



## Article

# Storage Behavior of “Seddik” Mango Fruit Coated with CMC and Guar Gum-Based Silver Nanoparticles

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**Abstract:** Mango fruit (cv. Seddik) is known as a delicate fruit for storage after harvest. Herein, carboxymethyl cellulose (CMC) and guar gum-based silver nanoparticles (AgNPs) were used as fruit coatings, and their effects on postharvest storage behavior and quality attributes were investigated. AgNPs were synthesized using a chemical reduction approach and then combined with CMC and guar gum as coating bases. Mango fruits were coated with the developed and pre-characterized CMC-AgNPs and guar gum-AgNPs, and then packed and stored at 13 °C for 4 weeks. The results showed an increase in weight loss, respiration rate, total soluble solids (TSS), total sugars, and total carotenoids over the storage period. However, this increase was comparatively less significant in coated fruits compared to uncoated fruits. Firmness and titratable acidity (TA) significantly decreased during storage, but this decrease was less in coated fruits. Silver traces in fruit pulp samples were not detected. These findings showed the efficacy of CMC-AgNP and guar gum-AgNP coatings in delaying mango fruit ripening and maintaining fruit quality during cold storage. Therefore, these coatings could be promising alternative materials for extending the postharvest life and marketing period of mango fruit.

**Keywords:** mango; edible coating; carboxymethyl cellulose (CMC); guar gum; nanoparticles; cold storage



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## 1. Introduction

Mango (*Mangifera indica* L.), a climacteric fruit, is considered the most economically important fruit crop in tropical and subtropical regions. It is harvested at the hard, green mature stage and ripens rapidly at ambient temperature [1]. The attractive appearance, intense aroma, delicious taste and flavor, and the high nutritional value of mango fruit (from vitamins, antioxidants, phenolic compounds,  $\beta$ -carotene, amino acids, minerals, and dietary fiber) have been well characterized [2,3]. Acceptance of mango fruit by consumers is highly correlated with both external and internal quality factors [4], and these factors are mainly determined by the flavor resulting from sugars, acids, and aroma volatile compounds [5].

Mango fruits are highly perishable with limited postharvest life through rapid ripening, weight loss, texture softening, conversion of starch to sugars, and decay [6,7]. Poor postharvest handling, disease incidence, and sensitivity to chilling injury contribute to high postharvest and marketing chain losses and reduce the storage period [6,8,9]. These losses can exist at all postharvest stages, from harvest to consumption [10]. In developing countries where mango fruit handling and storage techniques are not ideal, postharvest losses of mango fruit may be more than 50% due to physiological and pathological disorders [6,11]. One of the most popular mango cultivars in Egypt is “Seddik”, but it is known as a delicate mango fruit to store for a long period after harvest. Hence, postharvest practices play a pivotal role in controlling mango fruit losses by applying proper, safe,

and effective treatments during fruit harvest, handling, and storage in order to extend the postharvest shelf-life period [6,12].

One practical technique for extending the storage period is fruit coating to enhance the appearance and improve fruit quality [9,13]. Edible coatings are natural eco-friendly biodegradable products, mainly composed of polysaccharides, proteins, lipids, or a blend of these compounds [12,14,15]. Edible coatings with natural origin such as polysaccharides, the most widely used, are considered as a safe strategy for fruit preservation [12,16,17]. Polysaccharide-based edible coatings such as cellulose derivatives and pectin are highly stable, safe, nontoxic, and biodegradable [15]. Edible coatings have also been exploited to incorporate antimicrobial and antisenescence agents [14,18].

Carboxymethylcellulose (CMC) is a cellulose derivative extracted from plant cell walls composed of linear anionic chains of glucopyranosyl units with high molecular weight [19]. CMC has many food industry applications because it is nontoxic, nonallergenic, odorless, tasteless, biocompatible, and biodegradable [16,19]. CMC-based coatings are also transparent and slightly permeable to O<sub>2</sub>, CO<sub>2</sub>, and moisture [20]. Edible coatings based on CMC have been effective in preserving the postharvest quality of papaya (*Carica papaya*) [21], mandarin (*Citrus reticulata*) [22], and plum (*Prunus domestica*) [17].

Guar gum is a natural polysaccharide derived from guar bean seeds [23] and can also be used as an edible coating due to its high molecular weight, long polymeric chain, content of natural antioxidants and bioactive compounds, good availability, and ability to enhance water solubility [24]. A guar gum coating can be used commercially to extend the shelf life of fruit because it is biodegradable, easy to use, and low-cost [25]. Guar gum-based coatings have recently been used to preserve the postharvest quality of mango [26] and Valencia orange (*Citrus sinensis*) [27].

The application of nanotechnology is a new technique for extending the shelf life of fresh fruit [28]. Nanoparticles can be used for fruit preservation due to their unique physical and chemical characteristics in addition to antimicrobial properties [29,30]. Recently, the use of coating materials loaded with nanoparticles has represented an innovative and safe fruit preservation technique that guarantees less direct exposure and minimal penetration of nanoparticles into treated food products [31]. Silver nanoparticles (AgNPs) have been receiving the most attention from the research community due to their remarkable properties in size and efficient antibacterial activities [32,33]. AgNPs have been used as food additives and packaging materials to eliminate pathogens [28,34]. Edible coating formulations combined with silver nanoparticles can be used for coating edible fruit to inhibit the growth of microorganisms that cause postharvest diseases, thereby increasing their shelf life [20,35]. Many experiments have focused on the structural characteristics and bioactivity of nanocomposites as fruit coatings [36,37]. Nevertheless, it is essential to further investigate their applicability as a coating on the basis of the fruit's physicochemical and physiological responses.

Obviously, most previous studies focused on evaluating the application of different coating materials to mango fruit, but so far there are no published data on the application of coatings loaded with AgNPs to enhance the storage behavior of mango fruit, especially the fruit of the local mango cultivar "Seddik", which is known to be stored for only short period after harvest. Moreover, the studies that focused on the cold storage behavior and quality attributes of this local cultivar are few. Herein, we hypothesized that different nano-based coating materials would enhance the storage behavior and the quality attributes of "Seddik" mango fruit. Therefore, the present investigation aimed at evaluating the effects of the developed and pre-characterized CMC and guar gum-based AgNPs coatings on postharvest storage behavior and quality attributes of "Seddik" mango fruit.

## 2. Materials and Methods

### 2.1. Chemicals

All chemicals used in this study were reagent-grade and purchased from different companies worldwide. For instance, ethanol (C<sub>2</sub>H<sub>5</sub>OH, 100%), sodium chloride (NaCl,

99.8%), and sodium hypochlorite ( $\text{NaClO}/\text{H}_2\text{O}$ , 12/13%) were purchased from Chem-Lab (Zedelgem, Belgium); CMC, guar gum, beeswax, and sodium borohydride ( $\text{NaBH}_4$ , 97%) were purchased from LOBA Chemie PVT. LTD. (Mumbai, India); lecithin was purchased from Neogen (Lansing, MI, USA); silver nitrate ( $\text{AgNO}_3$ , 99.9%) was purchased from Merck (Darmstadt, Germany); polyvinylpyrrolidone was purchased from CARLO ERBA Reagents S.A.S. (Val de Reuil Cedex, France). All solutions were prepared with deionized water unless otherwise stated.

## 2.2. Preparation of CMC and Guar Gum-Based Silver Nanoparticles Coatings

### 2.2.1. Preparation of Coating Base

A coating emulsion base was prepared according to a method previously reported by Shah et al. [35] with slight modification. The following steps describe this method: 10 g of lecithin, 50 g of olive oil, and 200 mL of 0.85% saline water (0.85 g NaCl dissolved in 100 mL deionized water) were first combined and mixed well by constant stirring at room temperature. Then, beeswax (1 g) was added and the stirring temperature was increased to 65 °C with constant stirring. For each coating emulsion base, 150 mL of CMC solution (2%, *w/v*) and guar gum solution (2%, *w/v*) were prepared in separate flasks and shaken vigorously for emulsion formation. After the addition of 6.7 mL of sodium hypochlorite, the volume of each coating base was made up to 1000 mL using 0.85% saline water.

### 2.2.2. Preparation of Silver Nanoparticles

The silver nanoparticles were synthesized using the chemical reduction method of An et al. [28] with slight modification. First, solutions of silver nitrate (0.1 M), sodium borohydride (0.01 M), and polyvinylpyrrolidone (PVP 2%, *w/v*) were separately prepared. Then, a 1:1 volume ratio of sodium borohydride and PVP was mixed well and cooled in an ice bath for 30 min under constant stirring. After that, the silver nitrate solution was added to this mixture dropwise until a brown color formed, indicating the formation of silver nanoparticles.

### 2.2.3. Preparation of CMC and Guar Gum-Based Silver Nanoparticle Coatings

The above-prepared coating emulsion bases of CMC and guar gum were separately mixed with the prepared silver nanoparticle solution in a 1:1 ratio [35] to form a CMC-based silver nanoparticle coating (CMC-AgNPs) and a guar gum-based silver nanoparticle coating (guar gum-AgNPs).

## 2.3. Characterization of AgNPs and CMC and Guar Gum-Based Silver Nanoparticle Coatings

### 2.3.1. Ultraviolet (UV)/Visible Light (Vis) Spectroscopy

The AgNPs, CMC-AgNPs, and guar gum-AgNPs were characterized using a UV/Vis spectrophotometer (T80, PG Instruments Ltd., Leicestershire, UK) in the central lab of the Biochemistry Department, Faculty of Agriculture, Cairo University. The scanning range for the samples was 300–700 nm. Milli-Q water was used as a blank reference.

### 2.3.2. Transmission Electron Microscopy (TEM)

A morphological analysis, including the size and shape, of the synthesized nanoparticles was determined using transmission electron microscopy (JEOL JEM-1400, Peabody, MA, USA) at a bias voltage of 40–120 kV at the Cairo University Research Park (CURP). A drop (2  $\mu\text{L}$ ) of Milli-Q water, which dissolved synthesized nanoparticles, was placed on a carbon grid (C-grid). The size was obtained by measuring the diameter of particles present in the TEM image.

### 2.3.3. Fourier-Transform Infrared (FTIR)

The CMC coating base, guar gum coating base, CMC-AgNPs, and guar gum-AgNPs were characterized using a Fourier-transform infrared spectrophotometer (Thermo Scientific NICOLET 380 FT-IR, Waltham, MA, USA) at the Cairo University Research Park

(CURP). A sample (2 mg) was combined with potassium bromide (100 mg) and compacted to prepare a salt disc with a diameter of approximately 3 mm. The disc was immediately placed in the holder of the sample. FTIR spectra were detected in the absorption range between 400 and 4000  $\text{cm}^{-1}$ .

#### 2.4. Fruit Material

Freshly harvested “Seddik” mango fruits were obtained from a commercial mango orchard grown under well-managed conditions and located in Al-Sharkiya Governorate, Egypt. All fruits were picked on 21 July at a physiologically mature stage early in the morning and were transported to the postharvest laboratory of the Pomology Department, Faculty of Agriculture, Cairo University, within 2 h of harvest. All fruits were carefully washed with tap water and then kept at ambient temperature to dry. Mature, uniformly sized mango fruits free of mechanical damage were selected for coating application.

#### 2.5. Coating Application and Storage Conditions

Mango fruits were divided into three treatments, each containing 60 fruits. The first treatment group was immersed into CMC-AgNP coating material for 30 s and the second treatment group was immersed into guar gum-AgNP coating material for 30 s. The third group was left without coating as a control. There were three biological replicates in each treatment, and each replication had 20 fruits. All mango fruits were packed in carton boxes, 10 fruits in each, and stored in a cold chamber at 13 °C with 85–90% relative humidity. The cold chamber was disinfected using sodium hypochlorite solution followed by ethanol (70%, *v/v*) at the beginning of the storage period. Over the storage period, a fruit quality assessment of each treatment was carried out regularly after 0, 7, 14, 21, and 28 days.

#### 2.6. Fruit Quality Attributes

##### 2.6.1. Weight Loss

To calculate the weight loss in defined periods during storage, fruits ( $n = 10$ ) of each treatment were periodically weighed using a digital balance to compare the difference between initial fruit weight ( $W_1$ ) and weight at sampling date ( $W_2$ ), and weight loss (%) was calculated according to Shah and Hashmi [38] using the following equation:

$$\text{Weight loss (\%)} = ((W_1 - W_2)/W_1) \times 100. \quad (1)$$

##### 2.6.2. Respiration Rate

Three mango fruits of each treatment were separately incubated in 2 L airtight glass jars for 24 h at 13 °C to measure the respiration rate. The gas sampling was measured by analyzing carbon dioxide using a gas analyzer (Model Servomex1400; Servomex, East Sussex, UK). The respiration rate was calculated according to the equation provided by Pristijono et al. [39] and expressed as  $\text{nmol CO}_2 \cdot \text{kg}^{-1} \cdot \text{s}^{-1}$ .

##### 2.6.3. Fruit Pulp Firmness

Fruit pulp firmness ( $n = 3$ ) from each treatment was measured using a pressure firmness tester (L-10, AMETEK Inc., Lansdale, PA, USA) with a stainless-steel rod (8 mm diameter) and a displacement depth of 10 mm. The entire fruit was placed on a flat surface, and firmness was measured on the pared fruit surface in the equatorial zone. The results were expressed in N according to Liu et al. [40].

##### 2.6.4. Total Soluble Solids (TSS), Titratable Acidity (TA), and TSS/TA Ratio

Mango fruit juice was extracted from three fruits per replicate to determine TSS and TA according to the method of Khaliq et al. [41]. TSS concentration was measured using a digital refractometer (Pocket refractometer PAL-1, Atago, Tokyo, Japan) and data were expressed as a percentage of TSS. The TA (%) of the fruit pulp was measured by titrating a 10 mL aliquot of juice (1 mL juice + 9 mL distilled water) with 0.1 N NaOH using

phenolphthalein indicator and expressed as a percentage of citric acid equivalents on the basis of fresh weight. By dividing the TSS percentage with the corresponding acidity percentage, the TSS/TA ratio was calculated.

#### 2.6.5. Total Sugar Content

Total sugar content was determined using the phenol–sulfuric acid method [42]. Fruit pulp samples (0.25 g—collected from three fruits) were homogenized in 20 mL of 70% ethanol (*v/v*) and filtered. The filtrates (1 mL) were treated with 1 mL of 5% phenol (*v/v*) and 5 mL of 98% sulfuric acid (*v/v*). After 1 h, the absorbance of the cold and colored solutions was read at 490 nm using UV/Vis spectrophotometer (UNICO S2100, Cole Parmer Instruments, Chicago, IL, USA). A standard curve was generated using a standard glucose solution, and total sugar content was expressed as mg glucose equivalents per g fresh weight.

#### 2.6.6. Total Carotenoid Content

Fruit pulp samples (0.25 g—collected from three fruits) were homogenized in 20 mL of 80% acetone (*v/v*) for carotenoid extraction. Extracts were filtered and absorbance was measured using a UV/Vis spectrophotometer (UNICO S2100, Cole Parmer Instruments, Chicago, IL, USA) at 480 and 510 nm. Total carotenoid content was calculated according to Jensen [43] as  $\mu\text{g}\cdot\text{g}^{-1}$  of fresh weight using the following formula:

$$\text{Total Carotenoids } (\mu\text{g}\cdot\text{g}^{-1}) = 7.6 (OD_{480}) - 1.49 (OD_{510}) \times (V/1000) \times (W), \quad (2)$$

where *OD* = optical density, *V* = final volume of 80% acetone, and *W* = sample weight.

#### 2.6.7. Determination of Total Silver Concentration

Samples of coated fruit (CMC-AgNPs and guar gum-AgNPs) at 14 and 28 days of storage were digested in an acid solution (70% nitric acid + 30% hydrogen peroxide (*v/v*)) using a microwave digestion system (Multi-wave PRO, Anton-Paar, Graz, Austria) according to the method of Altundag and Tuzen [44]. All samples were digested to provide an acceptable matrix for assessing silver ions and having adequate and reliable recovery compatible with the analytical method. The silver content was determined according to Zhang et al. [45]. The silver concentration was calculated using inductively coupled plasma optical emission spectrometry (5100 ICP-OES, Agilent, Santa Clara, CA, USA) in an atomic spectroscopy lab, National Research Center, Giza, Egypt.

### 2.7. Statistical Analysis

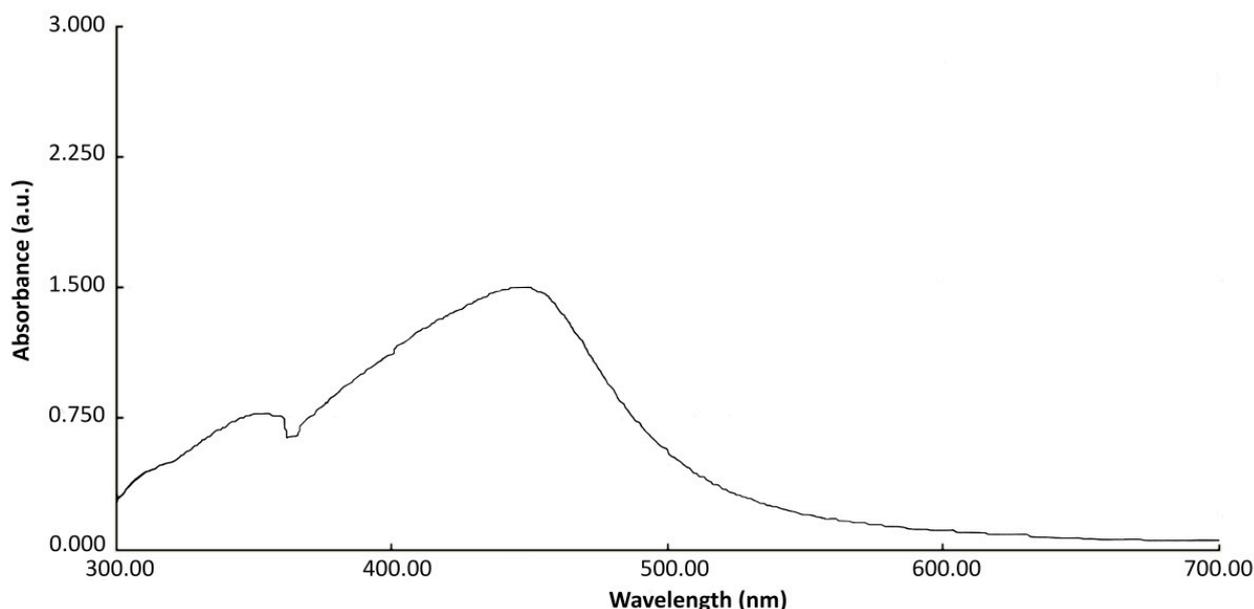
The experiment was carried out in a completely randomized design with three replications. The model assumptions of normality were tested using Shapiro–Wilk’s test ( $P \leq 0.05$ ) to perform an appropriate ANOVA. When these assumptions were not satisfied, data were transformed to ensure that the residuals followed an approximately normal distribution for further analysis. Statistical analysis was performed with normalized data, but all results are shown as original data. Two-way analysis of variance (ANOVA) was performed to investigate the effect of coating treatment, storage time, and their interaction. Significant differences ( $\alpha \leq 0.05$ ) were determined using Duncan’s multiple range [46] test using the general linear model (GLM) procedure—SAS software Version 9.0 (SAS Institute Inc., Cary, NC, USA). Results were plotted as the mean  $\pm$  standard error (SE) using SigmaPlot (version 14.0, Systat Software, Inc., San Jose, CA, USA). A linear model of regression was used to describe the storage time-related change in fruit quality attributes. Correlation analysis was also performed for analyzing the association between fruit quality parameters during storage using Pearson’s test ( $P \leq 0.05$  and  $P \leq 0.01$ ). Principal component analysis (PCA) was applied using XLSTAT (Addinsoft, New York, NY, USA) to identify relationships between factors (coating treatments, storage time, and their interaction) and fruit quality parameters.

### 3. Results and Discussion

#### 3.1. Characterization of AgNPs and CMC and Guar Gum-Based Silver Nanoparticle Coatings

##### 3.1.1. UV/Vis Spectroscopy

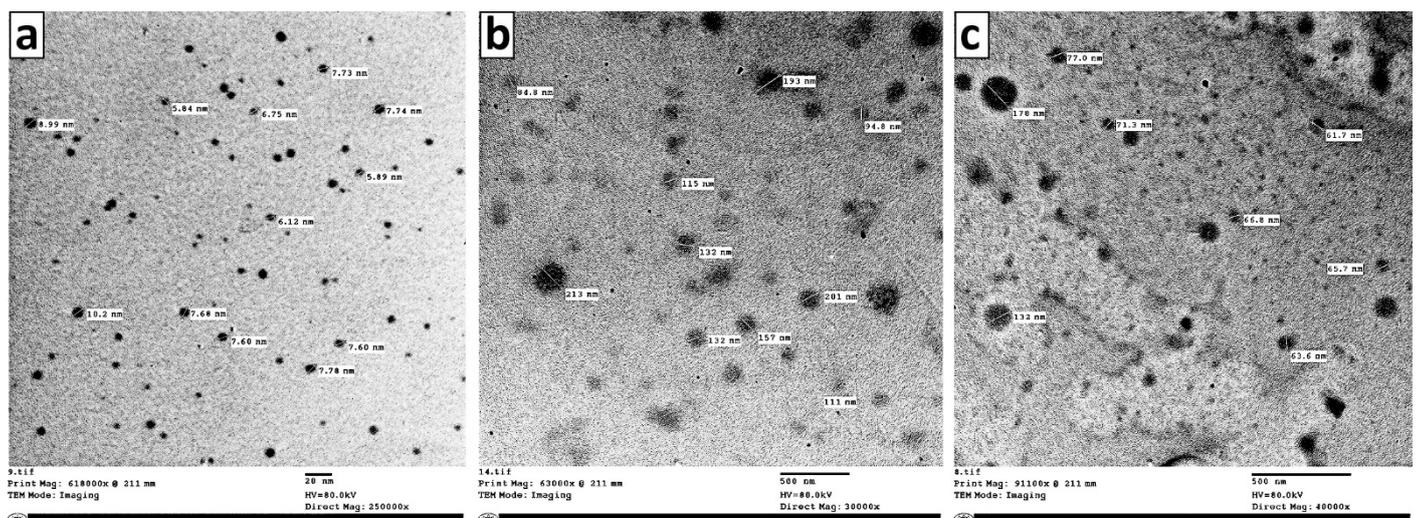
The UV/Vis spectrum of AgNPs (Figure 1) was recorded as a function of wavelength using a UV/Vis spectrophotometer. AgNPs exhibit peaks of absorption between 300 and 700 nm. The UV/Vis spectrum of the synthesized AgNPs had a maximum absorbance at 447 nm (Figure 1). The 420 nm wide absorption band is typical of AgNPs, and it is attributable to plasmon surface resonance excitation [47,48]. This single peak of the plasmon surface resonance (PSR) revealed that the AgNPs were spheres with a broad size distribution. Furthermore, other researchers confirmed the PSR peak between 410 and 450 nm as a sign of AgNP synthesis [49,50]. The absorption spectrum of Ag<sup>+</sup>/CMC showed a shift in wavelength where the absorbance value decreased, and the UV/Vis spectrum of Ag<sup>+</sup>/guar gum was also in the same range as that of Ag<sup>+</sup>/CMC (data not shown). This is probably due to the interaction of CMC with Ag<sup>+</sup> ions, although plasmon absorption of nanosilver also occurs [51,52], which may be assigned to a  $\pi \rightarrow \pi^*$  transition derived from carbonyl groups (C=O) or ethylene unsaturated bonds (=C=C=) of the type (-CH=CH-)CO in the CMC structure [53,54].



**Figure 1.** Ultraviolet (UV)/visible light (Vis) spectrum of the synthesized silver nanoparticles (AgNPs).

##### 3.1.2. Transmission Electron Microscopy

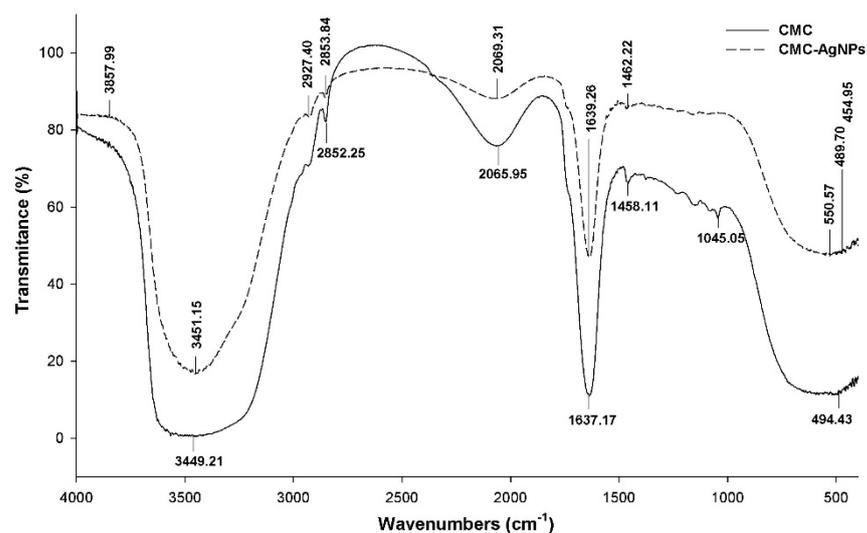
TEM images of the synthesized silver nanoparticles (dark spherical objects) are shown in Figure 2a. Their size ranged from 5.84 nm to 10.2 nm. TEM images revealed that nanoparticles were formed at sizes ranging from 84.8 to 213 nm for CMC-AgNPs and 61.7 to 132 nm for guar gum-AgNPs (Figure 2b,c). The size measured was greater than that measured for AgNPs due to the polymeric matrix of CMC and guar gum. The TEM images indicated that the AgNPs were spherical in shape and well scattered in the solution, and the nanoparticles were separated from each other without any aggregation.



**Figure 2.** The transmission electron microscope (TEM) micrographs of (a) AgNPs, (b) carboxymethyl cellulose (CMC)-AgNPs, and (c) guar gum-AgNPs.

### 3.1.3. Fourier-Transform Infrared

The FTIR spectrum of the CMC coating base showed peaks at 3449.21, 2852.25, 2065.95, 1637.17, 1458.11, 1045.05, and 494.43  $\text{cm}^{-1}$  (Figure 3). The broad peak at 3449.21  $\text{cm}^{-1}$  indicated the existence of the  $-\text{OH}$  stretching vibration of CMC, while the peaks observed around 2852.25 and 1045.05  $\text{cm}^{-1}$  may be assigned to  $\text{C}-\text{H}$  stretching and  $-\text{OH}$  bending vibration, respectively. The observed bands in the CMC coating base at 3449.21 and 2852.25  $\text{cm}^{-1}$  were shifted to a higher wave number and became sharper in CMC-AgNPs due to the interaction of AgNPs with the  $-\text{OH}$  group of CMC. The bands detected at 1637.17 and 1458.11  $\text{cm}^{-1}$  may be related to the carboxylate anion in the symmetric mode in CMC and olive oil [55,56].



**Figure 3.** Fourier-transform infrared (FTIR) absorption spectra of CMC coating base (solid line) and CMC-AgNPs (dashed line).

FTIR spectra of the guar gum coating base and guar gum-AgNPs are shown in Figure 4. The FTIR spectrum of the guar gum coating base showed peaks at 3455.67, 2042.88, 1637.81, 1535.12, 1458.82, and 509.10  $\text{cm}^{-1}$ . The first sizeable peak at 3455.67  $\text{cm}^{-1}$  demonstrated the existence of guar gum  $-\text{OH}$  stretching vibration, while the peak at 1458.82  $\text{cm}^{-1}$ , attributed to the symmetrical deformation of the  $-\text{CH}_2$  group, was of negligible intensity. The band

detected at  $1637.81\text{ cm}^{-1}$  is related to the carboxylate anion in symmetric mode of the coating base. In guar gum-AgNPs, the observed peak at  $3453.64\text{ cm}^{-1}$  shifted to a lower IR frequency value than the corresponding peak in the guar gum coating base. This probably indicated an interaction of AgNPs with the  $-\text{OH}$  group of guar gum. The presence of metal–oxygen bond participation in the biopolymer blend nanocomposite was clearly defined by the peak at  $584.30\text{ cm}^{-1}$ . Metal ions, such as silver, have a strong affinity to form coordination bonds with guar gum polar  $-\text{OH}$  groups. With increasing concentrations of AgNPs, the interaction between silver nanoparticles and the oxygen atom of the  $-\text{OH}$  group became more robust. Due to this interaction, the position and peaks of IR spectra of guar gum-AgNPs (Figure 4) changed as compared to the guar gum coating base. The shifting and broadness of the detected bands in the case of guar gum-AgNPs gave evidence of the presence of AgNPs inside the polymer matrix [57].

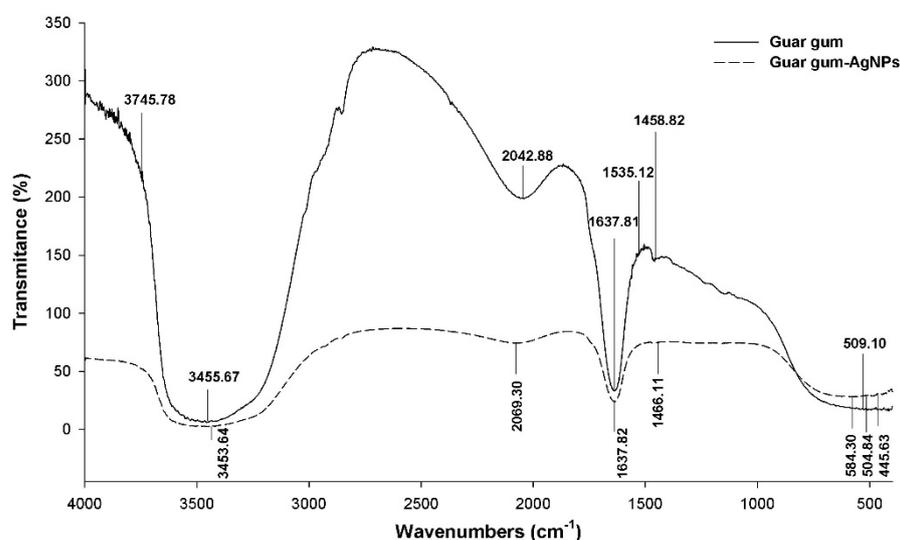


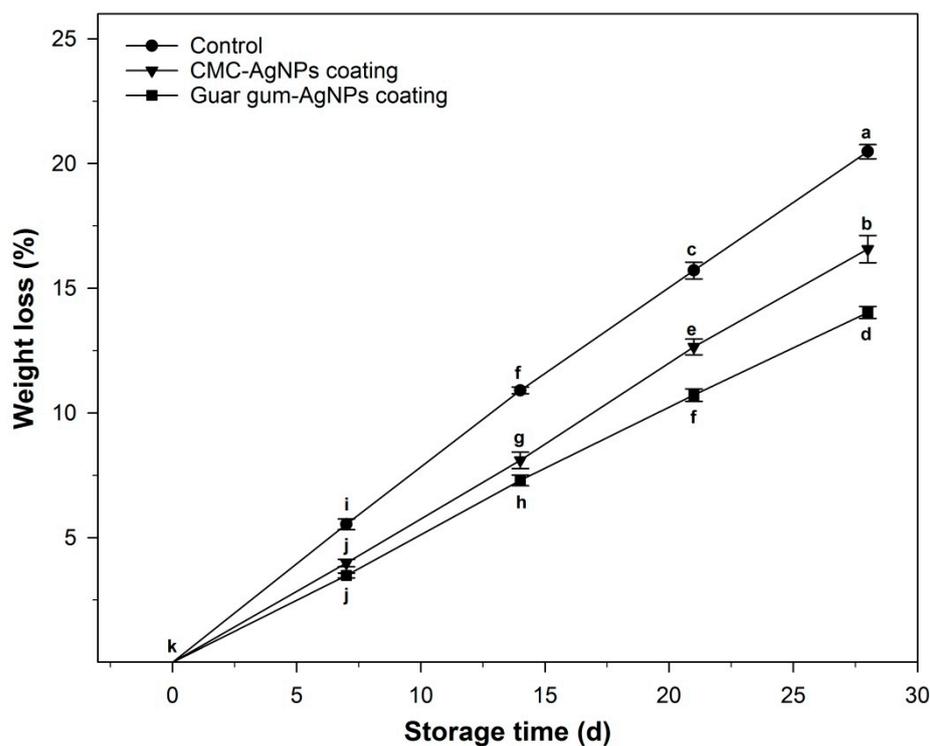
Figure 4. FTIR spectra of guar gum coating base (solid line) and guar gum-AgNPs (dashed line).

### 3.2. Fruit Quality Attributes

#### 3.2.1. Weight Loss

Mango fruit weight loss increased progressively in all treatments over the storage period of 28 days, as shown in Figure 5. The weight loss values were significantly affected by storage period ( $\alpha \leq 0.05$ ). The regression analysis showed that the fruit weight loss followed a linear increment during fruit storage. The high value of coefficient of determination indicated the existence of a strong relationship between weight loss and storage period (Table S1, Supplementary Materials), and the slope of the weight loss regression equation was higher in the control treatment ( $\beta_1 = 0.7304$ ;  $R^2 = 0.9987$ ) than for fruits coated with CMC-AgNPs ( $\beta_1 = 0.5973$ ;  $R^2 = 0.9995$ ) and guar gum-AgNPs ( $\beta_1 = 0.5043$ ;  $R^2 = 0.9994$ ). However, coatings markedly controlled weight loss during storage, particularly with the guar gum-AgNP coating compared to CMC-AgNP coating and uncoated fruits (control) over the storage period. Maximum weight loss was observed in uncoated fruits (20.48%) after 4 weeks of storage at  $13\text{ }^\circ\text{C}$ , while, on the same date, the weight loss values of coated fruits with CMC-AgNPs and guar gum-AgNPs were 16.57% and 14.03%, respectively. Water loss results in changes in texture, shrinkage, and other physical properties of the fruit, thus reducing the quality value of fresh fruit and affecting shelf life [58]. Mature climacteric fruits undergo a series of metabolic processes, such as transpiration and respiration, when detached from the tree; the correlation between weight loss and respiration rate was highly positive, while it was strongly negatively correlated with pulp firmness (Table S2, Supplementary Materials). These metabolic processes eventually result in fruit weight loss during the postharvest and storage period [9,59]. The coating material can mitigate the loss of water and reduce these deleterious effects by acting as a barrier of

moisture between the fruit and the environment around it. Moreover, polymeric coating films with efficient water vapor barriers can reduce the movement of water vapor from the stomata of fruit peels (reducing fruit transpiration) and maintaining the turgidity of cell walls [60]. Another study also reported that fresh weight preservation in fruits treated with films was reinforced with nanoparticles [61].

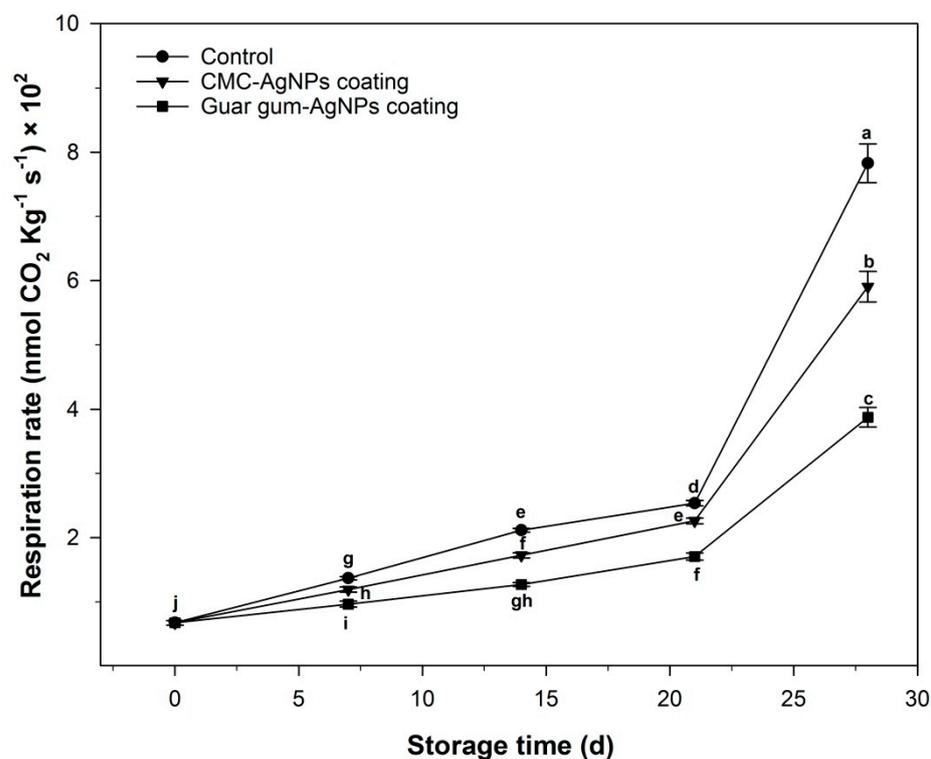


**Figure 5.** Effect of CMC-AgNPs and guar gum-AgNPs coatings on weight loss of “Seddik” mango fruits stored at 13 °C for 4 weeks. Vertical bars represent the standard error (SE) of means. Means with the same letter are not significantly different according to Duncan’s multiple range test ( $\alpha \leq 0.05$ ).

### 3.2.2. Respiration Rate

The respiration rate of fresh fruit, such as mango fruit, is a good indicator for estimating storage behavior. The change in respiration rate of mango fruits with or without coatings is represented in Figure 6. A significant increase in the respiration rate was recorded for both coated and control treatment fruits during the storage period. Respiration rate increased linearly during the storage period. The positive relationship between respiration rate and storage time was highly dependent on coating treatment (Table S1, Supplementary Materials). The regression equation of control fruits had a higher slope ( $\beta_1 = 22.11$ ;  $R^2 = 0.7408$ , not significant (NS)), while coating treatments recorded lower respiration rates during the storage period with slight differences in regression equation slopes (10.1947;  $R^2 = 0.7845$ , for guar gum-AgNPs and 16.484;  $R^2 = 0.7741$  for CMC-AgNPs). The lowest respiration rate values were observed for guar gum-AgNP-coated fruits followed by those coated with CMC-AgNPs. Respiration rates of control and coated fruits with CMC-AgNPs and guar gum-AgNPs were low during the first 21 days of storage; afterward, they exhibited a sharp increase with values equal to  $7.83 \times 10^2$ ,  $5.91 \times 10^2$ , and  $3.87 \times 10^2$  nmol  $\text{CO}_2 \cdot \text{kg}^{-1} \cdot \text{s}^{-1}$ , respectively (Figure 6). Respiration rates in the control were significantly and consistently greater than those in the coated fruits across the entirety of the storage period. Ripening in climacteric fruit such as mango is characterized by a significant and rapid increase in respiration rate accompanied by intensive metabolic changes [59]. The respiration rate was positively correlated with the increases in total sugar content in the fruit and with the total soluble solid content. Meanwhile, it had a strongly negative correlation with titratable acidity (Table S2, Supplementary Materials). The coat-

ing materials operate as a semipermeable barrier to the exchange of gases, movement of solvents, and moisture, thus decelerating the rate of respiration, oxidation reactions, and weight loss [62]. Ultimately, uncoated fruits showed more rapid perishability than coated fruits. Silva et al. [58] also noticed an increase in the rate of mango fruit respiration with an increasing frequency of decay.

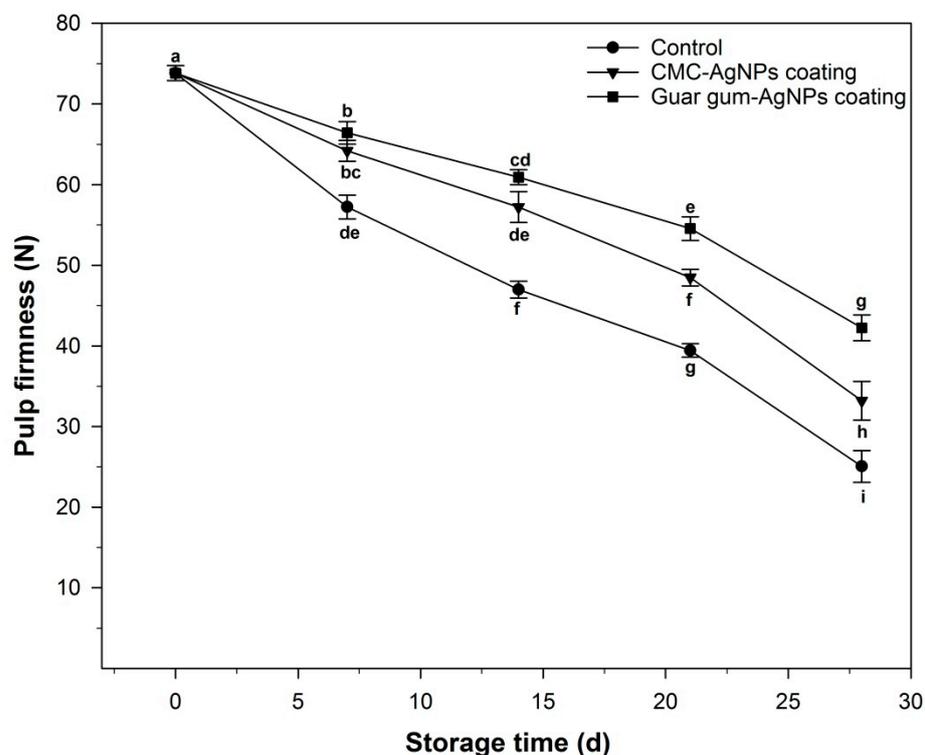


**Figure 6.** Effect of CMC-AgNP and guar gum-AgNP coatings on respiration rate of “Seddik” mango fruits stored at 13 °C for 4 weeks. Vertical bars represent the SE of means. Means with the same letter are not significantly different according to Duncan’s multiple range test ( $\alpha \leq 0.05$ ).

### 3.2.3. Fruit Pulp Firmness

Fruit pulp firmness, an essential factor for the postharvest fruit quality, was found to be significantly decreased for all treatments over the storage period (Figure 7). The regression analysis showed that the fruit firmness decreased linearly during the fruit storage period. Regression equations described the changes in pulp firmness during the storage period for each coating treatment (Table S1, Supplementary Materials). The slope of the regression equations revealed that the fruit softening rate was slower in fruits coated with guar gum-AgNPs ( $\beta_1 = -1.0717$ ;  $R^2 = 0.9751$ ) and CMC-AgNPs ( $\beta_1 = -1.3854$ ;  $R^2 = 0.9786$ ) than the control treatment ( $\beta_1 = -1.6479$ ;  $R^2 = 0.9841$ ). The loss of firmness during storage is the most apparent textural change in fruit. Notably, the coating greatly postponed the loss of fruit firmness, and the maximum loss of firmness was found at the end of the storage period in the control fruits (25.05 N). Meanwhile, mango fruits coated with CMC-AgNPs and guar gum-AgNPs maintained higher firmness than the control with values equal to 33.21 and 42.25 N, respectively, at the end of the storage period. Moreover, guar gum-coated fruits were significantly firmer than CMC-coated fruits. Mechanical properties of mango fruit such as firmness are fundamental for fruit handling, transport, storage, and consumer acceptability [63]. The maintenance of the pulp firmness of the coated fruits may be attributed to decreased respiration and other processes of ripening during storage, ultimately providing the fruit with greater firmness [9,38]. Coating material effectively slowed down the metabolic and enzymatic activities in the fruits, resulting in a slower degradation of pulp tissues. Obviously, the inhibition of respiratory metabolism related to the expression of enzyme activities, mostly polygalacturonase and pectin methylesterase

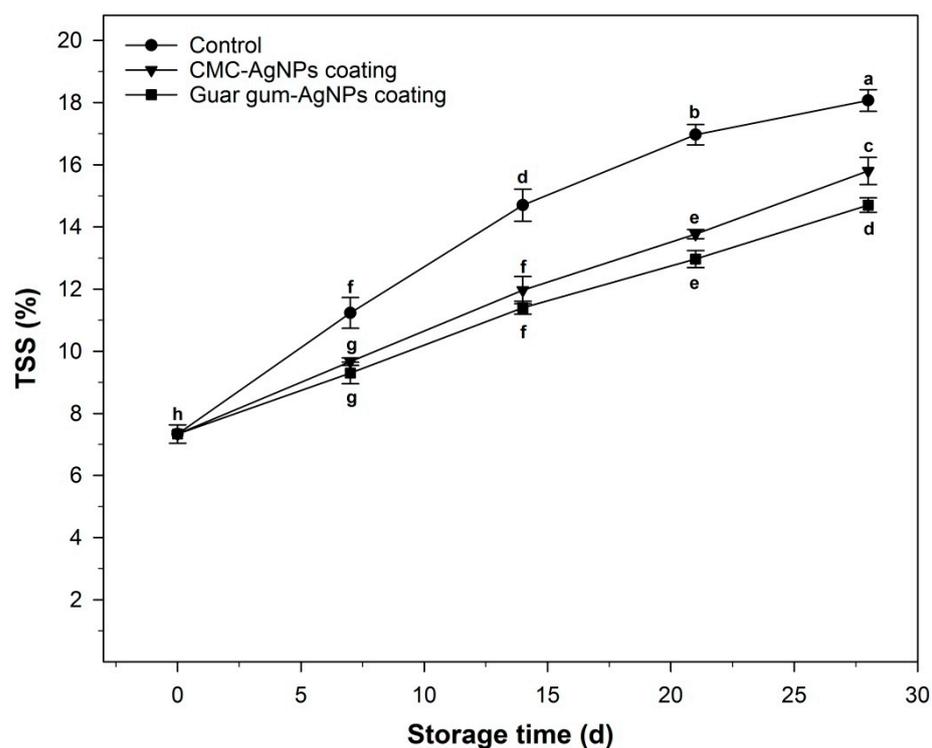
responsible for fruit softening, has been associated with internal gaseous modification of the fruit caused by coatings [64,65]. Similar to the data of Silva et al. [58], a significant negative correlation was observed between fruit firmness and some of the biochemical parameters such as fruit respiration rate and total sugar content (Table S2, Supplementary Materials). Silver nanocomposite films effectively delayed mango softening during the storage period [66].



**Figure 7.** Effect of CMC-AgNP and guar gum-AgNP coatings on pulp firmness of “Seddik” mango fruits stored at 13 °C for 4 weeks. Vertical bars represent  $\pm$  SE of means. Means with the same letter are not significantly different according to Duncan’s multiple range test ( $\alpha \leq 0.05$ ).

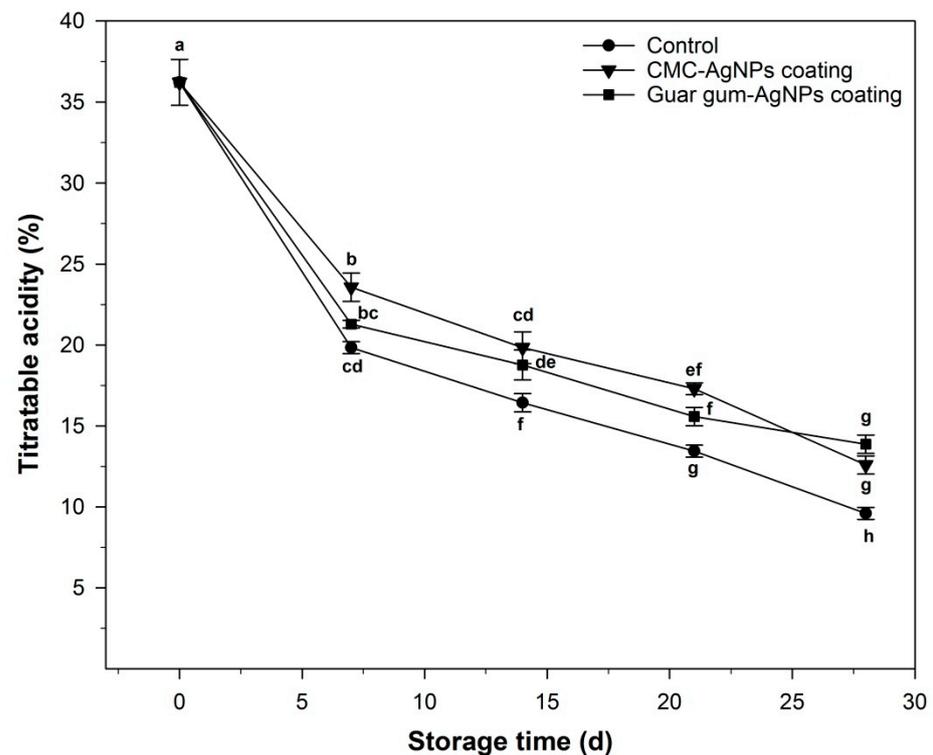
### 3.2.4. Total Soluble Solids (TSS), Titratable Acidity (TA), and TSS/TA Ratio

A gradual increase in total soluble solids (TSS) was observed as storage time progressed in all treatments (Figure 8). The increase was significantly lower in guar gum- and CMC-coated fruits than the control fruits. Fruits coated with CMC-AgNPs recorded slightly higher, but not statistically evident, TSS values compared with guar gum-AgNPs up to 21 days of storage. Generally, the control treatment showed the highest significant TSS value (18.06%) after 28 days of cold storage. Regression analysis showed that the TSS content increased linearly (Table S1, Supplementary Materials) during fruit storage. The predicted change in TSS content for mango fruits with different coating treatments could be estimated using the slope of the regression equation ( $\beta_1 = 0.3889, 0.3006,$  and  $0.263$  for the control, CMC-AgNPs, and guar gum-AgNPs, respectively) with the coefficient of determination ( $R^2 > 0.95$ ). During the storage period, the variation in TSS could be due to alterations of the cell wall and hydrolysis of complex carbohydrates by the activities of hydrolytic enzymes [67], which was shown by a highly negative correlation with pulp firmness and a strongly positive correlation with total sugar content (Table S2, Supplementary Materials). In previous studies on coated fruits, similar findings were also observed to decelerate the increase in TSS [35,60]. AgNPs-based coatings reduced the increase in TSS content of coated fruits compared with uncoated fruits during storage [35,68]. Gol et al. [69] suggested that the low TSS for coated fruits presumably occurred because of the barrier effect of coating against respiration and evaporation, thus decelerating the metabolic activities of the fruit.



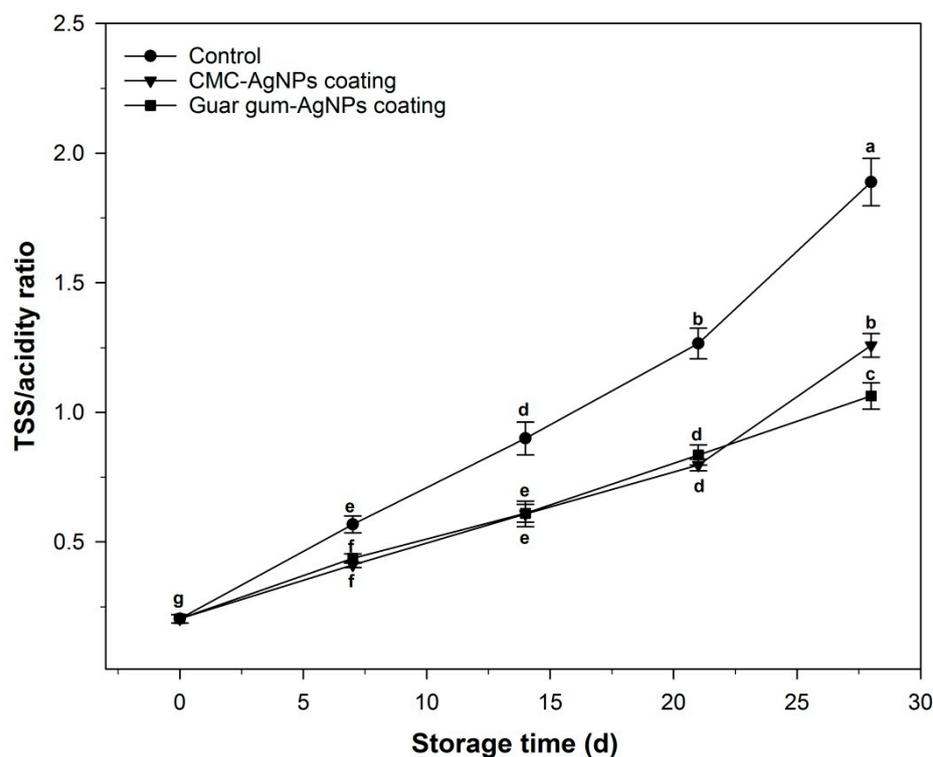
**Figure 8.** Effect of CMC-AgNP and guar gum-AgNP coatings on total soluble solids (TSS) of “Seddik” mango fruits stored at 13 °C for 4 weeks. Vertical bars represent the SE of means. Means with the same letter are not significantly different according to Duncan’s multiple range test ( $\alpha \leq 0.05$ ).

A gradual decline was detected in titratable acidity (TA) for all treatments over the storage period of 28 days (Figure 9). The TA results illustrated that CMC-AgNP and guar gum-AgNP coatings reduced the declining trend for mango fruits, compared to the control during the storage period. Fruits coated with CMC- and guar gum-AgNPs recorded statistically similar TA values until the end of the storage period, while control fruits had significantly lower values. The decline in fruit acidity during fruit ripening is associated with a reduction in organic acids, such as citric or malic acid, which are known to be the primary substrates for the respiration process of climatic fruit, such that a decrease in acidity can be anticipated in highly respiratory fruit [70]. The regression analysis showed that titratable acidity was significantly affected by storage period. The negative slope values (Table S1, Supplementary Materials) indicated that TA tended to decrease during cold storage (13 °C). The slope ( $\beta_1$ ) of the regression equations of the coated fruit was very similar ( $-0.7202$ ;  $R^2 = 0.8024$  for guar gum-AgNPs and  $-0.7647$ ;  $R^2 = 0.8959$  for CMC-AgNPs) and the change in TA was slower than for the control fruits. In line with the data of Silva et al. [58], changes in fruit acidity showed significant negative correlations ( $P \leq 0.01$ ) with several parameters such as weight loss, TSS, respiration rate, and total sugars with a correlation coefficient ( $r$ ) ranging from 0.614 to 0.911 (Table S2, Supplementary Materials). In other words, as previously mentioned, the application of coating decelerates the rate of respiration and metabolic processes that turn organic acid into sugars, thus limiting the excess increase of organic acids in respiration reactions [35].



**Figure 9.** Effect of CMC-AgNP and guar gum-AgNP coatings on titratable acidity (TA) of “Seddik” mango fruits stored at 13 °C for 4 weeks. Vertical bars represent the SE of means. Means with the same letter are not significantly different according to Duncan’s multiple range test ( $\alpha \leq 0.05$ ).

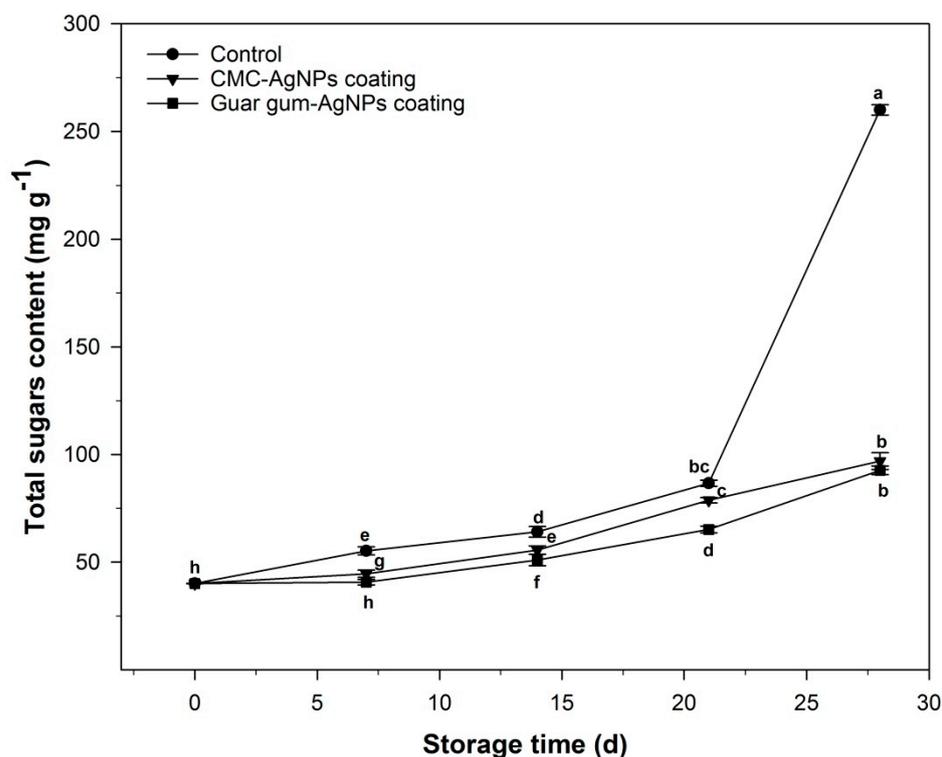
The TSS/TA ratio was significantly affected by coating treatments and storage period. The TSS/TA ratio values for all coated fruits were lower than those for the uncoated fruits (Figure 10). No difference was observed between CMC-AgNP and guar gum-AgNP coatings until 21 days of the storage period. However, the TSS/acidity ratio for CMC-AgNP-coated mango was slightly higher after 28 days of storage. The TSS/TA ratio showed a rapid increase in uncoated fruits, indicating fruit ripening velocity. The ratio of TSS and TSS/TA is known to be an indicator of fruit quality. Linear regression analysis illustrated that the TSS/TA ratio increased linearly during fruit storage. Control fruits had a higher regression equation slope value ( $\beta_1 = 0.05813$ ;  $R^2 = 0.9819$ ), while coating treatments recorded significantly lower slope values during the storage period with slight differences between coating types (0.03026;  $R^2 = 0.9981$  for guar gum-AgNPs and 0.03566;  $R^2 = 0.9583$  for CMC-AgNPs) (Table S1, Supplementary Materials). As predicted, the TSS/TA ratio had significant correlations ( $P \leq 0.01$ ) with TSS and with titratable acidity (Table S2, Supplementary Materials). Inverse relationships were observed between TSS values and TA values of mango pulp as a result of the decrease in acidity for their further utilization in fruit metabolic process during ripening [67].



**Figure 10.** Effect of CMC-AgNP and guar gum-AgNP coatings on TSS/TA ratio of “Seddik” mango fruits stored at 13 °C for 4 weeks. Vertical bars represent the SE of means. Means with the same letter are not significantly different according to Duncan’s multiple range test ( $\alpha \leq 0.05$ ).

### 3.2.5. Total Sugar Content

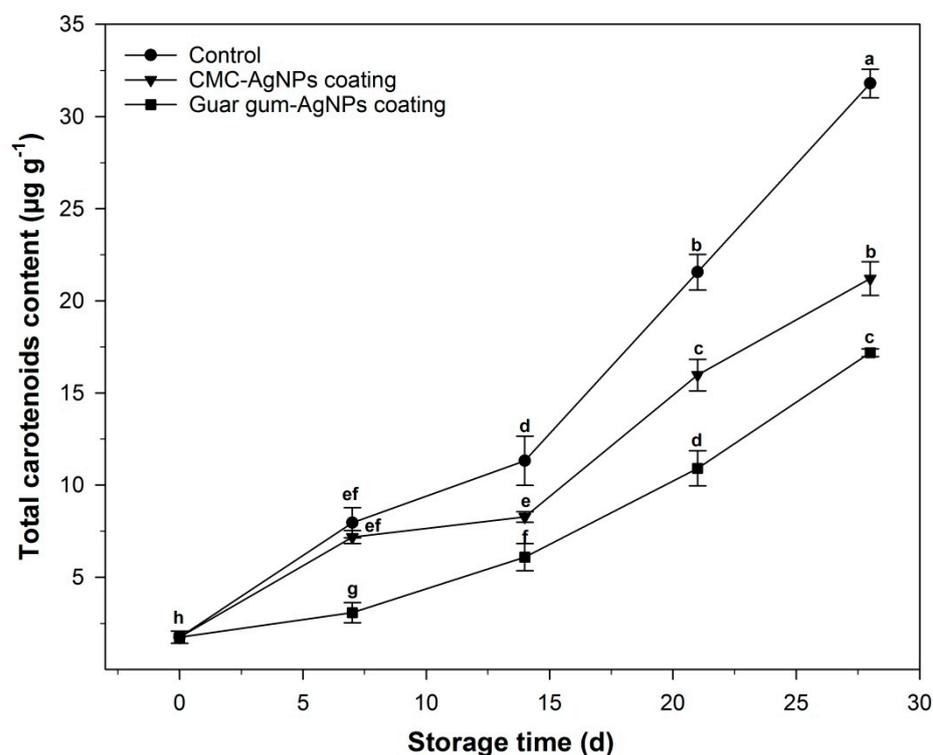
During the storage period, total sugars of coated fruit slowly increased (Figure 11). The highest total sugar content ( $260.03 \text{ mg}\cdot\text{g}^{-1}$ ) was with the uncoated fruits after 28 days of storage. A linear relationship was found between total sugar content and the length of the storage period. The slope of the regression equations showed that total sugar content was affected by coating treatments ( $\beta_1 = 6.7319, 2.111, \text{ and } 1.8499$  for the control, CMC-AgNPs, and guar gum-AgNPs, respectively); the positive estimated  $\beta_1$  values indicated that total sugar content tended to increase during cold storage (13 °C) (Table S1, Supplementary Materials). Total sugar content increased at a very slow rate in all fruits, while a sharp increase in uncoated fruits was observed after 21 days of storage. The total sugar content of the fruits coated with CMC-AgNPs and guar gum-AgNPs differed slightly. The accumulation of sugars increases with fruit ripening via the hydrolysis of complex carbohydrates to simple sugars during storage due to metabolic activities. Coating inhibits fruit ripening and the transition of complex carbohydrates into simple sugars [71]. In other words, changes in fruit biochemical activity accompany fruit ripening; the gradual increase in total sugar content during fruit storage results from the hydrolysis of insoluble polysaccharides to soluble sugars [72]. The rapid change in total sugars of the control treatment can be explained by the mango fruit respiratory burst, which is distinguished by major changes in fruit biochemical activity, leading to a decrease in starch content and an increase in sugar content [73]. The increase in sugar content has a strong positive relationship with some physical parameters such as weight loss and respiration rate. Conversely, sugar content was negatively correlated with titratable acidity and with pulp firmness (Table S2, Supplementary Materials). Silva et al. [58] highlighted that fruit coating treatments reduced respiratory activity and ethylene production, which slows down the ripening process, delays the climacteric peak, and increases fruit storage life. During the storage time, the effect of AgNPs on the suppressed production of ethylene, the critical factor for fruit ripening, may be attributed to the decomposition or oxidation of ethylene to water and carbon dioxide [74].



**Figure 11.** Effect of CMC-AgNP and guar gum-AgNP coatings on total sugar content of “Seddik” mango fruit stored at 13 °C for 4 weeks. Vertical bars represent the SE of means. Means with the same letter are not significantly different according to Duncan’s multiple range test ( $\alpha \leq 0.05$ ).

### 3.2.6. Total Carotenoid Content

As ripening progressed in all fruits, a steady increase in carotenoid content was noted, but the rate was slower in coated fruits (Figure 12). Statistical analysis showed significant differences among sampling dates, coating treatments, and their interaction. The regression analysis showed that the total carotenoid content increased linearly with time. The slope of the regression equations revealed that accumulation of carotenoids was slower in fruits coated with guar gum-AgNPs ( $\beta_1 = 0.553$ ;  $R^2 = 0.9386$ ) and CMC-AgNPs ( $\beta_1 = 0.6817$ ;  $R^2 = 0.9626$ ) than the control treatment ( $\beta_1 = 1.052$ ;  $R^2 = 0.9634$ ) (Table S1, Supplementary Materials). The increase in carotenoid content was significantly lower ( $\alpha \leq 0.05$ ) for fruit coated with CMC-AgNPs and guar gum-AgNPs than for uncoated ones. It seems that the guar gum-AgNP coating delayed synthesis and accumulation of carotenoids more than in fruits coated with CMC-AgNPs. Control mangoes significantly showed the highest carotenoids ( $31.79 \mu\text{g}\cdot\text{g}^{-1}$ ) after 4 weeks of the storage period. The mango fruit ripening process involves a series of biochemical reactions resulting in developing pigments via carotenoid biosynthesis [5]. According to the correlation analysis, total carotenoid content, which reflects fruit pulp color, had a positive correlation with total sugar content, respiration rate, and TSS (Table S2, Supplementary Materials). Postharvest treatments that control carotenoid biosynthesis and maintain the fruit color can delay fruit ripening [40]. The delay in the ripening of the coated fruit may be attributable to the modified internal atmosphere of the coated fruit, which decreases chlorophyll degradation and/or carotenoid biosynthesis [8,9]. Polysaccharide-based composite coatings have synergistic effects on color retention by delaying the development of coloring pigments in mango fruit [38,67].



**Figure 12.** Effect of CMC-AgNP and guar gum-AgNP coatings on total carotenoid content of “Seddik” mango fruit stored at 13 °C for 4 weeks. Vertical bars represent the SE of means. Means with the same letter are not significantly different according to Duncan’s multiple range test ( $\alpha \leq 0.05$ ).

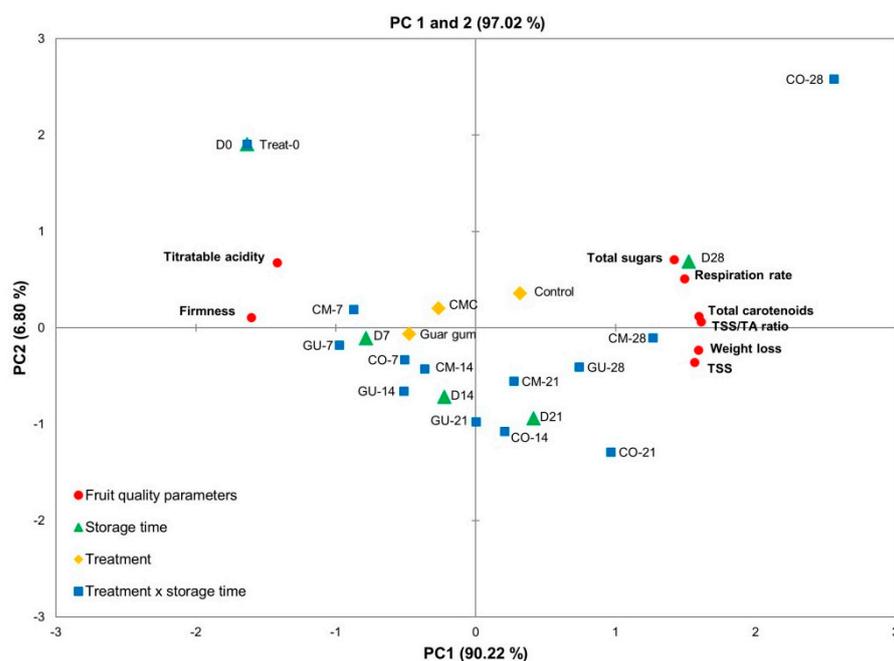
### 3.2.7. Determination of Total Silver Concentration

The CMC-AgNP and guar gum-AgNP emulsions formed a thin layer after coating application on the fruit surface when the water evaporated, leaving silver nanoparticles evenly distributed in the coating matrix [35]. It is worth mentioning that the silver traces in fruit pulp samples did not exist at 14 and 28 days after treatment (data not shown). This result indicates that no nanoparticle penetration occurred through the fruit tissues.

### 3.3. Principal Component Analysis (PCA)

PCA was performed to assess the effect of coating treatments on quality parameters of “Seddik” mango fruits during cold storage (Figure 13). The first two principal components (PCs) represented 97.02% of the total variability (PC1 = 90.22% and PC2 = 6.80%), with respect to coating treatments over the storage period of 28 days at 13 °C. The multivariate space of PC1 and PC2 showed that the fruit quality parameters were differently influenced by treatments during storage as highlighted in the score plot of the first two principal components. Early sampling dates (days 0, 7, and 14) were clearly separated as located on the left side of the PC1 axis, while days 21 and 28 were located on the right side of the same axis. Shifts in the PC average values from negative (days 0, 7, and 14) to positive (days 21 and 28) were observed with advances in storage time for coated and uncoated mango fruits. Uncoated fruits (control) correlated positively with weight loss, respiration rate, TSS, sugar and carotenoid content and negatively with titratable acidity and firmness. Shifting from negative to positive values in PC1, mango fruits showed a general increase in weight loss, respiration rate, TSS, and total sugars combined with a decline in titratable acidity and firmness as key indicators of fruit ripening velocity. Uncoated fruits showed a higher shift in PC score values compared to coated fruits, and lower average PC scores were observed with guar gum-AgNP-coated fruits. Furthermore, the ripening rate in coated fruits after 21 days of cold storage was comparable to that of uncoated fruits at 14 days according to measured quality parameters, indicating that coating treatments

had the ability to delay ripening of “Seddik” mango fruits with more efficiency for guar gum-AgNPs than CMC-AgNPs.



**Figure 13.** Principal component (PC) analysis (PC1: 90.2%, PC2: 6.8%) of the quality parameters of “Seddik” mango fruits over 28 days of storage period at 13 °C. D: day, CO: uncoated fruits, CM: CMC-AgNP-coated fruits, GU: guar gum-AgNP-coated fruits; numbers (0, 7, 14, 21, and 28) refer to the sampling dates.

#### 4. Conclusions

In the present study, synthesized edible nanoparticle coatings were proven to be effective alternative treatments to enhance the postharvest life of “Seddik” mango fruits. CMC and guar gum can be used effectively to improve the stability and mobility of AgNPs. CMC has anionic and reducing properties which can be used as a stabilizing agent for silver nanoparticles. Guar gum is also an excellent stabilizer and a magnificent surface capping agent due to its strong tendency to form hydrogen bonds in water. Therefore, coating with CMC- or guar gum-containing silver nanoparticles affected qualities such as the weight loss, firmness, and respiration rate of mango fruits. The CMC-AgNP and guar gum-AgNP coatings reduced the rate of fruit respiration, which influenced the other parameters of quality during storage at 13 °C compared to the uncoated fruits. Coatings also reduced the loss of mango fruit fresh weight and firmness. Increases in total soluble solids, total sugars, and total carotenoids in the coated fruit were controlled and slowed down. It should be emphasized that no traces of silver were detected in the mango fruit pulp coated with CMC- and guar gum-containing silver nanoparticles. These results suggest that the application of CMC- or guar gum-based AgNP coatings can be used efficiently to retard the ripening and prolong the postharvest life of mango fruit.

**Supplementary Materials:** The following are available online at <https://www.mdpi.com/2311-7524/7/3/44/s1>, Table S1. Linear regression analysis of changes in quality parameters of “Seddik” mango fruit stored at 13 °C; Table S2. Correlation matrix between different quality parameters in “Seddik” mango fruit stored for 28 days at 13 °C. Correlation coefficients were calculated by Pearson correlations at significant levels ( $P \leq 0.05$  and  $P \leq 0.01$ ).

**Author Contributions:** Conceptualization, I.H.; funding acquisition, I.H.; visualization, I.H. and M.A.-E.; project administration, I.H.; investigation, I.H., N.Z., B.M., A.A., and M.A.-E.; methodology, M.A.; resources, A.A. and M.A.-E.; data curation, N.Z. and B.M.; writing—original draft preparation, M.A., N.Z., and B.M.; formal analysis, M.A.-E.; writing—review and editing, I.H., A.A., and M.A.-E.; supervision, I.H. All authors have read and agreed to the published version of the manuscript.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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