 2019)^[21]. Pectin, starch, alginate, guar gum, and xanthan gum have all been employed as biopolymer materials in the development edible coatings (Mohamed *et al.*, 2020)^[27]. Their primary role is to protect food items from its surroundings, prevent them from spoiling agents such as microbes, water vapour, and oxygen, and preventing the loss of beneficial nutrients, thereby extending their shelf life and quality (Otoni *et al.*, 2017)^[30].

Gelatine is a highly refined protein obtained by the hydrolysis of high-molecular-weight collagen. Collagen is a type of connective tissue found in abundance in the skin and bones of cattle, pig, and fish. Because of its unique structural stability and other physiochemical properties, gelatine can be used for food packaging as binding, coating, stabilising, and glazing agents (Rohman *et al.*, 2020) ^[36]. Results of different studies proved that the application of gelatine reduces the respiration rate and delays the degradation process in pomegranate arils (Kahramanoglu, 2021) ^[16], blueberry (Piechowiak *et al.*, 2022) ^[32] and jack fruit (Rahman *et al.*, 2020) ^[33].

Guar gum is a high molecular weight polysaccharide obtained from the guar plant and has a white to yellowish white colour appearance and is odourless. Guar gum-based films are known for their great mechanical strength, improved barrier qualities, and antibacterial resistance (Sharma *et al.*, 2018)^[40].

It is frequently used to extend the shelf life of various fruits and vegetables. Several studies reported the use of guar gum reduced weight loss, firmness, and respiration rate in mango (Hmmam *et al.*, 2021)^[14], tomatoes (Naeem *et al.*, 2018)^[28] and kiwi fruit (Barman *et al.*, 2021)^[6].

A wide variety of natural extracts and essential oils could be used to enrich the edible coating in order to enhance their properties (Zam, 2019) [45]. Gelatine enriched with Mentha pulegium essential oil was found to be a good combination to extent the shelf of strawberries (Aitboulahsen et al., 2018)^[3]. Grapefruit seed extract (GSE) made from the seeds of grape fruit is a natural antibacterial agent that has been shown to successfully reduce a variety of bacteria that are responsible for causing food poisoning. It contains polyphenolic compounds, tocopherols, citric acid, ascorbic acid, and other antioxidant chemicals (Kim et al., 2021) [19]. Antioxidant activity found in grapefruit seed extract, is reported to be useful for food preservation and disease prevention (Deng et al., 2020)^[8]. As a result, incorporating grape fruit seed extract into edible coating materials could potentially improve the quality and shelf life of fig fruit. Several prior studies have used different polymers, such as guar gum and gelatine, to extend the shelf life of fig fruit. To our knowledge, there is no report on the use of guar gum and gelatine incorporated with grape fruit seed extract on fig fruit to improve its physical state during storage. The main goal of this study is to see how well gelatin, guar gum, and grape fruit seed extract worked as an active edible coating for Fig (Ficus Carica).

2. Materials and Methods

2.1 Plant material and Experimental design

Figs (*Ficus carica*) where brought from the local fruit market of Chennai city, Tamil Nadu, India. Fruits were selected at commercial maturity stage, free from any disease or physical injuries. They were sorted for uniform size, shape, and colour. Deselected fruits were washed with distilled water and dipped in 1% sodium hypochlorite for 10 minutes and then air-dried.

2.2 Preparation of edible coating material

To prepare gelatin coating solution, 20 g of gelatin powder (Sigma - Aldrich) were dissolved in 1 L of distilled water followed by the addition of 25 ml of glycerol (Sigma -Aldrich). The mixture was stirred at 60 °C for 30 minutes on a magnetic stirrer/hot plate and then filtered (Abdallah et al., 2018) ^[1]. The grapefruit seed extract (Vedaoils, New Delhi, India) was added and stirred thoroughly to obtain a final concentration of 0.5%. Fresh figs were dipped in the gelatin solution for 2 minutes and immersed in 3% calcium chloride aqueous solution (Sigma - Aldrich) for 2 minutes for gelation of the antimicrobial extract on the surface of the fig fruit by cross linking (Kim et al., 2018)^[18]. The coated figs were then air dried at room temperature for 5 minutes. To prepare guar gum coating solution, 1.5% (w/v) guar gum (Sigma -Aldrich) was dissolved in 100 ml of distilled water and 30% (v/v) of glycerol was added. The solution was then stirred at 800 rpm at 60 °C for 30 minutes (Ruelas Chacon et al., 2017) ^[37]. After stirring the GSE was added at a final concentration of 0.5%. To prepare guar gum – gelatin bilayer coating, figs were first soaked in guar gum solution for 2 minutes and later soaked in gelatin solution with CaCl₂ for 2 minutes. Similarly for gelatin-guar gum bilayer coating, fig fruits were first dipped in gelatin solution followed by guar gum solution. All the fruits were immersed in the prepared treatments for 2 minutes to assure uniform coating of the whole surface of the

fig fruits and the control group figs were immersed in distilled water. All the coated fruits were dried under air flow heater at 25 °C for 30 minutes, packed in plastic boxes and stored at 4 °C with $75\pm5\%$ relative humidity. Ten fruits were analysed in triplicate at an interval of 3 days up to 15 days, as the fruits become unacceptable for consumption due to decay or infection.

2.3 Weight loss

The weight loss was determined according to the method described by (Sipahi *et al.*, 2013)^[41]. The weight of all the treatment groups were determined using a Kerro Digital Weighing balance (BL-6002E, India). The weight loss percentage were calculated using the formula,

$$Percentage of weight loss = \frac{Initial weight of figs - Final weight of figs}{Initial weight of figs} \times 100$$

2.4 pH

The pH of the figs was measured using a digital pH meter (Labtronics LT-10, India). 50 g of fig was blended with 150 ml of distilled water for 2 minutes and the obtained mixture was used to measure the pH of fig fruits (Alharaty Ramaswamy, 2020)^[4].

2.5 Total Soluble Solids

Total Soluble Solids (TSS) was determined using a refractometer Erma hand refractometer (0 – 32^{0} Brix). The juice extract from each fig sample was used for the estimation of TSS and the results were expressed by means of% (°Brix) (Saki *et al.*, 2019) ^[38].

2.6 Titratable acidity

Titratable acidity was estimated according to the method suggested by (Saleem *et al.*, 2021)^[39]. 10 ml of fig juice extract was mixed with 40 ml of distilled water and titrated against 0.1 N NaOH with phenolphthalein as indicator. The titratable acidity of fig fruit samples was calculated using the following formula,

Titratable acidity (%) =
$$\frac{\text{Quantity of } 0.1 \text{ N NaOH used } \times 0.0064}{\text{Quantity of fig juice extract}} \times 100$$

Ripening index was calculated by taking the ratio of TSS to TA.

2.7 Firmness

The firmness of fig fruits was determined using a digital fruit penetrometer (Parissa Technology, India). The tip of the penetrometer was inserted into 3 random places of the fig fruits and the readings were recorded. The obtained results were expressed in Kg cm⁻² (Saki *et al.*, 2019)^[38].

2.8 Total phenolic content

Phenolic compounds in fig fruits were extracted and measured as described by (Zidi *et al.*, 2020) ^[46] with slight modifications. 200 ml of fig juice extract were added to 10 ml of aqueous acetone and the mixture was kept in a shaker bath (IG - 12HL, iGene Labserve, India) at 40 °C for 2 h. Then the extract was centrifuged at 1700 rpm for 10 min. 200 μ l of the extract was mixed with 1000 μ l of Folin - Ciocalteu reagent and 800 μ l of 7.5% sodium carbonate. The final mixture was incubated for 30 min in dark and the absorbance was read at 760 nm using UV Vis spectrophotometer (LT-291, LABTRONICS, India). The obtained results were calculated using gallic acid curve as standard and the total phenolic content was determined on the basis of mg of gallic acid equivalent per 100 g of fresh weight of figs.

2.9 Statistical Analysis

Statistical data was analysed using SPSS 28.0 Software. All the obtained data were composed of three replicated (n=3) and the results were performed in a completely randomised design. The statistical data was analysed using analysis of variance (ANOVA). The least significant difference (p < 0.05) was used to compare means among different coatings and storage periods. The mean values were calculated and reported as mean \pm standard deviation.

3. Results

3.1 Weight Loss

The important factor affecting the shelf life of fruits is the moisture loss, and the primary mechanism causing weight loss in fruits is due to the difference in water vapour pressure between the environment and the fruit surface (Suseno *et al.*, 2014)^[42]. Respiration is also another factor that contributes to weight loss from fresh fruits. The decrease in weight loss can be minimized by the action of coating layers which acts as a semi-permeable barrier against the flow of O₂, CO₂, moisture,

and solute movements, thereby decreasing the rates of respiration, water loss, and oxidation reaction (Al Juhaimi et al., 2012) ^[5]. The weight loss of figs in all the treatment groups increased with increasing storage period (Fig 1). However coating treatment groups showed significant impact on the weight reduction percentage, and the control groups showed maximum weight loss. After 15 d of cold storage of fig fruits highest weight loss (18.67%) was observed in control group and minimum weight loss (8.89%) was observed for Gelatin - Guar gum bilayer coated treatment group, which was most effective in reducing the weight loss of fig fruits. Furthermore, there was significant difference (p <0.05) between Gelatin – Guar gum coated fruits and other treatment at the end of storage period. Our results suggests that the combination of Gelatin – Guar gum bilayer coating incorporated with Grape fruit seed extract gives much better effect in preventing weight loss than sole Gelatin, Guar gum or Guar gum - Gelatin bilayer coating. These results are in agreement with the findings of Huang et al., (2019) ^[15] for the quality of shiitake mushroom (Lentinus edodes) with either eco - friendly chitosan and guar gum edible coating which extended the storage period of mushroom by reducing the water loss.

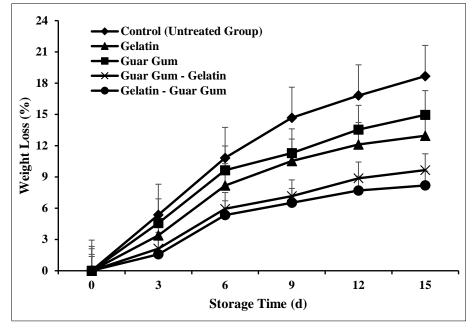


Fig 1: Effect of different coatings on the weight loss of fresh fig fruits during 15 days of storage at 4 °C. Vertical bars indicate standard deviation

3.2 pH

The change in pH of fruits is mainly caused due to the changes in titratable acidity and metabolic activities that convert them to sugars during storage. The edible coating on the fruits tends to slow down the respiration rate and metabolic activities of fruits so the change in pH value can be minimized (Ebrahimi Rastegar, 2020)^[9]. The effect of different coatings on the pH of fresh fig fruits is given in Fig 2. It is revealed from the results that increase in storage period had increased the pH of fig fruits in all the treatment groups. A Significant difference (p<0.05) was observed between Control, Gelatin, Guar gum, Gelatin – Guar gum and Guar gum – Gelatin treatment groups. The pH of figs used in the

study had an average value of 4.60 at day 0. The control group figs showed an increase in pH, rising from 4.60 to 6.38 on the 15th day of cold storage. Gelatin and guar gum coated figs showed slight increase in pH. At the end of 15 d figs coated with Gelatin – Guar gum and Guar gum – Gelatin bilayer coating showed pH value similar to day 0. This increase in pH is caused due respiration process where organic acids act as a substrate for enzymatic reactions of respiration for reducing acids to sugars. So, this reduction in acidity had led to the increase in pH of fig fruits (Lakshmi *et al.*, 2018) ^[24]. These results are in agreement with the findings of Mantilla *et al.*, (2013) ^[25] in fresh cut pine apple and Kumar *et al.*, (2021) ^[22] in mango.

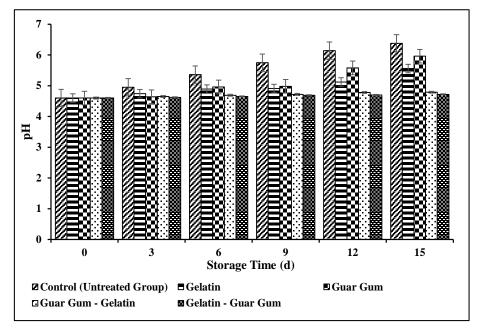


Fig 2: Effect of different coatings on the pH of fresh fig fruits during 15 days of storage at 4 °C. Vertical bars indicate standard deviation.

3.3 Total Soluble Solids

The primary cause for the increase in Total Soluble Solids (TSS) concentration during fruit ripening is mainly due to the starch hydrolysis, which releases a significant amount of glucose, fructose, and sucrose molecules. Later, the TSS level is steadily decreased, which may have been caused due to the decrease in the amount of pectin and carbs, partial protein hydrolysis, and the breakdown of glycosides into subunits during respiration (Hernandez Guerrero *et al.*, 2020) ^[13]. The effect of different coatings on the TSS of fresh fig fruits is given in Fig 3. In this study, a significant difference (p < 0.05)

was observed between Gelatin – Guar gum coated fruits and other treatment groups. The TSS of fig fruits increased with increase in storage time. After 15 d of cold storage, the highest value (17.83 °Brix) was recorded in control group and the lowest value (13.98 °Brix) of TSS was recorded in Gelatin – Guar gum bilayer coated group respectively. Similarly, Kumar *et al.*, (2020) ^[20] reported that the use of edible coating could be useful for reducing the rate of TSS in fruits and vegetables. The findings showed that the edible coating made of chitosan and pullulan maintained the TSS of litchi fruits under both room temperature and cold conditions.

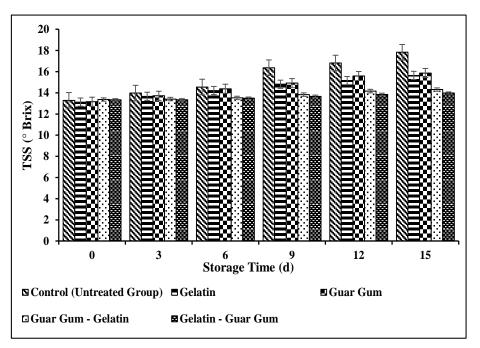


Fig 3: Effect of different coatings on the TSS of fresh fig fruits during 15 days of storage at 4 °C. Vertical bars indicate standard deviation.

3.4 Titratable acidity

The organic acids in fig fruits have a significant impact on their flavour. During the storage of fig fruits the acidity reduces due the respiration process where the organic acids are consumed (Riaz *et al.*, 2021) ^[34]. The effect of different coatings on the TA of fresh fig fruits is given in Fig 4. After

15 d of cold storage, the lowest level of TA (0.24%) was recorded in control fruits and the highest level (0.33%) was recorded in gelatin – guar gum bilayer coated group. The level of TA decreased slowly in coated fruits when compared to the control group. The gelatin – guar gum bilayer coated group showed significant difference (p < 0.05) when

compared to other treatment groups. Similar results were reported by Tokath Demirdoven, (2020)^[43] for sweet cherries

with chitosan edible coating and by La *et al.*, (2021) ^[23] for banana with chitosan-gum arabic and ZO Np edible coating.

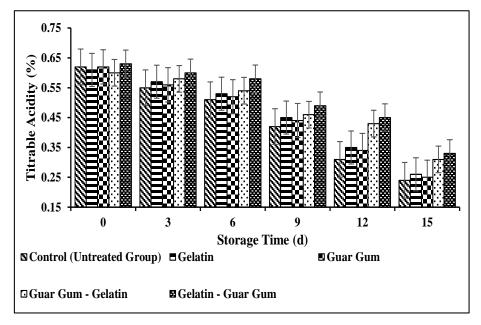


Fig 4: Effect of different coatings on the TA of fresh fig fruits during 15 days of storage at 4 °C. Vertical bars indicate standard deviation.

3.5 TSS/TA Ratio

TSS/TA ratio is an important parameter which determines the quality, flavor and consumer acceptability of fruits. An increase in the TSS/TA value, increases the sweetness and decreases the sourness of fruits. Organic acids degrade more quickly during storage than sugars, hence it is stated that the fruit would become slightly sweeter during storage period (Khorram *et al.*, 2017) ^[17]. The TSS/TA ratio of fig fruits is shown in Fig. 5. Significant difference (p < 0.05) between Gelatin – Guar gum coated fruits and other treatment was observed at the end of storage period. The TSS/TA ratio was found to be increased in all the treatment groups at the end of 15 d cold storage. The highest value of TSS/TA ratio (74.29%) was found in control group fruits.

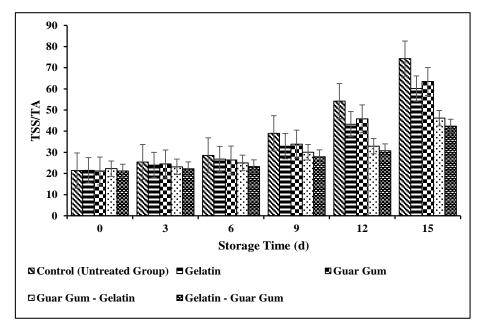


Fig 5: Effect of different coatings on the TSS/TA ratio of fresh fig fruits during 15 days of storage at 4 °C. Vertical bars indicate standard deviation.

3.6 Firmness

Firmness is the main quality trait for any fruits to be accepted by consumers. It is mainly dependant on the biochemical elements, cellular organelles, fruit moisture content, and fruit cell wall composition. Any external or internal factor impacting these characters during the storage period could alter the texture and result in unfavourable changes in the product's quality. The loss of firmness after storage is the most obvious textural change in fruits and vegetables (Nawab *et al.*, 2017)^[29]. Edible coating of fruits acts as a preventative measure against enzymes that degrade cell walls. It is stated that due to the reduction in oxygen exchange of the fruits, ethylene production is delayed, so the physical loss water through the pores of fruits is reduced (Hazrati *et al.*, 2017)^[12].

The firmness of fig fruits is shown in Fig. 6. The firmness of fruits in all the treatment groups decreased throughout the storage period. A Significant difference (p<0.05) was observed between Control, Gelatin, Guar gum, Gelatin – Guar gum and Guar gum – Gelatin treatment groups. Firmness of fig fruits was significantly affected by coating treatments and the higher value was found in control group fruits. After 15 d of cold storage, the control group fruits showed least firmness value (1.32 Kg cm⁻²) and the Gelatin – Guar gum incorporated with GSE showed firmness value 2.52 Kg cm⁻² which was the

most effective among all the groups. Furthermore, there was a significant difference among Gelatin – Guar gum coated fruits and other coated fruits at the end of storage period. Additionally, less water loss could be observed from the all the coated fig fruits which results in better-quality fruits. These results were in agreement with the findings of Dave *et al.*, (2017) ^[7] for pear fruit coated with soy protein and those of El Gioushy *et al.*, (2022) ^[10] who reported that Gum Arabic based edible coating retained the firmness and texture of guava than control group fruit.

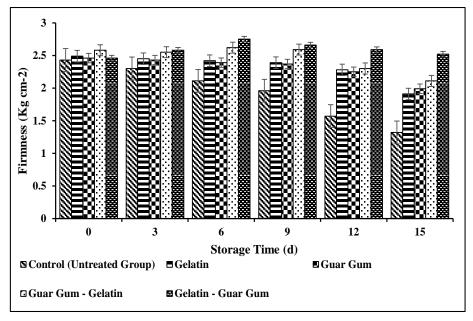


Fig 6: Effect of different coatings on the fruit firmness of fresh fig fruits during 15 days of storage at 4 °C. Vertical bars indicate standard deviation.

3.7 Total Phenolic Content

Fresh fruits and vegetables are highly perishable, and the majority of its losses come from postharvest deterioration and water loss. Some changes in secondary metabolism takes place during the postharvest period and during various technological processes that are used to increase their shelf life. The breakdown of cell structure causing senescence during storage may be the reason for the decrease in phenolic compound levels. To prevent such losses and to increase the shelf life, edible coating techniques can be used (Paolucci et al., 2020) [31]. Fruits and vegetables have high amounts of phenolic compounds, which are known to support health. Fresh fig fruits contain more phenols in the peel than in the pulp, and the amount depends greatly on the genotypes and cultivars of the figs. The phenol concentration and nutraceutical composition of fig fruits can be significantly influenced by postharvest treatments (Adiletta et al., 2019)^[2]. The total phenolic content of fig fruits kept in different coatings at 4 °C is given in Fig. 7. In our study, the total

phenolic content of fig fruits showed significant changes during the 15 d storage at 4 °C. Among all the treatment groups, the control group showed decreased polyphenol content and gelatin - guar gum coated group showed increased polyphenol content. A Significant difference (p<0.05) was shown after 15 d between untreated control group, Gelatin coated group, Guar gum coated group, Guar gum – Gelatin coated group and Gelatin – Guar gum bilayer coated treatment groups. From the obtained results it is clear that the bilayer edible coating was able to enhance the phenolic acid contents in fig fruits. The increase in polyphenols in gelatin – guar gum and guar gum – gelatin bilayer coating incorporated with GSE was probably due to the gradual release of polyphenols from the bilayer polymer matrix. Previous studies also state that decrease in polyphenol contents can be reduced by the usage of active edible coating made up of alginic acid and agar for fresh figs (Moccia et al., 2021) ^[26] and alginate for fresh cut Kent mangoes (Robles Sanchez et al., 2013) [35].

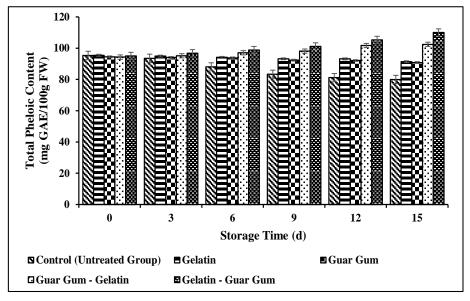


Fig 7: Changes in total polyphenol contents (mg Gallic acid equivalent (GAE)/100 g fresh weight (FW) during 15 d of cold storage with different coating groups

4. Conclusion

In this study, gelatin, guar gum, guar gum - gelatin bilayer coating, and gelatin-guar gum bilayer coating combined with GSE was used to keep fig fruits fresh during storage at 4 °C. The findings demonstrated that, the gelatin-guar gum bilayer coating on fig fruits had beneficial effects on weight loss, pH, TSS, TA, TSS/TA ratio, firmness and polyphenol content when compared to other coated groups. Therefore gelatin-guar gum bilayer coating incorporated with GSE can be used as a unique edible coating for commercial purposes to enhance the quality of figs during long-term storage. Additional research is required to determine whether the combination of gelatin, guar gum, and grapefruit seed extract can increase the postharvest shelf life of other fruits and vegetables.

5. Acknowledgment

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6. Conflict of Interest

The authors certify that they have no conflict of interest exist in the subject matter or materials discussed in this manuscript.

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