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Biomass derived cellulose nanofiber loaded PVA-nanocurcumin coating for extending the shelf life of Mandarin oranges (*Citrus reticulata*)

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ABSTRACT

The concern over agricultural waste disposal can be resolved by converting them into value-added products. Here, sustainable fruit coatings and films have been developed *via.* wealth from waste concept. The cellulose nanofibers (CNF) have been extracted from the waste onion skin. The isolated CNF has been characterised using scanning electron microscopy (SEM), transmission electron microscopy (TEM), optical microscopy (OM), dynamic light scattering (DLS) and solid-state ^{13}C NMR spectroscopy. A combination of cellulose nanofiber (CNF) and nanocurcumin in polyvinyl alcohol (PVA) matrix has been used for the fabrication of coating over fresh mandarin oranges. The effect of nanocurcumin in PVA-CNF nanocomposite on the post-harvest maintenance of mandarin oranges has been investigated. The morphology of the prepared films is studied by SEM and atomic force microscopy (AFM). The quality of the orange fruits coated using the above formulation by dip coating method was analysed *via*. weight loss analysis, total soluble solid (TSS), pH, titrable acidity (TA) and antioxidant activities. It was found that the nanocurcumin incorporated PVA-CNF coating was effective in reducing mass loss and maintaining the physiochemical properties of oranges in comparison with other formulations. The developed method could be extended to use as food packaging films, which would be a sustainable solution for agricultural waste valorisation and plastic pollution. Through this research work, we propose an environment friendly approach for the preservation of fruits and vegetables.

1. Introduction

Fruits and vegetables are mandatory for a healthy human diet as it contains essential vitamins, minerals and other nutrients prescribed in the dietary guidelines worldwide [1]. However, it was estimated that 20–30 % of fresh products are wasted during the post-harvest storage due to their perishable nature [2]. In addition, global climate changes play a vital role in the loss of horticulture products. This results in the increasing demand and thereby heavy rise in retail price of fruits and vegetables which makes them inaccessible to some population [3]. Cold storage is an effective way to delay the postharvest decay [4]. However, it is not an all-time convenient approach. A good packaging can help in the extending the shelf-life of post-harvest fruits and veggies. There are serious issues associated with the non-biodegradable packaging materials like lack of recycling and leaching of harmful chemicals to food and soil, which create concerns over human health and the ecology [5]. Also, the pollutants from plastic industries like diethylhexyl phthalate, cadmium, mercury and lead are toxic carcinogens as well as cause hormonal disorders in humans [6]. As a path of green chemistry, material science researches are now focusing on the use of eco-friendly and biobased materials for the development of the packaging system and is a promising alternative to non-biodegradable packaging [7,8]. Moreover, the ability to carry active compounds, including antioxidants [9] and antimicrobials [10] within the film matrix, enables the edible film and coating to enhance the safety and shelf life of food products [11].

Biobased materials mainly include polysaccharides, proteins, lipids

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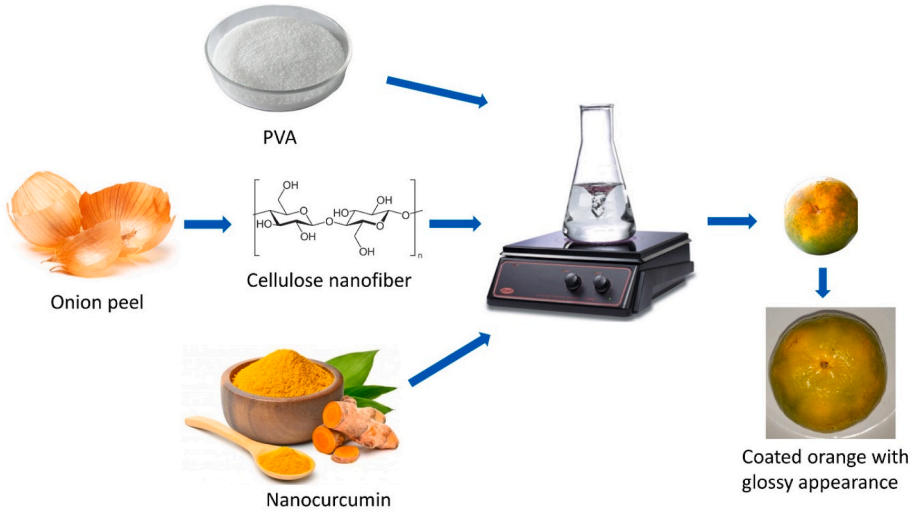

Fig. 1. Schematic representation of preparation PVA-CNF-nanocurcumin coatings for orange.

and their composites. Composites are more preferred since any one of the biopolymer alone might not offer the desired properties for the coating film like antimicrobial, antioxidant, low gas-water permeabilities, mechanical strength, appealing appearance etc [12]. In recent years cellulose nanofibers (CNF) are getting prime focus in the area of edible coating due to its availability and non-toxicity. CNF developed from carrot pomace has been used as an effective coating for bananas [13]. Still, CNF alone limits its application as edible coating especially in humid places and hot regions like India due to its high hydrophilic nature, poor water resistance properties and poor thermal stability [14]. Hence CNF can be incorporated in other matrices to improve its mechanical, optical and barrier properties [15]. A composite of chia seed mucilage containing 0.6 and 8.0 wt % CNF was found effective in maintaining the postharvest quality of strawberries [16]. Combination of sago starch with 9 % CNF edible coating delayed the deterioration and hence the shelf-life of bananas at room temperature [17].

PVA is a biodegradable, non-toxic synthetic polymer with odourless, tasteless properties and form soft transparent film [18]. Its efficacy to form edible films has already been reported in coatings for lemon [19], strawberries [20] and golden delicious apple [21]. A combination PVA and CNF extracted from coconut waste, together with essential oil is used to fabricate bio-degradable food packaging film with enhanced hydrophobicity, anti-microbial and antioxidant properties was reported recently by Arun et al. [22]. The edible coatings act as a carrier for active materials include curcumin [23–25] and essential oils [26–29] that enhance the fruit quality. Encapsulation of curcumin in the lipid-polymer hybrid nanoparticles dispersions act as a good edible coating for fruits and vegetables with anti-cancer properties, which is proven *via in vitro* studies in human cells [30]. Nanocurcumin, extracted from turmeric, is well known for its anti-microbial, anti-oxidant, anti-inflammatory, anti-fungal and cancer chemopreventive properties [31–33]. India is the largest producer of turmeric worldwide and is considered as an ancient medicine from long ago [34].

Mandarin oranges (*Citrus reticulata*) are the 3rd most cultivated crop in India which are exported worldwide and are rich in vitamin A, B and C, phosphorous along with digestive fibres. These citrus varieties can be stored up to a maximum of 14 days in cooler conditions which is not practical in tropical Indian markets. So additional storage settings have to be implemented for increasing the shelf-life of oranges while transportation and marketing. In the current investigation, Mandarin oranges has been coated using a PVA–CNF–nanocurcumin formulations to increase their self-life. Herein, CNF has been prepared from a biowaste, discarded onion peel, so as to target a cost-effective waste management in agriculture. The extracted CNF has been characterised using transmission electron microscopy (TEM), scanning electron microscopy (SEM), optical microscopy and ^{13}C NMR Spectroscopy. Nanocurcumin has been extracted from turmeric powder. The edible coating formulations such as PVA, PVA-CNF and PVA–CNF–nanocurcumin composites have been developed and the coated oranges were investigated for their quality during preservation *via.* weight loss analysis, total soluble salt (TSS), pH, titrable acidity etc. This work also focuses on the production of value-added products from waste materials. Furthermore, giving insight into the fruitful utilization of waste products to fabricate essential materials for food preservation.

2. Materials and methods

2.1. Materials

NaOH, hydrogen peroxide (H_2O_2), oxalic acid and ethanol were purchased from Sigma Aldrich (all of analytical grade). PVA (Mowiol 18–88; Mw∼130,00) was obtained from Sigma Aldrich. Raw onion peels were received from Chinnakkada market, Kollam, Kerala, India. Raw turmeric (*Curcuma longa*) obtained from local market (Kollam, Kerala).

The Indian mandarin oranges (*Citrus reticulate*) of similar size and shape with no visible defects were collected from a local market, Kollam, Kerala. A total of 20 oranges were collected and divided into four sets of five in each group. The collected oranges were thoroughly washed with water and pat dried followed by air drying. One set was kept as control and other 3 were investigated for the effect of 3 different coating formulations.

2.2. Preparation of CNF

For the preparation of CNF, the cleaned onion skins were dried and grinded into fine powder. 100 g of the dried onion skin were treated with 2 % NaOH solution in an autoclave at a temperature and pressure of 121 °C and 15 psi respectively for 15 min. This process was repeated for 3 times. After washing, the steam exploded fibers were bleached using a mixture of 10 % NaOH and 15 % H_2O_2 using a magnetic stirrer. The bleaching was repeated 3 times and the fibers were washed with distilled water. Later it was treated with 1000 mL of 5 % oxalic acid in an autoclave at a temperature of 121 °C and a pressure of 15 psi for 15 min. Two more subsequent cycles were performed. The fibers were then washed with distilled water to neutralize the pH. Finally, the obtained fibers were homogenized with distilled water using a homogenizer (D lab made) to get uniform nanocellulose suspension.

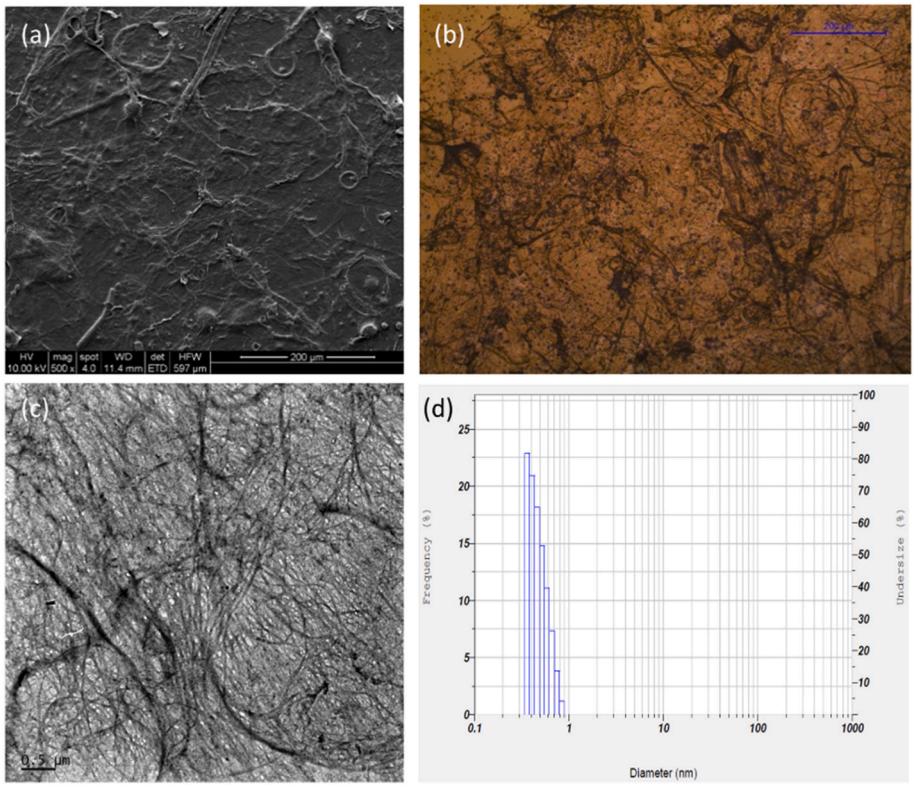

Fig. 2. a) SEM image **b)** optical microscopic image, **c)** TEM image and **d)** DLS size distribution of cellulose nanofibers extracted from onion skin.

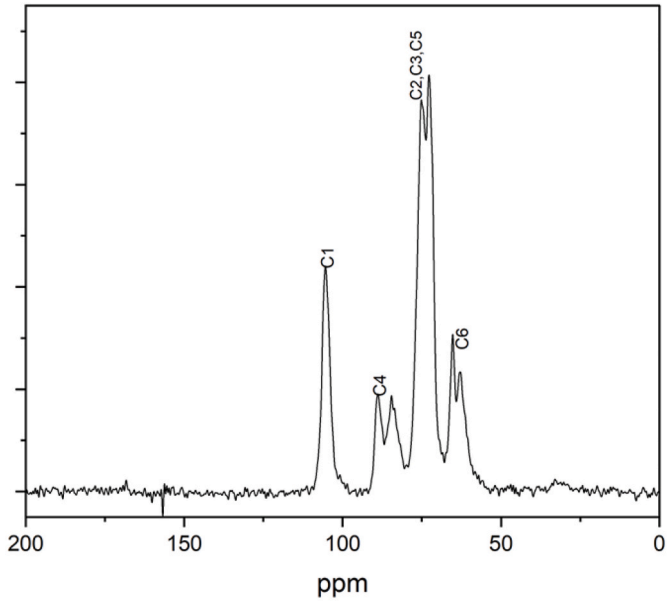

Fig. 3. ^{13}C NMR spectra of extracted cellulose nanofibers from onion skin.

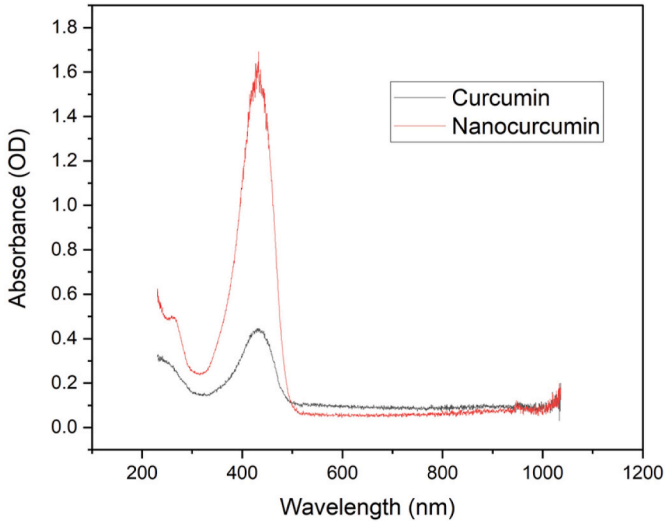

Fig. 4. UV–Visible spectrum of curcumin and nanocurcumin.

2.3. Preparation of nanocurcumin from turmeric powder

Raw turmeric (Curcuma longa) was dried and grinded into fine powder. 2 g of turmeric powder was weighed and dissolved in 10 mL ethanol followed by ultrasonication (GT Sonic®) for 30 min. The resultant solution was allowed to evaporate to get the curcumin particle. Formation of curcumin was confirmed by thin layer chromatography. The spot test was carried with a mixture of chloroform and ethanol in the ratio($CHCl_3$: C_2H_5OH) 9.5 :0.5 against pure curcumin obtained from sigma Aldrich. Also, red colour on treatment with H_2SO_4 is an assertion for the formation of curcumin [33].

The developed curcumin was dissolved in 50 % ethanol in 1:10 ratio followed by ultrasonication for 40 min to prepare nanocurcumin suspension. The formation of nanocurcumin was confirmed by UV–Visible spectroscopy.

2.4. Preparation of coating formulations

PVA (5 wt%) solution was prepared in distilled water with magnetic stirring (IKA-RET basic) at 60 °C for 5 h at 1200 rpm. One set of oranges were dipped in the PVA solution for 2 min and allowed to air dry.

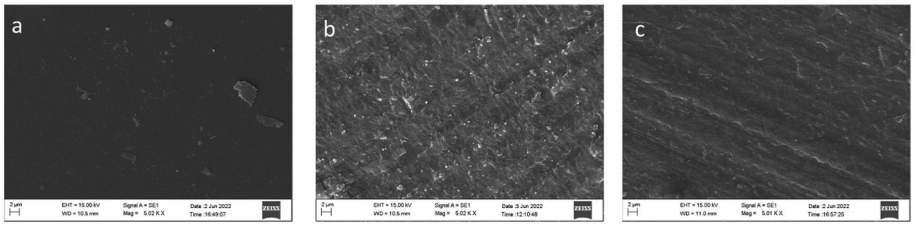

Fig. 5. SEM images of **a)** Neat PVA **b)** PVA-CNF **c)** PVA–CNF–Nanocurcumin films.

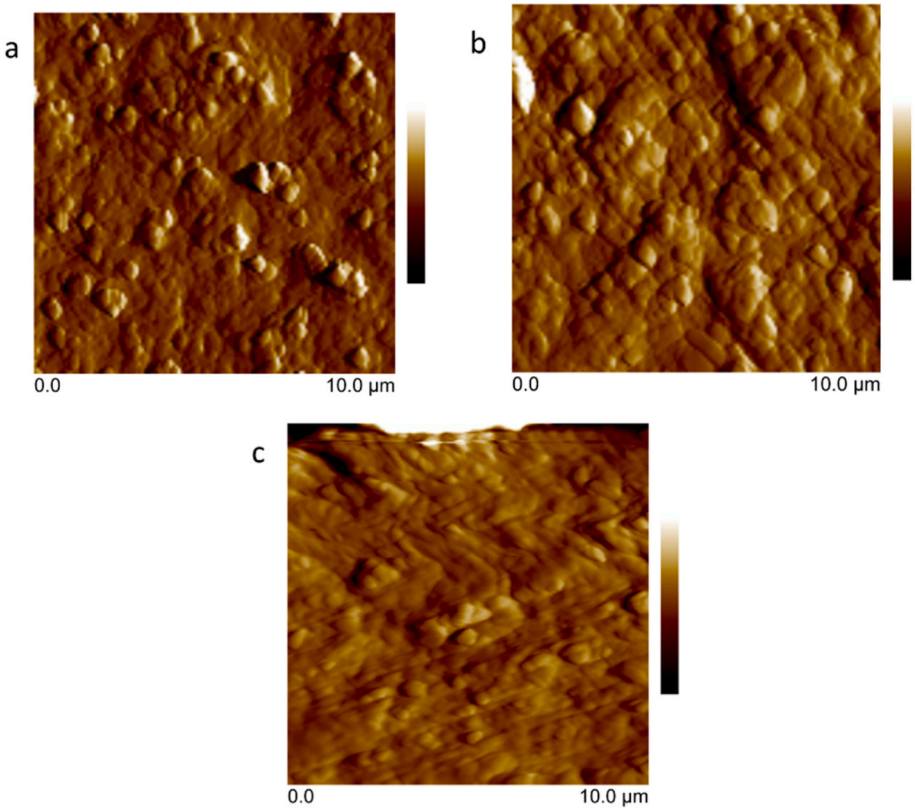

Fig. 6. AFM images of **a)** PVA, **b)** PVA-CNF and **c)** PVA–CNF–Nanocurcumin films.

PVA-CNF solution was prepared by adding 2 wt% CNF into PVA solution, followed by stirring at room temperature for 30 min. The obtained mixture was further ultrasonicated for 30 min. Another set of oranges were dipped in this solution, air dried and stored in room temperature.

PVA–CNF–Nanocurcumin coating formulation was fabricated by adding 0.05 % (w/v) nanocurcumin dispersion (50 % (v/v) ethanol) into PVA-CNF blend. The resulting solution was stirred for about 30 min followed by ultrasonication for the complete dispersion of the constituent particles. Third set of oranges were dipped in the above solution, air dried and kept at room temperature for the further physio-chemical observations in the interval of 4 days for two weeks. Parallelly, all the compositions were made into film for evaluating the film properties. The whole set up of the experimental procedure is pictorially represented in Fig. 1.

2.5. Characterisation of the extracted CNF

2.5.1. Optical microscopy

The morphology of the extracted nanofibers was analysed using Leica DMLP polarising optical microscope. Few drops of the CNF dispersion were placed in between two cover glass and allowed to air dry in an overnight. The images were captured at room temperature in unpolarized light in reflected light mode.

2.5.2. Transmission electron microscopy

Morphology of the extracted CNF was studied by JEOL 2100 Transmission Electron Microscope with an operating voltage of 200 KV. CNF dispersion was casted on the Cu grid and dried in an overnight.

2.5.3. Dynamic light scattering

Particle size distribution of the nano fibre suspensions was examined by dynamic light scattering in Horiba Scientific SZ-100, all the measurements were carried out at pH-7.

2.5.4. ^{13}C NMR

^{13}C NMR spectroscopic analysis of the extracted CNF were carried out to evaluate the purity and molecular structure of the cellulose nanofibers extracted from onion skin. 400 MHz Solid state ^{13}C NMR spectra of the dry film of cellulose nanofiber were recorded.

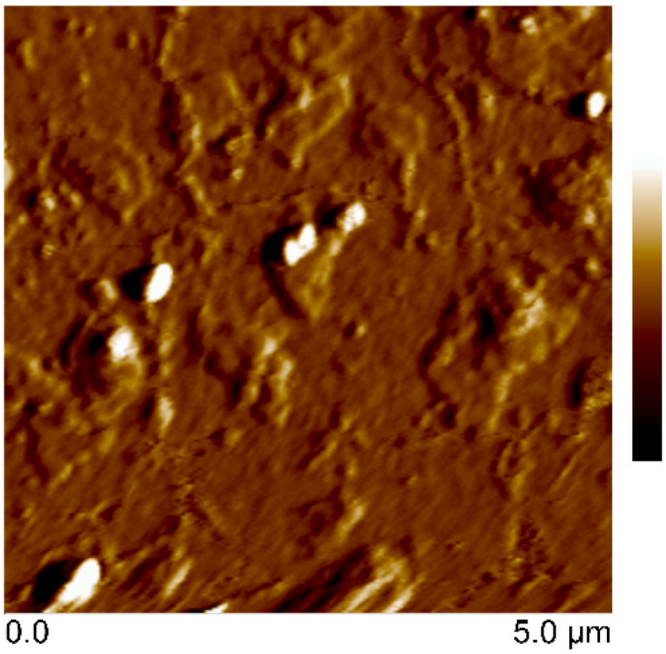

Fig. 7. AFM image of PVA–CNF–Nanocurcumin films in higher magnification.

2.6. Characterisation of coating films

2.6.1. Scanning electron microscopy

The morphologies of the coating film were examined using a scanning electron microscope (SEM) (Carl Zeiss EVO 18 Research). To examine the morphology of samples, the films were made into small pieces and placed on the aluminium mount with carbon conductive tape and coated with a 15 nm layer of gold using a Quorum SC 7620 sputter coater. Excessive charge accumulation can be avoided by making carbon tape bridges for each samples. Analyses were performed using a 120 µm aperture and 15 keV acceleration voltage. The working distance depended on the sample height and was chosen to achieve approximately 10 mm.

2.6.2. Atomic force microscopy (AFM)

The topography of the coating films was studied using an Atomic Force Microscope (Bruker Dimension Edge SPM) in contact mode of operation.

2.6.3. Testing of transparency

The films were cut in equal dimensions and placed over a hand written text and tested for the transparency of the film through the visibility of the text under the film.

2.7. Fruit quality assessment

2.7.1. Weight loss analysis

Weight loss analysis of oranges were done by measuring the weights before and after coating using a digital balance. The initial weight of oranges after coating is taken as m_0 and weights are measured during the storage, which has been remarked as m_i. Thus, weight loss is calculated by the equation;

$$\text{Weightloss}(\%) = \frac{(m_0 - m_i)}{m_0} \times 100$$

2.7.2. Determination of pH

The pH of juice of all orange samples obtained from uncoated control, fruits coated with PVA and PVA-CNF and PVA–CNF–Nanocurcumin solution was determined using digital pH meter (MKVI). The juice of each sample was collected at an interval of four days and measured the pH.

2.7.3. Total soluble solid (TSS)

The total soluble solid of all orange samples were measured using hand refractometer (RHB-32ATC) and expressed in Brix° (0-55°) software. The experiments were carried at 30 °C and replicated five replications for each treatment.

2.7.4. Titrable acidity (TA)

For the determination of acidity, 1 mL sample was taken on a conical flask and 9 mL of distilled water was added. The solution was titrated against standard 0.1 N NaOH using phenolphthalein as indicator (pH = 8.2 ± 0.1) and TA was calculated by the equation.

$$TA = \frac{V \times 0.1\ N\ NaOH \times 0.064 \times 100}{m}$$

Where, 'v' is the volume of NaOH, 'm' is the mass or volume of the orange juice and '0.064' is the milli equivalence of citric acid.

2.7.5. Antioxidant activity

The determination of antioxidant activity was done according to the Blois (1958) method of DPPH (2,2,-diphenyl-1-picrylhydrazyl) scavenging [35]. The main antioxidants present in fruits and vegetables are phenolic compounds and ascorbic acids [36]. Stock solution of the orange juice extracts was prepared to the concentration of 1 mg/mL. 100 µg of each extract were added, at an equal volume, to methanolic solution of DPPH (0.1 mM). The reaction mixture is incubated for 30 min at room temperature, the absorbance was recorded at 517 nm. Ascorbic acid was used as the reference compound. Higher antioxidant activity of the reaction mixture indicates the lower absorbance value which decreases with higher free radical-scavenging activity [37].

3. Results and Discussion

3.1. Morphological characterisation of extracted CNF

An important factor in finding the applicability of fibers in composites is determining its morphology. The structural features and morphology of the cellulose nanofiber extracted from onion skin were studied using SEM, OM and TEM analysis. Fig. 2a, b and c represent the SEM, OM and TEM images of the onion skin nanofibers respectively. The fibrous morphology of the isolated nanocellulose was well evident from all the micrographs. From the SEM image, it was clear that the fibers existed as entangled cellulose fibers as bundles. A similar observation

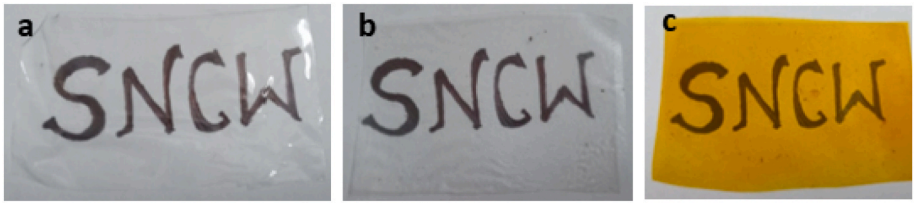

Fig. 8. The transparency of the **a)** PVA, **b)** PVA-CNF and **c)** PVA–CNF–Nanocurcumin films.

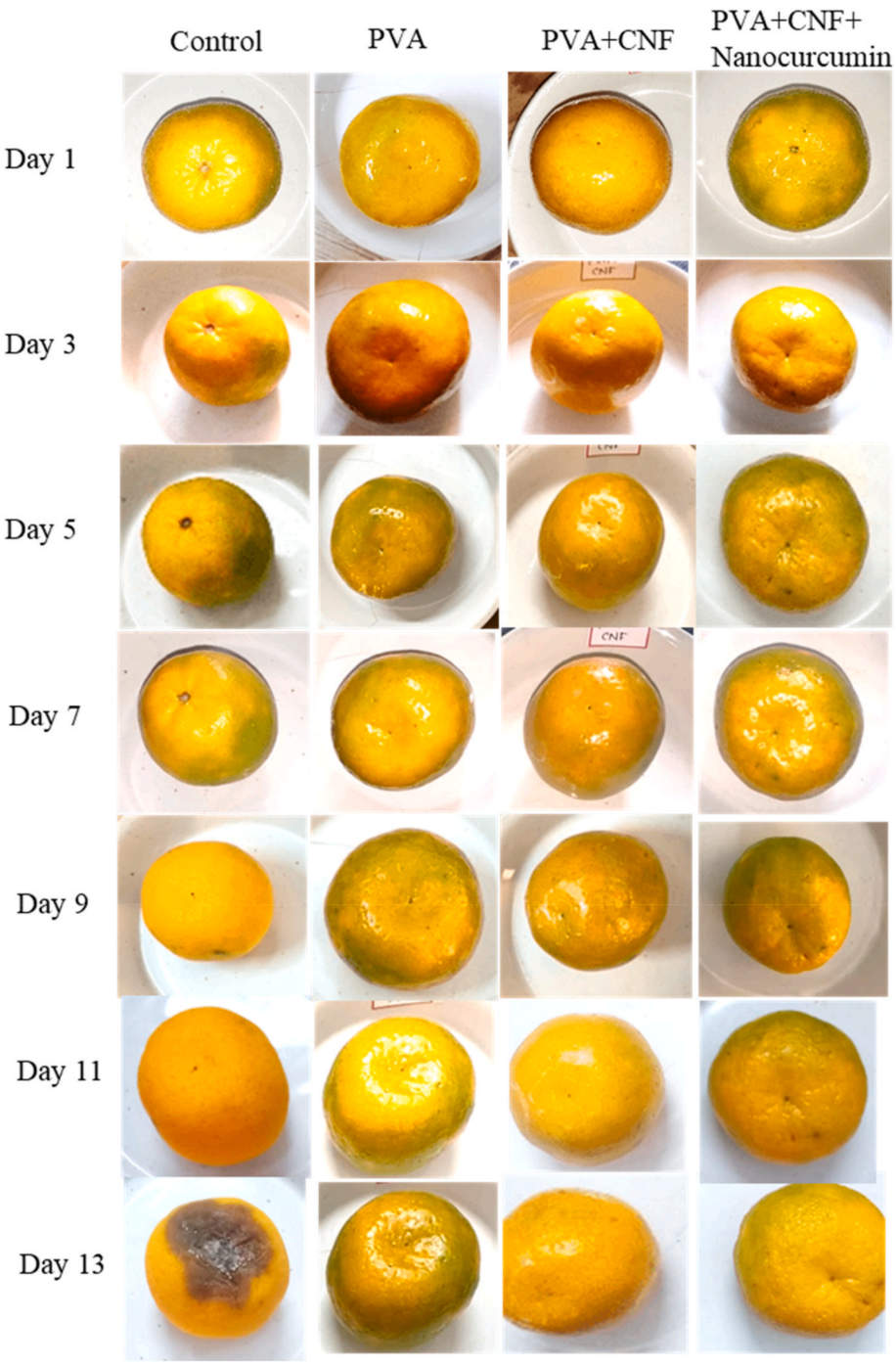

Fig. 9. Physical appearance of controlled and coated oranges for preservation up to 13 days of storage.

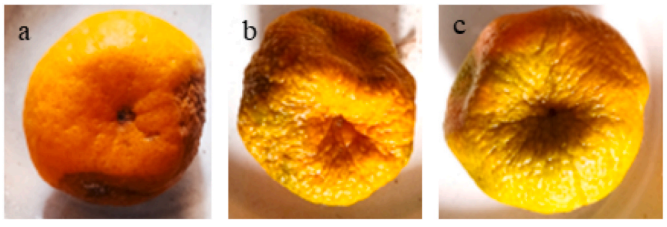

Fig. 10. Physical appearance of **a)** PVA **b)** PVA-CNF and **c)** PVA–CNF–Nanocurcumin coated oranges for preservation at the 20thday of storage.

was described by Guerrero et al. [38]. The notable irregular and rough surface figured in the SEM picture specifies the exclusion of non-cellulosic components from the onion skin fibers [39]. Guptha et al. [40] and Moreno et al. [41] also deduced a similar conclusion on isolating nanocellulose from amla pomace and garlic skin respectively. The optical microscopy image also depicted the fibrous nature of the nanocellulose with some aggregates. Due to the presence of surface hydroxyl groups on the cellulose nanofibers, effective hydrogen bonding takes place and accordingly aggregates were formed [42]. Even though SEM and OM images depict the structural features of the nanocellulose, the exact dimension and morphology of them was obtained from the TEM image. A network of nanofibers was revealed from TEM image. Chen et al. [43] and Xiao et al. [44] presented a comparable TEM image.

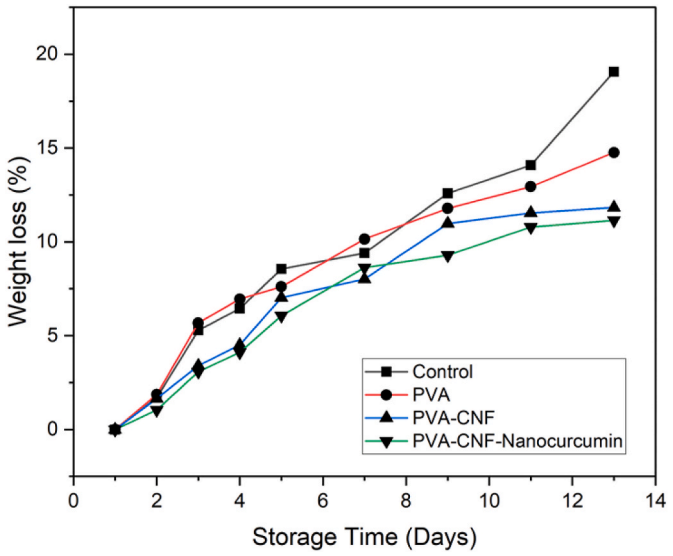

Fig. 11. Weight loss of control, PVA, PVA-CNF and PVA–CNF–Nanocurcumin coated oranges during the storage of 13days at room temperature.

The study of Naduparambath et al. [45] concluded that the self-assembled networks of nanocellulose stem from the diminished repulsive forces due to the effective strong hydrogen bonding among the cellulose molecules. This specific structural attribute of the nanofibers revealed its enhanced hydrogen bonding and trustworthy reactivity [46]. From TEM analysis, it was found that the fibers have a diameter of 18–25 nm. Rashid et al. [47] and Chen et al. [46] reported nanocellulose from short grain rice husk and lotus leaf stalks with a similar diameter of 11–28 nm and 20 nm individually. It was worth to note that the thin fibrous morphology of the nanocellulose implied the successful elimination of non-cellulosic components upon the chemical treatment. During acid treatment, the amorphous regions within the native fibers get hydrolases and the microfibers get converted into nanofibers [48]. The high aspect ratio of the obtained cellulose nanofibers is also evident from the images. The greater aspect ratio of the nanofibers offers superior reinforcing effect and consequently stable composites can be developed, according to Rashid et al. [47]. According to Cherian et al. [49] the acid hydrolysis coupled with steam explosion was much effective in formation of nanofibers. All the studied microscopic examinations showed a more refined fiber structure with nano dimension which publicized the adeptness of employed treatment ie. acid coupled stem explosion.

The nano dimension was further confirmed using dynamic light scattering analysis where the statistical distribution of the extracted nanofibers was determined. The DLS size distribution of the onion skin nanofibers was given in Fig. 2d. The average diameter of the fibers was 0.5 nm. Interestingly, from the analysis it can be concluded that all the fiber size was found to be less than 1 nm. This confirmed the formation of nanofibers with a unimodal narrow sized distribution. According to Gond et al. [39] alike nano range distribution of all the isolated fibers indicates its homogenous nature and efficiency of the chemo-mechanical treatment for the extraction of the nanofibers. All the aforesaid morphological and dimensional analysis endorsed the effective synthesis of nanofibers from onion skin.

Fig. 3 showed the solid-state ^{13}C NMR spectra of the extracted cellulose nanofiber. The NMR spectra gives clear evidence for the removal of majority of carbohydrates by the treatment with the acid. The peak at 102 ppm apportioned to hemicellulose disappeared during the treatment with acid indicating elimination of hemicellulose [50]. The absence of peaks at 21, 56 and 173 ppm assigned to the carbon present in the methyl group, methoxyl group in lignin and carbon present in the carboxylic group indicates that treatment with sodium hydroxide considerably removed the side chain from hemicellulose and xylans.

The atomic structure of the nanofibers was determined using solid-state ^{13}C NMR spectroscopy and the corresponding spectra were shown in Fig. 3. Herein, the study utilizes ^{13}C NMR spectroscopy to

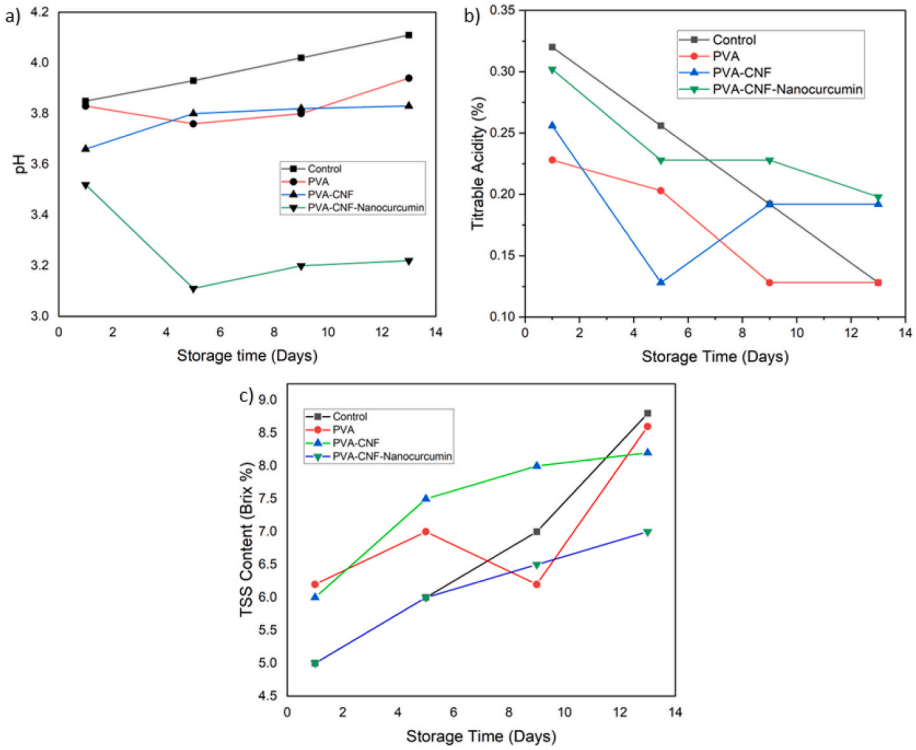

Fig. 12. Analysis of **a)** pH, **b)** TA and **c)** TSSof control, PVA, PVA-CNF and PVA–CNF–Nanocurcumin coated oranges during the storage of 13 days at room temperature.

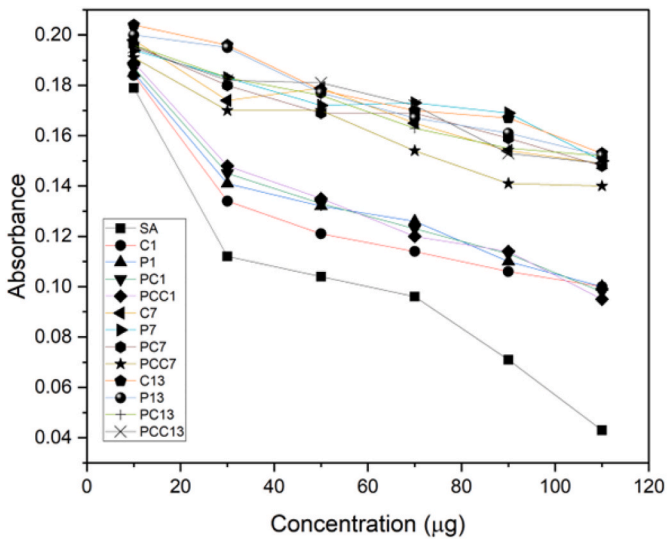

Fig. 13. Antioxidant activity of control, PVA, PVA-CNF and PVA–CNF–Nanocurcumin coated oranges by DPPH Scavenging. (Here, **SA**-Standard ascorbic acid**, C, P, PC** and **PCC** are the control, PVA coated, PVA-CNF coated and PVA–CNF–nanocurcumin coated oranges respectively and the numbers 1,7 and 13 shows the storage days at which the analysis has been done).

determine the chemical shift values correspondent to these 6 carbon atoms that make up each of the cellulose molecules. From the downfield side, the chemical shift value C_1 atom which appears in the glycosidic linkage has a chemical shift range of 106-100 ppm, the appearance of the doublet between 89 and 84 ppm indicated the presence of C_4, the unresolved bands near 81-70 ppm relates the C_2, C_3, and C_5 atoms and the final doublet between 65 and 60 ppm stand for the C_6 carbon atom [51]. A similar ^{13}C NMR peaks were reported elsewhere [52,53].

In addition to the atomic structure, the polymorphism and crystallinity of the sample can be investigated using ^{13}C NMR spectroscopy. According to Foster et al. [54] I_α cellulose was indicated by the singlet at 105 ppm. The NMR spectra of onion skin nanofibers recorded the resonating frequency at 105 ppm which validates the I_α polymorph within the sample. The evaluation of C_4 and C_6 frequencies were necessary for determining the crystalline (ordered) and amorphous (disordered) regions within the sample. The C_4 and C_6 frequencies characteristically seemed as doublets in which the one resonance wing indicates the crystalline phase and other represent the amorphous region accordingly. For C_4, the peak at 89 and 84 ppm denotes the crystalline and amorphous zones respectively. Similarly, the resonating frequency at 65 and 60 ppm for C_6 atom signifies the ordered and disordered fractions respectively [55]. From the ^{13}C spectra, it can be seen that the increased peak intensities of crystalline peaks with respective to amorphous peaks in the case of C_4 and C_6. This clearly demonstrates the significance of the applied treatment in removing the disordered phase (non-cellulosic components) and thereby increasing the crystallinity of the nanofibers. Usually, the lignin resonance takes place at higher resonating frequencies and the absence of such peaks at the downfield regions of the ^{13}C spectra signifies the successful removal of lignin on the pre-treatments. Moreover, the lack particular peak at 101, 80, 74 and 63 ppm in the ^{13}C spectra indicates the absence of hemicellulose within the nanofibers [56]. Subsequently, it can be concluded that afterward the pre-treatment, adequate removal of non-cellulosic components takes place and acceptable nanofibers were produced.

3.2. UV–Vis spectrum of curcumin and nanocurcumin in ethanol

The UV–Visible spectrum shows the formation of nano curcumin from commercially available curcumin Fig. 4.The broad absorption band around 430 nm shows the presence of curcumin in ethanol medium attributed to n-π* and π-π* transitions. A sharp peak around 430 nm shows the presence of nanocurcumin [57].

3.3. Morphological characterisation of prepared coating films

3.3.1. Scanning electron microscopy (SEM)

The morphology of the PVA, PVA-CNF and PVA–CNF–Nanocurcumin films were observed using SEM images. Fig. 5 shows the SEM image of the three different films. It can be seen from the images that the bare PVA has a smooth surface (Fig. 5 a) whereas the incorporation of CNF makes the surface rough to an extent (Fig. 5b and c). This variation in the surface morphology were probably affected by the crosslinking process, indicating good toughness and high surface area, which improves the mechanical properties. The image showed uniform and homogeneous distribution of CNF with a lamellar appearance. This uneven distribution of bright spots indicates occasional agglomeration of CNF in the films. However, in the presence of nanocurcumin the nanocomposite surface persists the lamellar fracture without any agglomeration. From SEM, it is clear that the nanocurcumin further assist the dispersion of the CNF in PVA matrix with more compactness.

3.3.2. Atomic force microscopy (AFM)

Fig. 6 shows the AFM images of the surface of the PVA, PVA-CNF and PVA–CNF–Nanocurcumin films. AFM images gives more evidence on the nanoscale distribution in the polymer film. The surface characteristics of films confirmed the results obtained by SEM. The roughness of the surface gets increased upon the incorporation of CNF (Fig. 6b). In AFM image, the lamellar structuring is evident in PVA-CNF nanocomposite film. Presence of nanocurcumin makes the microstructure more compact and fine distribution of nanofillers (Fig. 6c). Further Fig. 7, is the high magnification AFM image of PVA–CNF–nanocurcumin, depicts the relative position of nanocurcumin and CNF in the PVA matrix. The bright dots indicate nanocurcumin, which are seen occasionally along with the nanofiber network.

3.3.3. Film transparency

Appearance of the fruits determines the acceptance to the consumers. So, for making the fruits appealing the films should be transparent and should able to convey the actual colour of the fruits, which is the most important parameter to define the quality of a fruit as well as coating. The PVA, PVA-CNF and PVA–CNF–Nanocurcumin films showed good transparency (Fig. 8). Among them, bare PVA film was highly transparent and colourless while addition of CNF gave a hazy appearance and addition of nanocurcumin gives a yellow colour to the film.

3.4. Fruit quality parameters

The fruit quality is the most important parameter from the perception of consumers. The quality of fruits can be measured as internal qualities like aroma, flavour and nutritional qualities (TSS, TA, pH etc) and external qualities like appearance, colour and texture. The analysis of quality parameters gives an additional affirmation to the results obtained.

3.4.1. Analysis physical appearance of the fruits during storage

Physical appearance is an important quality parameter with which consumers evaluate the quality of the fruits. Browning of oranges shows its decay and low quality. The change in physical appearance of both controlled and coated oranges during a period of 13 days of storage is shown in Fig. 9. It is clearly visible that the coated oranges have a glossy texture than the controlled ones. At the beginning of the storage there is no observable physical changes, but towards the end at 11^{th} day browning and deterioration was profound in the control oranges while there were no visible changes for the coated ones. At the 20th day of

observation, the PVA–CNF–Nanocurcumin coated orange had a better appearance than the other two type of coated orange (Fig. 10).

3.4.2. Weight loss analysis

The weight loss is one of the most crucial parameters predicting the deterioration in postharvest fruits caused by the loss of water which resulted from the respiration and transpiration during storage [58]. Generally microbial attack enhances the weight loss of fruits and veggies. The percentage weight loss of mandarin oranges with and without coating is given in Fig. 11. From the Figure it is clear that at the initial days of storage the weight loss was almost similar in range but towards the end of storage period weight loss was observed maximum with the control ones when compared to the coated ones. The weight loss percentage of controlled orange reached 19 % at the 13^{th} day of storage while the coated oranges showed much lesser values from the beginning of the storage. At the 13th day of the storage, control, neat PVA, PVA-CNF and PVA–CNF–Nanocurcumin coated oranges showed 19 %, 14 %, 11.8 % and 11.1 % weight loss, respectively. In this regards incorporation of anti-microbial agents like nanocurcumin to the film matrix can reduce the bacterial attack on fruits and vegetables [32].

3.4.3. Determination of pH, Titrable acidity and Total soluble solid

Fig. 12 depicts the variation of pH, Titrable acidity and Total Soluble Solid with storage time in uncoated and coated oranges. The pH of both control and coated oranges, except PVA–CNF–Nanocurcumin coated, was found to be increased at the last days of storage on comparison with the 1st day. But the increase was more significant and steadier with control orange which shows a faster ripening rate than other samples. The pH of PVA–CNF–Nanocurcumin coated orange decreased at the beginning up to five days of storage and there is only a slight increase in the pH level towards the last days, which shows the efficacy of the coating film to control the fruit ripening. (Fig. 12a). This is possibly because of the formation of a good network in the film as seen from the SEM images and might have modified the internal atmosphere by controlling the CO_2 and O_2 concentration of the fruit, thus retards ripening.

The titrable acidity (TA) measures the total acid content in the fruit. As the fruit ripens the organic acids present in the fruits are converted into sugars due to enzymatic activities and hence, reduction in the TA values of fruits during post-harvest storage happens [59]. The major organic acid present in the orange is citric acid [60]. Here also, the TA value of control oranges goes on a steady decrease throughout the storage period while PVA and PVA-CNF coated oranges could maintain the acidity level in the last days of storage. Even though the TA value of PVA–CNF–Nanocurcumin decreases during observation time, the value is higher at the 13^{th} day among all the samples giving evidence for the efficacy of the coating film (Fig. 12b).

The TSS is the best indicator for the fruit ripening because starch get hydrolysed into glucose during the ripening process [61]. The total soluble solid (TSS) of the oranges varies between 4.5 and 9 °Brix during the storage (Fig. 12c). TSS includes the measure of carbohydrates, organic acids, proteins, fats and minerals of the fruits [62]. As the fruit ripens the TSS also increases due to the enzymatic activities of microbes. In this study, it is visible that at the beginning of the storage period TSS of control and PVA–CNF–Nanocurcumin coated oranges were same but towards the end the TSS of control oranges goes on a steady increase having a higher difference than that of PVA–CNF–Nanocurcumin coated orange. In addition, PVA–CNF–Nanocurcumin coated oranges could maintain the TSS in comparison with other samples probably because of the formation of well network of coating which protects the fruits from external factors which enhances the enzymatic and microbial activities on fruits, which in turn helps to maintain the quality of the fruits.

3.4.4. Antioxidant activity

The antioxidant activity of controlled and coated oranges were studied at the 1^{st}, 7^{th} and 13^{th} day of storage by the Blois method (1958) DPPH scavenging Assay and it shows the anti-oxidant activity is inversely proportional to the absorbance [37]. The principle of this method is that antioxidants react with the stable free radical DPPH* and convert it to 2,2-diphenyl-1-picrylhydrazine, which is accompanied by a colour change from purple to yellow [63]. From Fig. 13, it is evident that at the 1st day of storage, the anti-oxidant activity is almost same and pretty good for all the samples regardless of coating type. But as the number of storage day increases, there is a significant reduction in the anti-oxidant activity for both control and coated oranges. On the 7^{th} day of storage PVA–CNF–Nanocurcumin (PCC7) shows better anti-oxidant activity than that of control (C7) and other two coatings. Also, on the last day of storage the PVA–CNF–Nanocurcumin (PCC13) shows a high anti-oxidant activity than all other control and coated oranges. Hence, it is an assertion for the SEM analysis that PVA–CNF–Nanocurcumin with well dispersed CNF and nanocurcumin lowers the gas permeability and hence the decomposition of complex sugars and thereby delay the ripening of fruits [16].

Finally, an effort has been taken to compare the outcomes of the current investigation with that of previous studies on various fruit coatings. Khorram et al. [64] fabricated polysaccharide as well as emulsion based edible coating for 'Kinnow' mandarin and studied the change in fruit's quality with storage time. All the coatings helped to reduce the weight loss in comparison to the controlled mandarins, which is in line with the current study. But there is no significant interaction of treatment and storage time in case of TSS and TA values. In another study, Robles-Sánchez et al. [65] observed that anti-oxidant activity of fresh cut mangoes coated with alginate matrix incorporated with anti-browning agents like ascorbic acid and citric acid were higher than that of the controlled ones, by following DPPH assay. Xanthum gum encapsulated with zinc oxide based edible coating prepared by Joshy et al. [11] was a novel strategy to enhance the shelf life of tomatoes and apples, which provided anti-bacterial property and reduced weight loss for coated fruits along with good mechanical and barrier properties. There has been no works reported on CNF/Nanocurcumin incorporated PVA based edible coating for mandarin oranges till the date and this study shows an enhancement in the postharvest quality of treated mandarin oranges than the controlled fruits.

4. Conclusion

The current investigation put forward an agriculture waste valorisation method for the development of sustainable fruit coatings and films. Cellulose nanofibers were successfully extracted from waste onion skin and their morphology was well studied by TEM, AFM and SEM. PVA-CNF-Nanocurcumin coating was fabricated using the extracted CNF and are dip coated on mandarin oranges. The developed PVA-CNF-Nanocurcumin coatings are considered as a potential method to maintain the postharvest quality of mandarin oranges. All the developed coating provided a transparent and glossy appearance to the oranges which makes them appealing to the consumers. Among three coating solutions, the PVA–CNF–Nanocurcumin was more effective in maintaining the physio-chemical qualities of the oranges such as percentage weight loss, pH, TA, TSS and antioxidant activity. The output of this investigation would be considered as a general strategy for agricultural waste valorisation and a solution for plastic pollution.

Data availability

The research data are not shared.

Ethical guidelines

Ethical approval is not required for this research.

CRediT authorship contribution statement

Anjana Krishna S V: Methodology, Investigation, Formal analysis,

Data curation. **Umadevi S:** Investigation, Formal analysis. **Midhun Dominic C D:** Writing – review & editing, Resources. **Jyotishkumar Parameswaranpillai:** Writing – review & editing, Resources. **Asha Bhanu A V:** Resources, Project administration. **Jesiya Susan George:** Validation, Resources. **Sreedevi T:** Investigation. **Sabu Thomas:** Writing – review & editing, Supervision. **Poornima Vijayan P:** Writing – original draft, Supervision, Project administration, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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