Jurnal
Teknologi

Full Paper

ASSESSMENT OF CHITOSAN-BASED COATING FORMULATION ON PAPAYA FRUIT PRESERVATION AND ITS WEIGHT LOSS KINETICS DURING STORAGE

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Article history
Received
21 August 2024
Received in revised form
12 February 2025
Accepted
24 February 2025
Published Online
24 October 2025

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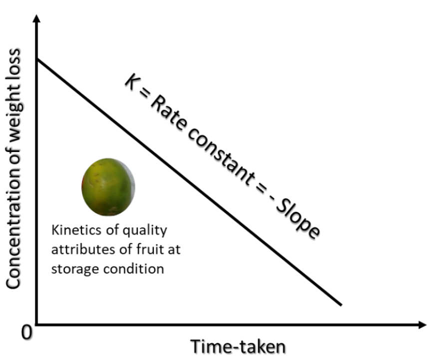
Graphical abstract



Abstract

The considerable postharvest losses of papaya during storage, compounded by inadequate postharvest management practices, are alarming in the context of escalating global food insecurity, malnutrition, and environmental impact. Addressing this issue necessitates the development of sustainable preservation strategies. Notably, chitosan-based coating emulsions have exhibited efficacy in preserving the overall quality parameters of papaya, mitigating weight loss when active components are judiciously formulated. The ionic gelation technique was used to obtain the nanoparticles and subsequently, the coating emulsion was formulated by solvent blending. The emulsion coating was applied to the papaya by dipping while the concentration variant impact of acetic acid, Tween 80, and chitosan molecular weight on the weight loss of coated papaya fruits was systematically investigated during an 8-day room temperature storage period. Optimal concentrations were identified, suggesting suitability for acetic acid at 0.6% to 1.0%, Tween 80 at 0.3% to 0.6%, and a preference for 0.5% low molecular weight (LMW)-chitosan. The coating solutions were characterized by using an FTIR spectrometer and a master sizer. Furthermore, kinetic regression analysis revealed adherence to zero-order kinetics, enabling the formulation of predictive models for different samples and the control group. Empirically and statistically, the sample comprising 0.5% LMW-Chitosan, 1.0% Acetic acid, 0.6% Tween 80, 0.3% sodium tripolyphosphate (NaTPP), and 0.3% ginger oil emerged as particularly suitable for preserving the overall quality parameters of papaya during storage. In addition, the optimal proposed kinetic model for the coated papaya, $[A]f = -0.5273t + [A]_0$, for predicting weight loss decreased with time following the zero-order reaction kinetics.

Keywords: Carica papaya, Chitosan, Weight loss, Kinetic modeling, Reaction order

Abstrak

Kehilangan besar selepas tuaian betik semasa penyimpanan, ditambah dengan amalan pengurusan lepas tuai yang tidak mencukupi, adalah membimbangkan dalam konteks peningkatan ketidakselamatan makanan global, kekurangan zat makanan, dan kesan alam sekitar. Menangani isu ini memerlukan pembangunan strategi pemeliharaan mampan. Emulsi salutan berasaskan kitosan telah menunjukkan keberkesanan dalam memelihara parameter kualiti keseluruhan betik dan mengurangkan penurunan berat badan apabila komponen aktif dirumus dengan bijak. Teknik gelilasi ionik digunakan untuk mendapatkan zarah nano, dan seterusnya, emulsi salutan dirumuskan dengan pencampuran pelarut. Emulsi salutan ini digunakan pada betik dengan mencelup, dan kesan variasi kepekatan asid asetik, Tween 80, dan berat molekul kitosan ke atas kehilangan berat buah betik bersalut disiasat secara sistematik semasa tempoh penyimpanan suhu bilik selama 8 hari. Kepekatan optimum telah dikenal pasti, mencadangkan kesesuaian untuk asid asetik pada 0.6% hingga 1.0%, Tween 80 pada 0.3% hingga 0.6%, dan keutamaan untuk 0.5% berat molekul rendah (LMW)-kitosan. Penyelesaian salutan telah dianalisa menggunakan spektrometer FTIR dan saiz zarah. Tambahan pula, analisis regresi kinetik menunjukkan pematuhan kepada kinetik tertib sifar, membolehkan perumusan model ramalan untuk sampel yang berbeza dan kumpulan kawalan. Secara empirikal dan statistikal, sampel yang terdiri daripada 0.5% LMW-Kitosan, 1.0% Asid asetik, 0.6% Tween 80, 0.3% sodium tripolyphosphate (NaTPP), dan 0.3% minyak halia menunjukkan hasil yang sangat sesuai untuk mengekalkan parameter kualiti keseluruhan betik semasa penyimpanan. Di samping itu, model kinetik yang dicadangkan untuk meramalkan penurunan berat badan menurun dengan masa mengikuti kinetik tindak balas tertib sifar.

Kata kunci: Carica papaya, Chitosan, Peratusan kehilangan berat, Pemodelan kinetik, Tertib tindak balas

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1.0 INTRODUCTION

Papaya (*Carica papaya*), a climacteric tropical fruit renowned for its nutritional and medicinal attributes, is characterized by a high moisture content of 79.75 ± 0.18% [1]. During ripening, it exhibits a soft, fleshy texture, appealing flavor, taste, and aroma [2]. Notably, Papaya serves as a valuable source of natural antioxidants, including lycopene and pro-vitamin A carotenoids such as β-carotene and β-cryptoxanthin [3]. Lycopene in papaya is associated with immune system enhancement and the amelioration of various human health disorders, spanning hepatic, cancerous, diabetic, cardiac, redox-related, neural, reproductive, inflammatory, skin, and bone conditions [4]. While global papaya production has seen an upward trend, nearly half of it is consistently wasted due to postharvest challenges, such as chilling injury, vulnerabilities, and high shipping costs resulting in environmental impact [5]. In 2022, global papaya exports totaled 375,000 tonnes, while imports reached 351,000 tonnes, resulting in a deficit of 24,000 tonnes and contributing to waste. This situation poses a significant threat to achieving the Sustainable Development Goals (SDGs - 2) of Zero Hunger by 2030, necessitating a decisive and sustainable approach.

Amidst these challenges, scientists have developed various methods to preserve papaya, including refrigeration, synthetic packaging, and modified atmosphere packaging [6,7]. However, the susceptibility of papaya to low temperatures, the accumulation of plastic packaging leading to environmental degradation, and the high cost required for MAP have necessitated the advent of natural biopolymers [8, 9]. Natural biopolymeric method have emerged as a promising technology for fresh fruit preservation due to their eco-friendliness, biocompatibility, biodegradability, affordability, non-toxicity, film-forming ability, and antimicrobial activity [10], [11], [12], [13]. Chitosan, an edible biopolymer derived from polysaccharides through the deacetylation of chitin, sourced from crustacean and canning industry waste, stands out for its biocompatibility. Due to chitosan insolubility in a neutral medium, it dissolved in acetic acid and crosslinked with other compounds like sodium tripolyphosphate (NaTPP) in the presence of surfactants, such as tween 80 to enhance wettability, stability, and composite binding. Some formulations also include fibers as binders and essential oils such as ginger oil as bioactive agents. Recent research endeavors have aimed to address postharvest losses by employing chitosan-based coatings [14], [15], [16], [17] to preserve the intrinsic and extrinsic qualities of papaya, thus extending its shelf life. However, existing studies lack comprehensive comparisons of the significant effects of coating formulation components on weight loss in coated papayas. Moreover, previous research on papaya preservation has insufficiently addressed weight loss kinetics, but rather kinetic modeling for specific attributes other than weight loss [18], [19], [20], [21]. Thus, this empirical study investigated the influence of acetic acid, Tween 80 concentrations, and chitosan molecular weight on the weight loss of coated papaya fruits and subsequently modeled the papaya weight loss kinetics under an 8-day room temperature storage condition.

2.0 METHODOLOGY

Preparation and Characterization of the Chitosan-based Emulsion Film

Chitosan-based nanoparticles were synthesized employing the ionic gelation technique, following the protocol outlined by [16], [22] with slight modifications. Acetic acid solutions with concentrations of 0.2%, 0.6%, and 1.0% v/v were prepared by diluting pure acetic acid assays in deionized water. Low and high-molecular-weight (LMW and HMW) chitosan flakes were then dissolved in the acetic acid solutions, resulting in 0.5% w/v chitosan solutions with continuous agitation overnight. Subsequently, the chitosan solution was sonicated using a DSA300 ultrasonic water bath at 60°C for 10 minutes, and a high frequency. A 0.3% w/v NaTPP solution was prepared by dissolving its powder in deionized water, followed by overnight mechanical agitation, and the resultant solution was filtered through a 0.45 µm syringe. The NaTPP solution was gradually added to the chitosan solutions under continuous stirring to increase the chitosan surface area by forming a network between chitosan backbones and improving its thermomechanical properties -. The pH of the coating was measured and adjusted between 5.7 and 6.1 using 2M NaOH to protect the pericarps of the papayas and ensure chitosan solubility. Additionally, Tween 80, preheated at 60°C for 10 minutes in a water bath, was combined with ginger oil at a constant 0.3% in the coating solutions and homogenized at 8,000 rpm for 15 minutes using a Silverson L5 M-A High Shear Mixer at room temperature. Consequently, six different formulations of chitosan-based coating emulsion were prepared.

The physicochemical analysis involved utilizing Malvern Mastersizer 2000 (MAI1020331+MAL140679, Malvern, UK) to assess uniformity, span, and surface mean area diameter. Additionally, an FTIR Spectrometer (IQLAADGAAGFAHDMAZA, Thermo Scientific™, USA) was employed to examine chemical bonds within the crosslinked components.

Application of Chitosan-based Coating Emulsion on Papaya Fruits

Papayas, weighing between 0.9 and 1.0 kg, were procured in a healthy and ripened state. After rinsing, they were disinfected with 2% v/v sodium hypochlorite, NaOCl. The dipping deposition method involved immersing papaya fruits for 5 minutes, followed by storage at room temperature. Periodic assessments of weight were conducted during the decay period, while total soluble solids (TSS) were analyzed after 8 days using the method described by [23]. Each experimental run involved three sets of papayas, and the entire process was repeated in triplicate.

Weight Loss Determination

The percentage of papaya weight loss was determined using Equation (1), where m = weight of papaya, i = day, and j = sample.

$$\text{Weight loss, } \%m_{ij} = (m_{ij\ original} - m_{ij\ new\ day})/m_{ij\ original} \times 100\% \quad (1)$$

Kinetic Models

The kinetics of papaya weight loss were fitted into two different models using the integral rate law analysis. Thus, the papayas' weight loss was modeled for the 8 days of storage. Therefore,

$$d[A]/dt = -K[A]^n \quad (2)$$

Where K = Rate constant, [A] = % Concentration of Weight loss in a sample at time t, and n = Reaction orders. The equation was used to develop two models based on % concentration for zero and first-order kinetic reaction and their associated half-lives, $t_{1/2}$.

Derivation of the Reaction Orders

The deterioration of food generally could be expressed using zero- and first-order rate laws [24].

$$\text{Zero order, } n = 0, \text{ hence } [A]_f = -Kt + [A]_0 \quad (3)$$

Where $[A]_f$ = final concentration, $[A]_0$ = initial concentration, t = time taken, and K = slope

The half-life of the zero-order model, $t_{1/2} = [A]_0/2K$ (4)

The first-order kinetic model, n = 1, hence $\ln[A]_f = -Kt + \ln[A]_0$ (5)

The half-life of a first-order model, $t_{1/2} = \ln 2/K$ (6)

Thus, the slope K is estimated by plotting % weight loss against time (day) for each model to determine the suitable model that best characterizes the deteriorating rate.

Statistical Analysis

The regression analysis was performed using Microsoft Excel (MS Office Professional Plus 2016) and Minitab® 15.1.20.0 to determine the 'Goodness of Fit' characteristics including coefficient of determination (R^2), sum of squared errors (SSE), and root mean sum of errors (RMSE). The proposed model having the highest R^2 and least RMSE is recommended as the best [17], [19].

3.0 RESULTS AND DISCUSSION

Characterization of Acetic Acid Stock Solution and Chitosan Solutions

The FTIR spectra results exhibited absorbance bands at 3322.05 cm^{-1}, 3319.95 cm^{-1}, and 3309.84 cm^{-1}, corresponding to O – H stretching of hydroxyl groups and strong-broad intermolecular bonding with the carbonyl group C=O in acetic acid at concentrations of 0.2%, 1.0%, and 0.6%, respectively. This observation aligned with the findings by Gofurov S. *et al*. [25], suggesting that the increase is attributed to high molecular interactions between water and acetic acid molecules. The FTIR spectra revealed broad bands in the chitosan solution between 3000 to 3500 cm^{-1}. These bands were associated with N−H stretching overlapping with O−H stretching. Notably, the chitosan solution with LMW and diluted 1.0% acetic acid exhibited the highest wavenumber peak compared to other chitosan solutions. The observation indicates that LMW corresponds to a higher wavenumber and stronger intermolecular attraction resulting from the overlapping of N−H stretching with O−H stretching bonds [26].

Moreover, the chitosan particle sizes in the solutions, ranged from 135 to 1140 μm, showing a consistent and less variable distribution of particle sizes. Notably, chitosan solutions with lower acetic acid concentrations (0.2% and 0.6%) produced larger particles of surface area mean diameter. In comparison. solutions with 1.0% acetic acid concentration produced moderate-sized particles, regardless of molecular weight. This observation aligned with Lukman Hekiem *et al*. [27], who noted that greater acetic acid concentration enhances chitosan solubility. However, the chitosan solution with 1.0% LMW-Chitosan produced the smallest particles, suitable for fruit coating, but with lesser uniformity and span to 1.0% HMW-Chitosan. This finding is consistent with Reijenga *et al*. [26], who suggested that the degree of protonation is proportional to increased acid concentration, thereby enhancing chitosan solubility. Higher acid concentration and lower pKa values usually increase the tendency of the molecule to release a proton (H^+), elucidating the effect of acetic acid concentration in chitosan-based formulation. Regarding chitosan molecular weight, 1.0% LMW-Chitosan exhibited a smaller surface area mean diameter (135.20 μm) to 1.0% HMW-Chitosan (370.78 μm). This observation aligned with the findings of Gonçalves *et al*. [28] and Qin *et al*. [29], who asserted that chitosan solubility decreases with increasing molecular weight and vice versa.

Effect of Chitosan-based Coating Formulation on Overall Papaya Quality Parameters

All coating formulations, ranging from samples 1 to 6, successfully extended the shelf life of the papayas, even with the least efficient one which showed a difference of 3.78% (9.70 minus 5.92) average weight loss compared to the control, as reflected in Table 1. However, the intrinsic properties, particularly total soluble solids, of papayas in samples 4, 5, and 6 were poorly preserved by the coating formulations, exhibiting lower averages than the control. Secondly, the impact of chitosan molecular weight in the coating formulations can be conveniently analyzed by considering samples 5 and 6, keeping other components constant. Table 1 indicates that chitosan solubility decreases with an increase in molecular weight, attributed to the more compacted structure in HMW-chitosan due to the high polymerization degree [30]. Additionally, Table 1 revealed the effects of the surfactant (Tween 80) in coating formulations. Samples 1, 2, 5, and 6, with a progressive concentration of Tween 80, reflected the influence of this surfactant on particle sizes. Increasing Tween 80 concentrations (0%, 0.3%, and 0.6%) resulted in increment of the particle sizes of coating formulations, as indicated by surface area mean diameters as shown in Table 1. . This aligned with the findings of Çakır *et al*. [31], highlighting that an increase in surfactant concentration enhances particle efficiency and stability.

Table 1 Coating formulation and chitosan-based emulsion physicochemical properties

Tmt	Ch	AA	T80	ATSS	AWL	SAMD
1	LMW	1.0	0.0	10.02±0.20	3.53	129
2	LMW	1.0	0.3	9.78±0.08	5.92	154
3	LMW	0.6	0.6	9.72±0.12	5.64	180
4	LMW	0.2	0.6	6.58±0.24	5.14	107
5	LMW	1.0	0.6	6.78±0.17	4.45	158
6	HMW	1.0	0.6	4.88±0.32	4.68	221
C				6.80±0.32	9.70	

Note: Tmt=Treatment, Ch=Chitosan, AA=Acetic acid (%v/v), ATSS=Average total soluble solids, AWL=Average weight loss (%w/w), SAMD=Surface area mean diameter (nm), T80=Tween 80 (%v/v), C=Control, ginger oil=0.3% v/v (constant)

Moreover, the efficiency of each coating formulation in terms of weight loss followed the order 1>5>6>4>3>2 compared with the control. This finding aligned with previous research by Cheng *et al*. [32], emphasizing a linear downward trend in moisture content and weight loss in fruits over storage time due to respiration and transpiration. Furthermore, the study explored the influence of chitosan molecular weight on papaya weight loss, particularly between samples 5 and 6. The results indicated that HMW-

Chitosan contributed to higher papaya weight loss than LMW-Chitosan by an average of 0.23%. This suggests a preference for LMW-Chitosan in papaya preservation. The concentration of acetic acid also played a role in papaya weight loss, as observed in samples 3, 4, and 5, where acetic acid concentrations varied between 0.2% and 1.0%. Sample 4 with 0.2% acetic acid resulted in a 5.14% weight loss, increasing to 0.6% in sample 3, which further elevated weight loss by 0.5%. However, an additional increase in concentration to 1.0% led to a drop in weight loss by 1.19% on average. Again, the impact of Tween 80 on weight loss was examined in samples 1, 2, and 5, where all active components remained constant except for the Tween 80 concentration. A gradual increase in Tween concentration from 0% to 0.3% resulted in a significant average increment in weight loss of 2.39% between samples 1 and 2. However, a further increase in concentration to 0.6% in sample 5 led to a sharp drop in weight loss by 1.47% on average. The findings suggest that a concentration between 0% and 0.3% Tween 80 is suitable for formulations with 1.0% acetic acid and LMW-Chitosan, while a concentration of 0.6% acetic acid would be preferable for better overall quality parameters in papayas [16].

Weight Loss and the Kinetic Reaction Order

The kinetic model regression analysis results for zero and first reaction orders, as summarized in Tables 2–3, provide insights into the "Goodness of Fit" characteristics for each model (Equations 3 and 5).

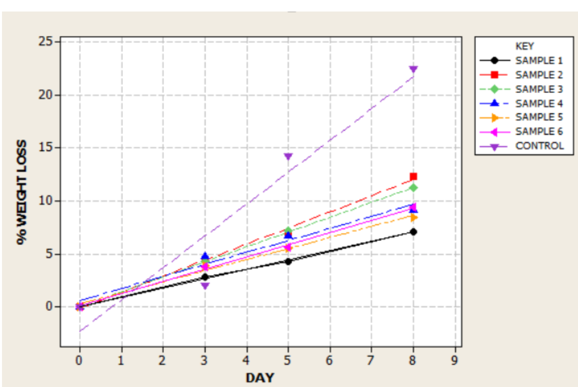

Figure 1 Weight loss of coated papaya during storage conditions

A comparative analysis between the two investigated models indicates that the zero-order kinetic model best describes the weight loss during the 8-day storage period, as shown in Figure 1. The figure presents statistically suitable parameters with the highest R^2 and relatively low Root Mean Square Error (RMSE) [18]. Visual assessments of the kinetic models plotted in Figures 2–3 confirm that the model (Equation 1) consistently fits the kinetic data for all samples. However, an anomaly was observed in the parameters for sample 1 of the zero-order kinetic model regression analysis (Table 2), characterized by an unusually low R^2 and a negative adjusted R^2. This discrepancy could be attributed to the absence of Tween 80 of the chitosan-based coating formulation, resulting in a distant average weight loss compared to other samples. The computed rate constant and half-life for sample 1 further support its outlier status, making the model unreliable. Aside from this, the zero model exhibited the highest R^2 and moderately low RMSE [21]. Thus, the zero-order reaction can best describe the weight loss kinetics in papaya at the storage of both uncoated and coated with chitosan-based emulsion.

Table 2 Results of kinetic model regression analysis for papaya at storage for zero order

Sample	R^2	Adjusted R^2	SSE	RMSE
1	0.0022	- 0.4968	0.0558	0.1671
2	0.9841	0.9761	0.2213	0.3326
3	0.9956	0.9934	0.0417	0.1444
4	0.9446	0.9169	0.1259	0.2509
5	0.9603	0.9405	0.1052	0.2294
6	0.9195	0.8792	0.0793	0.1991
Control	0.9183	0.8774	12.5309	2.5031

Table 3 Results of kinetic model regression analysis for papaya at storage for first-order

Sample	R^2	Adjusted R^2	SSE	RMSE
1	0.0113	- 0.4830	0.0260	0.1141
2	0.0327	- 0.4510	0.0006	0.0174
3	0.0345	- 0.4482	0.0012	0.0242
4	0.0006	- 0.4991	0.0060	0.0547
5	0.0067	- 0.4900	0.0053	0.0513
6	0.0157	- 0.4764	0.0100	0.0705
Control	0.2317	- 0.1524	0.0000	0.0002

Furthermore, in a parallel study of the treatments in the zero-order model, treatment 3 stood out with the highest R^2 of 0.9956 and the least SSE (Sum of Square Error) and RMSE of 0.0417 and 0.1444 respectively. The high R^2 implies that the model's predictors sufficiently addressed the dependent variable disparity, while the low SSE described the quality response from the experiment [22]. Therefore, this indicates that the treatment 3 coating formulation is statistically feasible to perform better than other formulations. Thus, the model $[A]_f = -0.5273t + [A]_0$ as shown in Table 4 was recommended for accurately predicting the weight loss of papaya coated with the sample 3 formulation. This model suggests that the coated papaya would degrade by 0.5273g in 0.021 days (30 minutes, 14 seconds, 24 microseconds). Similarly, the kinetic model for the control $[A]_f = -2.123t + [A]_0$ was considered suitable for predicting the weight loss and required degradation time for uncoated papaya stored at room temperature.

Table 4 Model proposition for coated and uncoated papaya at storage with zero order kinetic parameters analysis

Sample	Rate constant, K (g/day)	Half-life, $t_{1/2}$ (day)	Model proposition	Remarks
1	0.00001	1105	$[A]_f=-0.00001t+[A]_0$	NR
2	0.6383	0.0173	$[A]_f = -0.6383t + [A]_0$	R
3	0.5273	0.0210	$[A]_f = -0.5273t + [A]_0$	R
4	0.2553	0.0433	$[A]_f = -0.2553t + [A]_0$	R
5	0.2763	0.0400	$[A]_f = -0.2763t + [A]_0$	R
6	0.1653	0.0668	$[A]_f = -0.1653t + [A]_0$	R
Control	2.123	0.0052	$[A]_f = -2.123t + [A]_0$	R

R= Recommended, NR= Not Recommended

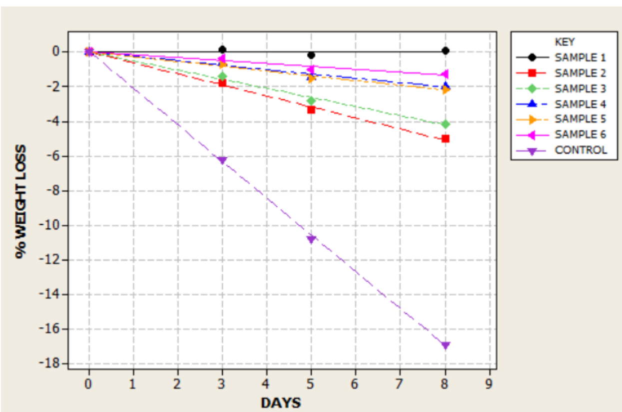

Figure 2 Weight loss kinetics of zero order for coated papaya

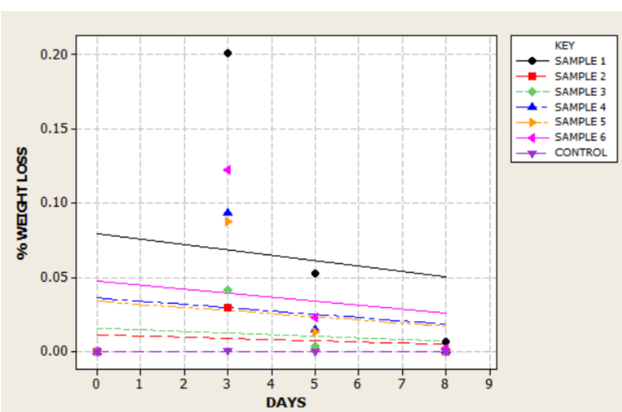

Figure 3 Weight loss kinetics of first order for treated papayas

4.0 CONCLUSION

The empirical and statistical analyses of this study demonstrated the HMW-Chitosan type effectively preserved the freshness of coated papayas. In contrast, the LMW-Chitosan type excelled in maintaining the general quality parameters of the coated papayas. The study concluded that formulations involving LMW-Chitosan, 1.0% acetic acid, 0.6% Tween 80, 0.3% NaTPP, and 0.3% ginger oil, or LMW-Chitosan, 0.6% acetic acid, 0.3% Tween 80, 0.3% NaTPP, and 0.3% ginger oil are suitable for an overall postharvest sample of papayas. However, further investigations are recommended to explore the performance of HMW-Chitosan with variations in the concentration of other active components, facilitating a comprehensive comparative analysis of chitosan molecular weight formulations. Secondly, the study established and proposed the kinetic models for predicting the weight loss of coated papayas with chitosan-based emulsion under room temperature storage. Weight loss of the samples decreased with time following the zero-order reaction kinetics. These models can be used for papaya attribute evaluation and aid in making effective managerial decisions within the supply chain. However, investigating other factors such as microbial activity, and total soluble solids as they affect weight loss will help for further studies.

Acknowledgment

The authors are grateful for the financial support from the Ministry of Education Malaysia through the IIUM-UMPSA SUSTAINABLE Research Collaboration Grant with reference code (IIUM/504/14/11/1/IUMP-SRCG), and Universiti Malaysia Pahang Al-Sultan Abdullah (UMPSA) for additional financial support from Collaborative Research Grant (RDU223225) and Internal Research (RDU220391).

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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