**Abstract:** In the current study, novel films with chitosan/nano/SiO$_2$/nisin films and their antimicrobial application on cantaloupe fruit shelf-life have been studied. Novel films were prepared by the addition of 1% chitosan, 1% nano silicon dioxide, and 1% nisin, and freeze-dried for the performance study. Physicochemical properties such as tensile strength, optical, and thermal properties with the performance characteristics of the novel films were measured. Coated and uncoated cantaloupes with various coating solutions were stored and chilled at 4 °C in a relative humidity of 70% for up to nine days. The microbial population measurements have been detected every three days. Results show that the fourier transform infrared intensity (FTIR) of nano/SiO$_2$ and with the addition of nisin (nano/SiO$_2$/n) were higher than chitosan (CH) film except in the wavenumber (3150–3750 cm$^{-1}$) films peaks. Novel nanofilms enhanced tensile strength as well as optical and thermal properties. XRD analysis reported two distinct peak values of 32.08 and 45.99 to correspond to nano/SiO$_2$ orientation (7095) and (3316), respectively. Zeta potential values and turbidity were increased, while the fourier transform infrared intensity (FTIR) of nano/SiO$_2$/n novel film improved the functional properties of coating films, and those bio-nanocomposites are effective in food packaging.

**Keywords:** nano/SiO$_2$ films; performance study; antimicrobial application; cantaloupe; shelf-life

1. Introduction

Nanocomposite films are environmentally friendly polymers which become a part of very essential techniques in various research fields such as for preserving the shelf life of foods in recent years due to imparting new chemical, mechanical, and physical properties [1–4]. Nanomaterials such as nano/ZnO, nano/TiO$_2$, nano/SiO$_2$, and nano/clay are well known in academia and industry for their excellent characteristics such as high rigidity, strength, and slightly toxic [5–10]. Silicon dioxide (SiO$_2$) is known as silica which is found in nature as silica sand, quartz, crystal, vapor-silica, colloidal silica, silica gels, and aerogels. Nano/SiO$_2$ films are widely used in many fields such as antireflection coating [11] and packaging industry against the permeation of gases which prevent fruits,
vegetables, and drinks to be rotten quickly, inhibit the volatile compounds, and maintain the sensorial and physiological qualities. Nano/SiO$_2$ films are considered to have excellent ecological performance due to the uniform emulsion particle size distributions, anti-wear, low-water-absorption, chemically-inert, biocompatible, and friction-reduction properties [12,13]. Furthermore, nano-SiO$_2$ can prevent bacterial attacks in vivo and in vitro [14]. There are several techniques for producing nano/SiO$_2$ films that allow better control of the thickness such as sol-gel, chemical and physical vapor depositions [15], etc. The agglomeration occurs between particles when the particle size is less than 25 nm with a reduction in the electrostatic repulsion between particles [16,17]. Many studies aimed to improve the nano/SiO$_2$ film properties by new techniques and by modifying particles that are highly cost and not environmentally friendly [18]. Nano/SiO$_2$ has been approved for safety rating that will not be digestible by humans [19]. Though, several studies focused on the effect on preservation properties. Shi et al. [20] and Kou et al. [21] used nano/SiO$_2$/chitosan on jujube and longan fruits preservation. Rarely reported studies on the modified characteristics of nano/SiO$_2$ films after the addition of chitosan and nisin which could exert certain effects on microorganisms.

In the current research, novel films prepared by chitosan, nano/SiO$_2$, and nisin were characterized by evaluating the antimicrobial effect on cantaloupe fruit shelf-life extension. Besides, the researchers wish the application may make a reference to a large-scale packaging industry in the future.

2. Materials and Methods

2.1. Materials

The materials used were chitosan (85% deacetylation), acetic acid, glycerol, nisin, and nano/SiO$_2$ (30 nm) from Technolab, SABIC, Riyadh, Saudi Arabia. Fresh cantaloupe fruits with uniform characteristics (color, size, and shape) were obtained from local fruits and vegetable stores in Taif City, Saudi Arabia as experimental materials. Table 1 presents the key elements used for preparing films and evaluating the antimicrobial effect on cantaloupe fruit.

**Table 1.** Structure and properties of the key elements used for preparing films: (a) chitosan (b) nano/SiO$_2$ (c), and nisin for evaluating the antimicrobial effect on cantaloupe fruit.

(a) CAS Registry Number: 9012-76-4
Mol weight: 50,000–190,000 Da (based on viscosity)
Viscosity: 20-300 cP, 1 wt.% in 1% acetic acid (25 °C, Brookfield)(lit.)
Solubility: Dilute aqueous acid: soluble

(b) CAS Registry Number: 7631-86-9
Chemical formula: SiO$_2$
Molar mass: 60.08 g/mol
Density: 2.648 (α-quartz), 2.196 (amorphous) g·cm$^{-3}$
Melting point: 1.713 °C (Amb)
Thermal conductivity: 12 (|| c-axis), 6.8
(⊥ axis), 1.4 (am.) W/(m·K)
Table 1. Cont.

| Property                      | Value                        |
|-------------------------------|------------------------------|
| Solubility                    | Dilute aqueous acid: soluble |
| Melting point                 | 1.713°C (Amp)                |
| Thermal conductivity          | 12 (|| c-axis), 6.8 (⊥ axis), 1.4 (am.) W/(m K) |
| CAS Registry Number           | 1414-45-5                    |
| Molar mass                    | 3354.07 g/mol                |
| Formula                       | C₁₄₃H₂₃₀N₄₂O₃₇S₇             |
| Boiling point                 | 2966 °C                      |
| Classification                | Bacteriocin                  |
| Density                       | 1.402 g/mL                   |

2.2. Films Preparations and Samples Treatments

Chitosan (CH film) solution (1%) was prepared by dissolving with acetic acid (1%) and homogeneously blended with a magnetic stirrer overnight at room temperature. Silica solution film (nano/SiO₂ film) was prepared by dissolving the same amount of (CH film) solution with (1%) nano silicon dioxide, while the third coating film (nano/SiO₂/n film) was prepared by the addition of nisin (1%). Fresh cantaloupe fruits were washed, peeled, cut into similar square shapes, and dipped into various coating solutions for 5 min then allowed to be dry for 30 min. The square pieces were stored in polyethylene bags at 4 °C for evaluations every three days until nine days of storage period. The control samples were dipped into distilled water as a coating film. Films’ solutions were freeze-dried [22] and all the dimensions and weights were detected according to Sami [23–26].

2.3. Scanning Electron Microscopy (SEM)

The morphology of the three novel films was measured after freeze drying and films formation by scanning electron microscopy (SEM) (SHIMADZU, SSX-550, Kyoto, Japan) [4,27].

2.4. Fourier Transform Infrared (FTIR) Spectroscopy Measurements

The FTIR spectra of the novel films were detected within the range 600–4000 cm⁻¹ by using FTIR spectral analysis (Thermo Nexus 470, Nicolet Co., Ltd., Waltham, MA, USA) [28,29].

2.5. Tensile Strength Measurements

The mechanical properties of the novel films were evaluated by a texture analyzer (TA.XT2, Stable Micro System, Surrey, UK) with the help of exponent 32 software and a crosshead speed of 30 mm/min. Films strips were 170 mm long and 13 mm wide, while the thickness was measured with a dial-micrometre at eight random points and the average value was calculated [23,28].

2.6. Optical Measurements

The light transmission and transparency of the novel films were investigated by UV-Vis spectrophotometer (UV-2550, Shimadzu Co., Shanghai, China) at 200–800 nm regions while the air was used as a reference [10,27].
2.7. Thermal Measurements

The thermal behavior of the novel films was measured in triplicate by a differential scanning calorimeter (DSC-6000, PerkinElmer, Singapore). Aliquots of 15 mg were placed into standard aluminium pans, sealed, and scanned between 30 °C to 150 °C with a heating rate of 10 °C/min while the empty pan was used as a reference [9,30].

2.8. X-ray Diffraction (XRD) Measurements

The XRD of the novel films were tested by using an X-ray diffractometer (Bruker D8, Karlsruhe, Germany) in the range from 5° to 50° with Cu and Kα radiation (working voltage, 40 kV; current, 40 mA) [9,31].

2.9. Turbidity, Partial Size, and Polydispersity Index (PDI) Measurements

Film solutions were diluted 40 times in PBS (0.01M, pH 7.0) and determined at 600 nm by a spectrophotometer (UV-2550, Shimadzu Co., Shanghai, China) with a blank of PBS [9]. Various solutions were scattered in water as 0.04 wt% to determine the partial size and PDI [32].

2.10. Zeta Potential and Contact Angle Measurements

The zeta potential of the novel films was determined by using a zeta potential analyzer (Zetasizer, Nano-ZS90, Malvern, UK). Film solutions were diluted 50 times in PBS (0.05M, pH 7.0) by (10–100 runs/analysis) after the equilibration for 120 s at 25 °C [9,33]. A drop of colorful distilled water was put onto the surface of the film with an injection 24-gauge flat-tipped. The contact angle (degree) was evaluated by a digital camera (Sony, Model F707, Shanghai, China). The images were an average of eight measurements of each film and processed by Adobe Photoshop 6.0 software to calculate the air-water contact angle measurement [34].

2.11. Antimicrobial Measurements

The measurements of yeast and mold count were carried on different days (0, 3, 6, and 9) during the storage period. A chloramphenicol glucose agar (CGA) (ISO 21527, 2008) was used as a medium for cantaloupe fruits. To aseptically prepare 1:10 dilutions, 10 g of cantaloupes was added to a stomacher bag with 90 mL of sterile 0.1% peptone water. Samples were blended, stomached, diluted, and plated in triplicate. Plates were incubated at room temperature (20–25 °C) for 3 days. The obtained microbial colonies were counted using the AOAC official method 997.02 [35].

2.12. Statistical Analysis

ANOVA and Tukey’s post hoc tests were used for the performance study of nano/SiO₂ films for the antimicrobial properties on fresh-cut-cantaloupe fruits shelf-life at multiple-range test (p ≥ 0.05) to compare the differences. Statistical analyses were conducted by SPSS ver. 20 (SPSS Inc., Chicago, IL, USA) to analyze the variances.

3. Results and Discussion

3.1. SEM Study

As shown in Figure 1, it presents top-view SEM images of the novel films; CH film (a), nano/SiO₂ film (b), nano/SiO₂/n film (c). Figure 1a shows the typical character of the chitosan film in good dispersion and distribution uniformity. Nano/SiO₂ film reported irregular surface aspect reveals and was nonhomogeneous in white molecules which suggested a surface reaction of nano silicon dioxide with chitosan component (Figure 1b). Arlet et al. [36] reported a significant density in silicon films with limited inhomogeneity. We noticed a rough layer with grain sizes in nano/SiO₂/n film which influences the crystallization (Figure 1c). From these images, it is observed that the nisin addition process leads to non-uniform porous thin films, roughness, agglomeration problem, and poorer particle distribution compared to the original films.
3.2. FTIR Spectroscopic Study

FTIR presents the interaction between the raw materials of the novel films that may affect the characteristics of the films at 600–4000 cm\(^{-1}\). FTIR spectra of the cross-linked CH film and nano/SiO\(_2\) films are presented in Figure 2. FTIR patterns were relatively similar in all the novel films with several differences in CH film. When the nano-SiO\(_2\) was added, the strong absorption band was transferred to the high wavenumbers. The intensity of nano/SiO\(_2\) film peaks was higher than CH film only, which was perhaps due to the nano-material Si—O—Si resonance mode of vibrations [15]. Consequently, when the 1% nisin was added, the peak did not change except in the wavenumber (3150–3750 cm\(^{-1}\)) due to the nisin chemical structure. According to Figure 2, it was noticed that the major peaks associated were at 1027.39, 1348, and 1558.2 cm\(^{-1}\). The FTIR results preserved that the compatibility between 30 nm nano-SiO\(_2\) and nisin would be scattered uniformly during the film formation and presented excellent miscibility.

![FTIR spectra deposited for different novel films.](image)

3.3. Tensile Strength Analysis

The tensile strength of the novel films with nano-SiO\(_2\) is presented in Figure 3. The nano/SiO\(_2\)/n film showed lower tensile strength (2.62 MPa) than that CH and nano-SiO\(_2\) films. The matrix of the films might be strengthened by the incorporation of nano-SiO\(_2\) by electrostatic attraction or hydrogen bonding [27,37]. The maximum tensile was obtained for CH and nano-SiO\(_2\) films, thus enhancing the interphase adhesion strength inbetween the nano-SiO\(_2\) and chitosan components. The finding was in agreement with the SEM study results and with the previous study of chitosan-silica hybrid porous membranes by Pandis et al. [38].
3.3. Tensile Strength Analysis

The tensile strength of the novel films with nano-SiO$_2$ is presented in Figure 3. The nano/SiO$_2$/n film showed lower tensile strength (2.62 MPa) than that of CH and nano-SiO$_2$ films. The matrix of the films might be strengthened by the incorporation of nano-SiO$_2$ by electrostatic attraction or hydrogen bonding [27,37]. The maximum tensile was obtained for CH and nano-SiO$_2$ films, thus enhancing the interphase adhesion strength between the nano-SiO$_2$ and chitosan components. The finding was in agreement with the SEM study results and with the previous study of chitosan-silica hybrid porous membranes by Pandis et al. [38].

![Figure 3. Tensile strength analysis for different novel films.](image)

3.4. Optical Properties

The study of the optical properties for the novel films is extremely functional for the elucidation of the electronic structure of those materials. Figure 4 presents the optical absorption spectra of the novel films as a function of dopant concentration annealed at 200–800 nm regions. The result reported that CH film exhibited the lowest absorption, while with nano-SiO$_2$ and nisin additive, it would be seen that the optical absorption range becomes higher and more intense especially at a wavelength of 200–388 nm, where an intense absorption was observed. It could be due to the oscillation of electrons of silica nanoparticles in resonance with the light wave radiations [39,40].

![Figure 4. Optical absorption of thin novel films at 200–800 nm regions.](image)

3.5. Thermal Stability Properties

The thermal stability of novel films was evaluated by thermogravimetric analysis, Figure 5. The results showed that nano-SiO$_2$ films were unable to alter the thermal behavior, therefore inducing better thermal stability of the other films. The weight of the silica decreased slightly with increasing temperature (>100 °C) due to the decomposition and the oxidation of methyl groups (ACH$_3$) on the surface of the nano-silica [41]. It was noticed that the addition of nisin preserved the thermal stability of the nano/SiO$_2$/n film. While the decomposition temperature varies with different reagent ratios, CH film and nano/SiO$_2$/n film reported few similar values, especially from (30–115 °C). These results were in agreement with the tensile strength analysis of the current study.

![Figure 5. Thermal curves of thin novel films between 30 °C to 150 °C.](image)
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![Figure 5. Thermal curves of thin novel films between 30 °C to 150 °C.](image)

3.6. XRD Analysis

XRD characterizes the compatibility of films. The XRD patterns of the novel films at 5 °C to 50 °C are presented with the characteristic peaks in Figure 6. In general, nano-silica has been structured in an amorphous phase when the noncrystalline and crystalline ingredients in the films exhibit good miscibility [10]. Nano-SiO$_2$ films showed that the diffraction peak weakened with an increase in size and was easier to reunite. Two distinct peak values of 32.08 and 45.99 correspond to nano/SiO$_2$/n film orientation (7095) and (3316), respectively. Those peaks turn out to be narrower and more intense with increasing temperature. High temperature elevated for nano/SiO$_2$ film, implying the phase transformation of intermediate Si—O—Si structure [39]. These results supported the conclusion drawn from the SEM of the current study.

![Figure 6. XRD patterns of novel thin films.](image)
3.7. Performance Characterizes

Turbidity, polydispersity index, zeta potential, contact angle, and partial size distribution were measured and presented in Figure 7. Nano/SiO$_2$/n film recorded the highest turbidity 0.41 due to the presence of nisin, while CH film recorded the lowest, which might be due to the steric hindrance and sufficient electrostatic, Figure 7a. Polydispersity index of the novel films ranged from 0.41 in nano/SiO$_2$ film to 0.68 CH film, Figure 7a. O’Callaghan and Kerry [42] reported that the distribution is regarded as broad when the polydispersity index $\geq 0.5$, while the ideal formulation is regarded as monodispersed for $\leq 0.3$ [42]. The zeta potential is known as the repulsion degree among the adjacent particles in a dispersion [43]. The zeta potential trend for the novel films indicated a positive overall load. The zeta potential (>30 mV) is considered stable due to enough repulsive force present for avoiding the aggregations among particles [42]. According to Figure 7b, nano/SiO$_2$ and nano/SiO$_2$/n films reported that the nanoparticles showed high stability, 44.17 and 46.11 mV, respectively. The higher zeta potential value may be attributed to the higher number of NH$_3^+$ groups. That can clarify the increase in the nano/SiO$_2$ with the combination of chitosan ratio reporting high zeta potential value [44]. It is known that the wetting property is an essential factor in adhesion, and wettability applications, which is influenced by the various materials [41]. The measured reactions of the contact angle among the novel films are illustrated in Figure 7b. CH film recorded 78.56°, while the addition of nano-silica decreases the hydrophobicity of the film surface by 80.07°; the addition of nisin reported better hydrophobicity than both CH and nano/SiO$_2$ films. Changes in contact angle could be due to the attachment of the selected nano-molecules and nisin. The partial size distribution is one of the mainly significant parameters because it evaluates the destination and application of the formulated biocomposite [43,44]. Syamdidi et al. [45] reported that nanoparticles are considered as solid particles with a range size from 10 nm to 1000 nm. Figure 7 presents the partial size distribution of the novel films which ranged from 567.86 nm to 2511.43 nm. The results reported a decrease in the response by increasing the nano-molecules and nisin ratio. Chitosan/nano/SiO$_2$ ratio is a vital factor during the ionic gelation, crosslinked particles, and agglomeration, that might describe the detected trend [45,46].

![Figure 7. Cont.](image-url)
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Figure 7. Performance characterizes of thin novel films; (a) turbidity and polydispersity index; (b) zeta potential and contact angle; (c) partial size distribution.

3.8. Antimicrobial Application on Cantaloupe Fruit

Coating treatments on cantaloupe fruits were done according to the method of Sami et al. [16]. Cantaloupes were evenly dipped into various coating solutions, distilled water, 1% chitosan, 1% nano silicon dioxide, and 1% nisin and chilled at 4 °C on the relative humidity of 70%. After incubation at the ambient conditions (20–25 °C) for up to nine days, the measurements have been detected every three days. An increase in the occurred microbial population may be due to the process of slicing and the temperature condition during storage [47]. Besides, high acidity and sugar in cantaloupe fruits can enhance the microbial attachment during the storage period [1,5,48]. To avoid the survival of the microorganisms, one should take preservation methods, coating assays, and storage conditions under significant consideration [49]. The findings of the current study presented that the population reductions of approximately 0.59, 0.56, 0.54, and 0.51 log CFU/g were detected after coating treatments with distilled water only, chitosan, chitosan/nano/SiO<sub>2</sub>, and chitosan/nano/SiO<sub>2</sub>/nisin, respectively, on day zero. Yeast and mold counts increased in all treatments as the storage period increased.

Table 2 shows a lower quality of the cubes in control samples of the fresh-cut cantaloupe fruits and reported the highest yeast and mold counts (2.94 log CFU/g) on day nine of storage compared to the other coated samples throughout the whole storage. CH samples were a little effective in maintaining the yeast and mold counts below 2.67 log CFU/g on day nine of storage. The coating treatments with chitosan/nano/SiO<sub>2</sub>, and chitosan/nano/SiO<sub>2</sub>/nisin both reduced the yeast and mold counts 2.49 and 1.92 log CFU/g, respectively, on day nine. Chen et al. [50] achieved a similar reduction of microbial activity, especially in Salmonella spp., by coating with chitosan and nisin. The current finding established that chitosan/nano/SiO<sub>2</sub>/nisin treatment would inactivate the microbial growth, thus prolonging the shelf-life of the treated samples for nine days longer relative to the uncoated fresh-cut cantaloupe fruits.
Table 2. Effects of novel coating on yeast and mold counts (log CFU/g) on cantaloupe fruits during the storage period.

| Days | Control | CH Film | nano/SiO$_2$ Film | nano/SiO$_2$/n Film |
|------|---------|---------|-------------------|---------------------|
| 0    | 0.59 ± 0.29 $^a$ | 0.56 ± 0.25 $^b$ | 0.54 ± 0.21 $^c$ | 0.51 ± 0.22 $^d$ |
| 3    | 1.32 ± 0.31 $^a$ | 1.02 ± 0.25 $^b$ | 0.99 ± 0.37 $^b$ | 0.81 ± 0.08 $^c$ |
| 6    | 1.93 ± 0.17 $^a$ | 1.45 ± 0.22 $^c$ | 1.78 ± 0.31 $^b$ | 1.21 ± 0.40 $^d$ |
| 9    | 2.94 ± 0.31 $^a$ | 2.67 ± 0.26 $^b$ | 2.49 ± 0.40 $^c$ | 1.92 ± 0.31 $^d$ |

Different small letters within the same line $^{a,b,c,d}$ mean significant differences between treatments at $p \leq 0.05$.

4. Conclusions

In the current study, nano/SiO$_2$ films (30 nm) and their antimicrobial application on cantaloupe fruit shelf-life have been studied. Introduction of the nanoparticles enhanced tensile strength as well as optical and thermal properties of the films made from silicon dioxide. Zeta potential and turbidity values were increased, while nano/SiO$_2$ films decreased the hydrophobicity of the film surface against chitosan films. The retrieved investigated application on cantaloupe fruit shelf-life reported that the nano-silica/nisin coating treatment was effective for population reductions of yeast and mold counts during storage. The results established that the bio-nanocomposites based on nano/SiO$_2$ and nisin can have potential applications in the mechanical fields, and food preservation techniques.

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