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RESEARCH ARTICLE

Fabrication and Characterization of Cellulose Grafted Glycidyl Methacrylate-Based Film for Food Packaging

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ABSTRACT

The use of persistent plastic in food packaging continues to raise practical and environmental concerns, prompting interest in biodegradable alternatives derived from biomass. In this work, cellulose derived from coconut dregs was used as a film-forming material. The cellulose was isolated using a combination of chemical and ultrasonic treatments, followed by grafting with glycidyl methacrylate (GMA). Citronella oil (CitO) was incorporated at different loadings to produce GraftCell/CitO0.15, GraftCell/CitO0.6, and GraftCell/CitO1.2 films. Fourier-transform infrared (FTIR) analysis confirmed grafting, while structural changes were reflected in an increase in the crystallinity index from 10.55% to 16.04%. Thermogravimetric (TGA) analysis further revealed a shift in the onset degradation temperature from 254.6˚C (Cell) to 369.6˚C (GraftCell), with a maximum decomposition temperature reaching 477.8˚C, indicating improvement of thermal resistance after modification. The mechanical response varied depending on CitO content. The GraftCell/CitO1.2 film exhibited a tensile strength and elongation at break of 25.63 MPa and 21%, respectively. Approximately three times higher than that of the untreated cellulose film. Shelf-life testing revealed that grapes wrapped with the GraftCell/CitO1.2 film remained visually acceptable for up to 9 days, compared to conventional plastic packaging (ony 5 days). Indicating an approximate 80% improvement in preservation performance. These findings highlight the potential of the GraftCell/CitO1.2 film as a sustainable biodegradable packaging material.

Keywords: Biodegradable film, Cellulose, Citronella oil, Glycidyl methacrylate (GMA), Grafting

Introduction

Traditional plastics are now an essential part of everyday life.[1] Because of their superior flexibility, mechanical strength, and protective qualities, these plastics, which are mostly made from petroleum, are preferred.[2] However, the non-biodegradable nature of petroleum-based plastics is a major disadvantage that contributes to several environmental problems. These include disturbances to natural ecological cycles, the spread of hazardous substances, the development of mosquito and pest breeding grounds, and blocked drainage systems.[3–5] Additionally, the accumulation of traditional plastics in aquatic environments poses serious environmental risks.[6] Organisms can consume small plastic particles, which can then infiltrate and spread throughout the food chain.[7,8] Extensive research has focused on creating alternatives to traditional plastics to address these urgent issues. One promising solution is the production of biodegradable films from natural polymers.[9]

Natural and environmentally friendly biopolymers, such as starch, cellulose, lactic acid, hydroxyalkanoic acids, and other materials derived from microbes or plants, are used to make biodegradable films.[10–14] The most popular polymer for creating biodegradable

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films among them is cellulose.[15,16] Films made of cellulose are comparatively simple to make and use little energy. Furthermore, cellulose films easily decompose in soil and water.[2] Cellulose derivatives from plants offer several advantages over other biomaterials due to their unique properties. These consist of biodegradability, high flexibility, narrow pore size, large surface area, and simplicity of modification.[17] Structurally, cellulose comprises repeating glucose units characterized by abundant hydroxyl groups and extensive hydrogen bonding. These structural features enhance its reactivity and versatility for various applications.[18] Numerous investigations have documented cellulose derived from natural sources, including sugarcane bagasse,[19,20] ginger,[21] bamboo,[22,23] pineapple leaves,[24] red velvet tamarind rind,[16] leftover coconut milk pulp,[25] and others. In the present investigations, cellulose was extracted from leftover coconut milk pulp waste and assessed for the fabrication of biodegradable films.

World coconut production in 2020–2022 reached 62 Mt, mainly from Asian countries such as Indonesia, the Philippines, Sri Lanka, Vietnam, Thailand, and Malaysia, making Asia the largest coconut-producing region globally.[26–28] Previous studies have reported a significant increase in coconut production due to rising global demand for coconut-based products, including coconut water, coconut meat, coconut milk, and coconut oil. This increased demand has consequently resulted in the generation of substantial amounts of waste. About 80% of the weight of the coconut is wasted as fiber, husks, shell trash, and dregs.[29] Although considered low-value materials, Coconut dregs are rich in cellulose content.[30] Utilizing coconut dregs as a cellulose source for film production represents a promising approach to enhancing their value. Our previous study produced cellulose film from coconut dregs residue without any modification.[25,31] However, the mechanical properties of the resulting film were suboptimal, indicating the need for further modification to improve its mechanical properties and performance.

Researchers have explored various methods to modify cellulose and enhance its properties, including oxidation, etherification, halogenation, and esterification, as well as cross-linking and grafting techniques.[17] Among these, grafting copolymerization is a versatile and effective approach for modifying polymer surfaces by introducing different functional groups.[32] Free radical copolymerization, a widely used polymerization technique, is particularly appealing due to its simplicity, efficiency, and versatility. Prior research has documented using several monomers, including methyl methacrylate, styrene, and glycidyl methacrylate (GMA), to modify the surface of bacterial cellulose aerogels.[33] Additionally, the preparation of cellulose nanocrystal particles grafted with GMA has been documented.[34] Another study described the modification of cellulose derived from sugarcane bagasse waste with GMA for developing chitosan composites used in wastewater treatment.[35] Our previous study also reported that cellulose extraction from coconut dregs residue was successfully modified with GMA, though it had not yet been applied in biopolymer-based film production.[36] Previous researchers have also reported that the incorporation of essential oils can enhance the mechanical properties of films, contributing to their potential application in various fields, including sustainable packaging solutions. This dual approach of modifying cellulose and incorporating essential oil fillers offers promising avenues for producing films with superior performance characteristics.[37,38]

Recent developments have also demonstrated that combining biopolymers with natural extracts or nanoparticles can improve mechanical, barrier, and active functionalities of biodegradable films. For example, carboxymethyl cellulose films modified with onion peel waste and boron nitride nanoparticles showed improved antioxidant release and lower vapor permeability,[39], while sodium alginate/flaxseed mucilage films containing WO_3 nanoparticles and norbixin enhanced oxygen-barrier and antioxidant properties.[40] Likewise, photoluminescent and sensoractive films based on gelatin, chitosan, or PVA matrices have been explored for smart and active food packaging applications.[41,42]

No research has been reported on fabricating grafted cellulose-based films incorporated with citronella oil (CitO). In this study, we developed cellulose powder using GMA-forming grafted cellulose. The obtained grafted cellulose powder was combined with citronella oil to form grafted cellulose/citronella oil film (GraftCell/CitO film).

Materials and methods

Materials

Coconut dregs residue was collected in a coconut milk factory in Banda Aceh, Indonesia. The citronella oil (*Cymbopogon nardus* (L.), CAS No. 8000-29-1), hereafter referred to as CitO, was purchased from the Atsiri Research Center–Universitas Syiah Kuala (ARC–USK), Indonesia. The oil was originally supplied by PT. Aroma Atsiri Indonesia (Bogor, Indonesia). The oil was used as received without further purification. The GC–MS profile of this batch was not available; however, the composition is consistent with typical *Cymbopogon nardus*

oil, commonly dominated by citronellal, geraniol, and citronellol as reported in the literature. Glycidyl methacrylate (GMA, ≥97%), potassium persulfate (KPS, ≥99%), sodium hydroxide (NaOH, ≥98%), n-hexane (≥95%), ethanol (≥99%), hydrochloric acid (HCl, 37%), sodium hypochlorite (NaClO, 15%), and glacial acetic acid (CH_3COOH, ≥99.7%) were all of analytical grade and purchased from Sigma-Aldrich (Singapore).

Isolation of cellulose from coconut dregs

Cellulose isolation was performed according to the procedure described in our previous work.[31] The coconut dregs were first separated from impurities such as plastics, wood, sand, and stones, then air-dried at room temperature for 24 hours. 40 g of dried coconut dregs were immersed in a 2:1 (v/v) hexane–ethanol solution (40 g sample: 100 mL solvent) and agitated at 60°C and 500 rpm for 24 hours to remove residual waxes and lipids. The mixture was then rinsed and filtered with distilled water until a neutral pH (≈7) was achieved. The dewaxed pulp was oven-dried at 60°C for 24 hours. Delignification was carried out by stirring the dried pulp in a 5% (w/v) NaOH solution at a solid-to-liquid ratio of 1:20 (w/v) at 75°C for 4 hours. The sample was rinsed with distilled water until neutral pH (≈7) and then subjected to acid hydrolysis using 7 M HCl at a 1:20 (w/v) ratio. The mixture was stirred at 500 rpm and 75°C for 2 hours, followed by washing until the pH was neutral. The hydrolyzed pulp was bleached by mixing it with a 15% NaClO–glacial acetic acid solution at a 4:1 (v/v) ratio, stirred at 200 rpm and 60°C for 2 hours, and then rinsed until pH 7. The final ultrasonication process was conducted using a Vibra-Cell ultrasonic processor (750 W, Sonics & Materials, USA) at 25°C. A 10 g portion of the bleached cellulose was dispersed in 100 mL of distilled water and sonicated for 1 hour at a pulse rate of 5 s on and 5 s off to prevent overheating and ensure uniform fibrillation.

Grafting process on cellulose–derived coconut dregs

Grafting of GMA onto cellulose was performed according to our previous protocol under identical reaction conditions. The grafted cellulose obtained in this study was verified in the previous work to exhibit grafting percentage (GP%) and grafting efficiency (GE%) values of approximately 299.2% and 16.3%, respectively. After the grafting process, all samples were sequentially washed with distilled water and methanol to remove residual monomers and initiator, as confirmed in Ref.[36] by the disappearance of GMA monomer peaks in the FTIR spectra.

Preparations of films

Grafted cellulose was dispersed in distilled water at a ratio of 1:5 (g/mL) in a 250 mL beaker and stirred continuously for 15 minutes. Citronella oil was added to the suspension at different concentrations of 0.15%, 0.6%, and 1.2%. The mixtures were then poured into a Teflon (14 cm in diameter) and dried in an oven at 40°C for 24 hours. The obtained films were marked as GraftCell/CitO0.15, GraftCell/CitO0.6, and GraftCell/CitO1.2, respectively. Cellulose film was also prepared for comparison.

Characterizations

Fourier Transform Infrared (FTIR) analysis was performed by using a Thermo Nicolet 6700 FTIR spectrometer. The samples were scanned in the wavelength range of 4000–500 cm^{-1}. To determine the crystallinity and phase properties of the samples, the samples were scanned by using PANalytical X-pert PRO at 25°C, 40 kV, and 30 mA with $2\theta = 5°–70°$. The crystallinity index (CI) was determined using the Segal method.[43] Thermal degradation behavior of the films was analyzed using a thermogravimetric analyzer (TGA, PerkinElmer STA 6000) under a nitrogen atmosphere with a flow rate of 40 mL/min. Approximately 10 mg of each sample was heated from 30 to 600°C at a rate of 10°C/min.[10] The tensile strength (TS) and elongation at break (EAB) of the films were determined according to ASTM D882 using a Universal Testing Machine (HT8503, Hung Ta Instrument Co., Ltd., Taichung, Taiwan) at a crosshead speed of 20 mm/min. Film specimens (100 mm × 10 mm) were conditioned at 23 ± 2°C and 50 ± 5 % relative humidity for 48 h prior to testing. Film thickness was measured by the testing laboratory using a digital micrometer at multiple points, and the average value was used for tensile calculations. A scanning electron microscope (SEM, JEOL JSM-6510LA, Belgium) was used to examine the films' surface and cross-sectional morphologies. Before imaging, samples were placed on aluminum stubs and sputter-coated with a thin layer of gold (10 nm) to improve conductivity. An accelerating potential of 10 kV was used, and surface and cross-sectional images were obtained at magnifications of 3000× and 150×, respectively. For dimensional reference, scale bars were incorporated into every image.

Film evaluations

The soil burial method[44] was used to analyze the films' biodegradability with a few minor adjustments. To avoid ripping, square film samples (3 × 3 cm^2) were wrapped in gauze and buried 10 cm deep in

natural soil. After three, five, and seven days, the samples were collected, professionally cleaned, dried, and weighed. The percentage of weight loss was calculated using Eq. (1).

$$\text{WeightLoss}\,(\%) = \frac{(W_0 - W_1)}{W_0} \text{x } 100 \quad (1)$$

where, W_0 is initial weight and W_1 is weight after test

The water absorption ratio was determined by first drying the films in a vacuum oven at 60°C for 24 h to remove moisture. The dried samples were then immersed in distilled water for 30 min, blotted with tissue paper to remove excess water, and weighed immediately. The water absorption ratio was calculated using Eq. (2).

$$\text{Water absorption ratio} = \frac{(M_0 - M_1)}{M_0} \text{x } 100\% \quad (2)$$

where M_o is the initial weight before the test and M_1 = weight after the test [45]

Porosity was measured by drying the films (3 × 3 cm^2) in an oven at 105°C for 24 h, after which the specific gravity was calculated from the dry weight and film volume. Porosity was determined using Eq. (3). The swelling ratio was determined by weighing the initial dry film, immersing it in water for a fixed time, gently removing it with tweezers, blotting off surface water, and recording the swollen weight. The percentage swelling (Ps) was calculated using Eq. (4).

$$\text{Porosity} = \left(1 - \frac{\rho_1}{\rho_2}\right) \text{x } 100\% \quad (3)$$

$$\text{Ps} = \frac{(W_s - W_d)}{W_d} \text{x } 100 \quad (4)$$

where, ρ_1= density of sample, ρ_2= density of cellulose, Ws = polymer swelling weight and Wd = dry weight of polymer

Performance of film as grapes packaging

The performance of the developed films was evaluated by applying them as packaging for fresh grapes. Selected grape samples of uniform size and ripeness were gently cleaned and air-dried prior to packaging. Grapes were wrapped using the GraftCell/CitO1.2 film, while another batch was wrapped with conventional commercial plastic as a control. Each packaged sample was stored under ambient conditions (25 ± 2°C, 60 ± 5% RH) for 9 days. Changes in color, texture, surface integrity, and odor of the grapes were observed on the 7th and 9th days of storage. The evaluation was performed visually by direct observation, following a previously reported approach, [16,46] No instrumental color or texture analysis was employed; observations focused on visible wrinkling, color darkening, and signs of mold or decay to assess the films' preservation.

Statistical analysis

All measurements were performed in duplicate (n = 2), and the results are expressed as mean values. Due to the limited number of replicates, no inferential statistical analysis (e.g., ANOVA or t-test) was performed. Variations between replicates were within acceptable experimental error limits.

Results and discussion

Mechanical properties

Cellulose was extracted from coconut dreg residue using chemical and ultrasound-assisted methods prior to film production. The obtained cellulose was grafted with GMA to produce grafted cellulose. Cellulose grafted with GMA was incorporated into citronella oil at varying concentrations, and the resulting GraftCell/CitO film was obtained. The mechanical properties of GraftCell/CitO films with different concentrations of citronella oil were evaluated using a tensile test and compared with those of a cellulose film, as shown in Fig. 1.

Based on Fig. 1, the cellulose film has the lowest TS and elongation, recorded at 7.75 ± 0.3 MPa and 3.92 ± 0.2%, respectively. These values increased markedly after modification of cellulose with

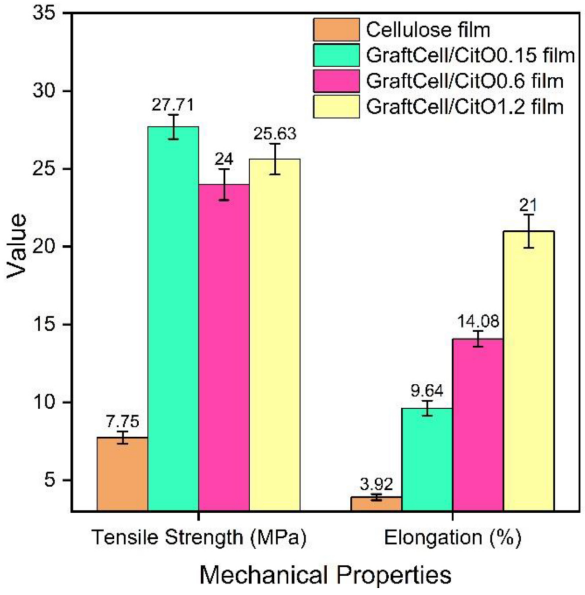

Fig. 1. Mechanical properties of films.

GMA and incorporation of citronella oil. The tensile strength of GraftCell/CitO films was approximately threefold higher than that of the unmodified cellulose film. The use of citronella oil at different concentrations did not cause a substantial variation in tensile strength among the grafted samples, whereas elongation at break increased progressively with increasing citronella oil concentration. The highest elongation was found in the film containing 1.2% citronella oil (GraftCell/CitO1.2 film), reaching 21 ± 0.5%.

This indicates that GMA grafting predominantly enhanced tensile strength, while citronella oil primarily acted as a plasticizer, improving flexibility and elongation at break.[47] The increase in tensile strength was more strongly influenced by the grafting process applied to cellulose before film production, as reflected by the nearly similar tensile values among all GMA-modified samples. Additionally, the progressive increase in elongation with citronella oil content supports the role of essential oils as natural plasticizers, as also reported in previous studies.[47] The improvement in both TS and elongation of the cellulose-based films in this study addresses the mechanical limitations reported in our previous work.[31] Considering the overall balance between strength and flexibility, GraftCell/CitO1.2 was selected as the most promising formulation for potential food-packaging applications.

Fourier transform infrared (FTIR)

The influence of GMA addition on the cellulose structure was analyzed using FTIR spectroscopy, as shown in Fig. 2. The FTIR spectrum of cellulose Fig. 2a exhibits characteristic absorption bands, where the broad band at 3449 cm^{-1} corresponds to the stretching vibration of hydroxyl groups (–OH), while the band at 1632 cm^{-1} is attributed to the bending vibration of adsorbed water molecules rather than structural –OH groups. The absorption band at 1246 cm^{-1} is assigned to the C–O–C glycosidic stretching vibration, a characteristic peak of cellulose. The band at 2922 cm^{-1} corresponds to C–H stretching of methylene ($–CH_2–$) groups.[48] Additionally, a weak absorption band at 1746 cm^{-1} is observed, corresponding to C=O stretching vibrations from residual hemicellulose impurities present in cellulose.

FTIR spectrum of grafted cellulose Fig. 2b exhibits several differences compared to pure cellulose Fig. 2a, indicating the successful incorporation of GMA into the cellulose backbone. The –OH stretching vibration shifts to a lower wavenumber (3431 cm^{-1}), suggesting a reduction in free hydroxyl groups and an increase in intermolecular hydrogen bonding due to grafting.[35,49] The reduced intensity of the 1636 cm^{-1} band also indicates lower water interaction. A prominent carbonyl band appears at 1727 cm^{-1}, corresponding to the ester carbonyl (C=O) group of GMA, while enhanced peaks in the 1260–1160 cm^{-1} region correspond to C–O–C stretching of ester linkages. These observations confirm the presence of methacrylate ester groups introduced by GMA grafting, as reported in previous studies.[36,50–52] As illustrated in Fig. 3, the proposed reaction mechanism for cellulose grafting with GMA supports the functional group transformations observed in the FTIR spectra and confirms the successful incorporation of GMA onto the cellulose backbone. However, FTIR alone can-

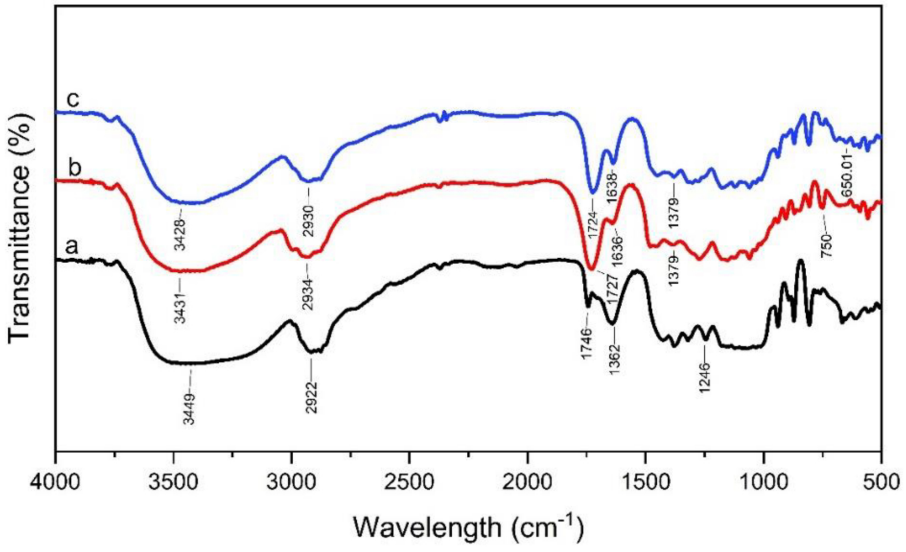

Fig. 2. FTIR spectra of (a) Cellulose, (b) Grafted cellulose, and (c) GraftCell/CitO1.2 film.

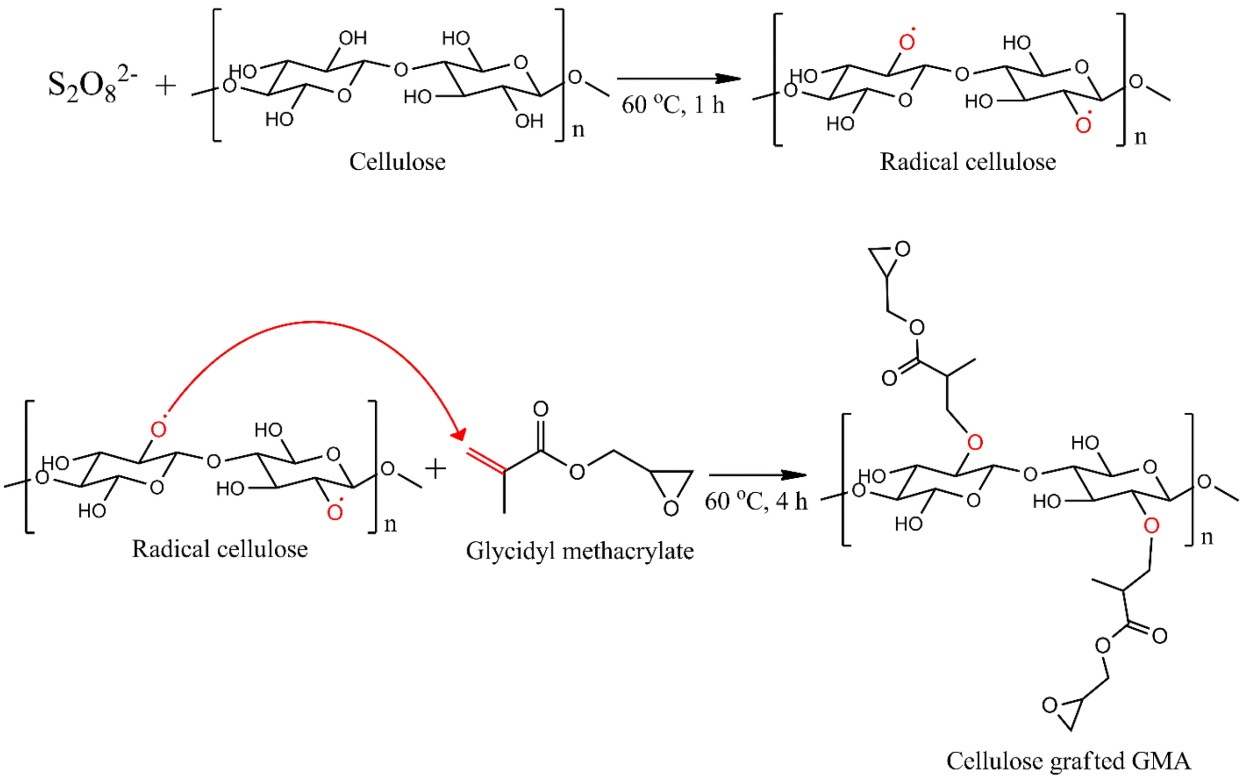

Fig. 3. Reaction scheme of cellulose grafting with GMA.

not verify epoxy ring opening or covalent linkage formation, and these findings are interpreted as indicative rather than conclusive evidence of successful grafting.

The FTIR spectrum of GraftCell/CitO1.2 film Fig. 2c shows a broad absorption band at 3428 cm^{-1}, corresponding to O–H stretching vibrations, slightly shifted compared to grafted cellulose. This shift suggests possible hydrogen-bonding interactions between citronella oil constituents and the cellulose–GMA matrix.[53] The band at 2930 cm^{-1} is assigned to aliphatic C–H stretching, consistent with the terpenoid content of citronella oil. A sharp carbonyl band at 1724 cm^{-1} is associated with both the ester carbonyl of grafted GMA and aldehyde functionalities of citronellal.[53] The interaction between citronella oil and GMA-grafted cellulose is now attributed to physical incorporation and hydrogen bonding rather than chemical reaction. Fig. 4 illustrates a potential hydrogen-bonding and/or physical interaction mechanism. Although epoxy ring opening is theoretically possible, it was not confirmed experimentally in this study. Additionally, the band near 1638 cm^{-1} may correspond to C=C stretching or adsorbed water, while signals below 700 cm^{-1} arise from complex bending vibrations of citronella oil components. These results indicate that citronella oil was successfully incorporated into the GMA-grafted cellulose matrix, likely via non-covalent interactions, without forming new chemical bonds.[54]

X-ray diffractions (XRD)

The XRD analysis was performed to analyze the effect of GMA and citronella oil on cellulose structure. Fig. 5 shows the XRD pattern of GraftCell/CitO1.2 film, which is compared with cellulose film and grafted cellulose film. Cellulose film shows an amorphous structure based on the XRD pattern Fig. 5a, where no crystalline peaks were found. This indicates that the cellulose polymer chains are irregularly arranged in the film.

Based on Fig. 5, the cellulose film Fig. 5a exhibits a semi-crystalline structure, with diffraction peaks observed at $2\theta = 37.75°$, 43.98°, and 64.32°, resulting in a crystallinity index (CI) of 10.55%. This indicates that the native cellulose possesses a relatively low degree of crystallinity. After grafting with GMA, the cellulose-GMA film Fig. 5b exhibits new diffraction peaks at $2\theta = 24.96°$ and 27.68°, indicating structural modification of cellulose during grafting. With diffraction peaks at $2\theta = 43.98°$ and 64.32°, this film's CI rises to 16.04%. The more regular arrangement of cellulose polymer chains brought about by the

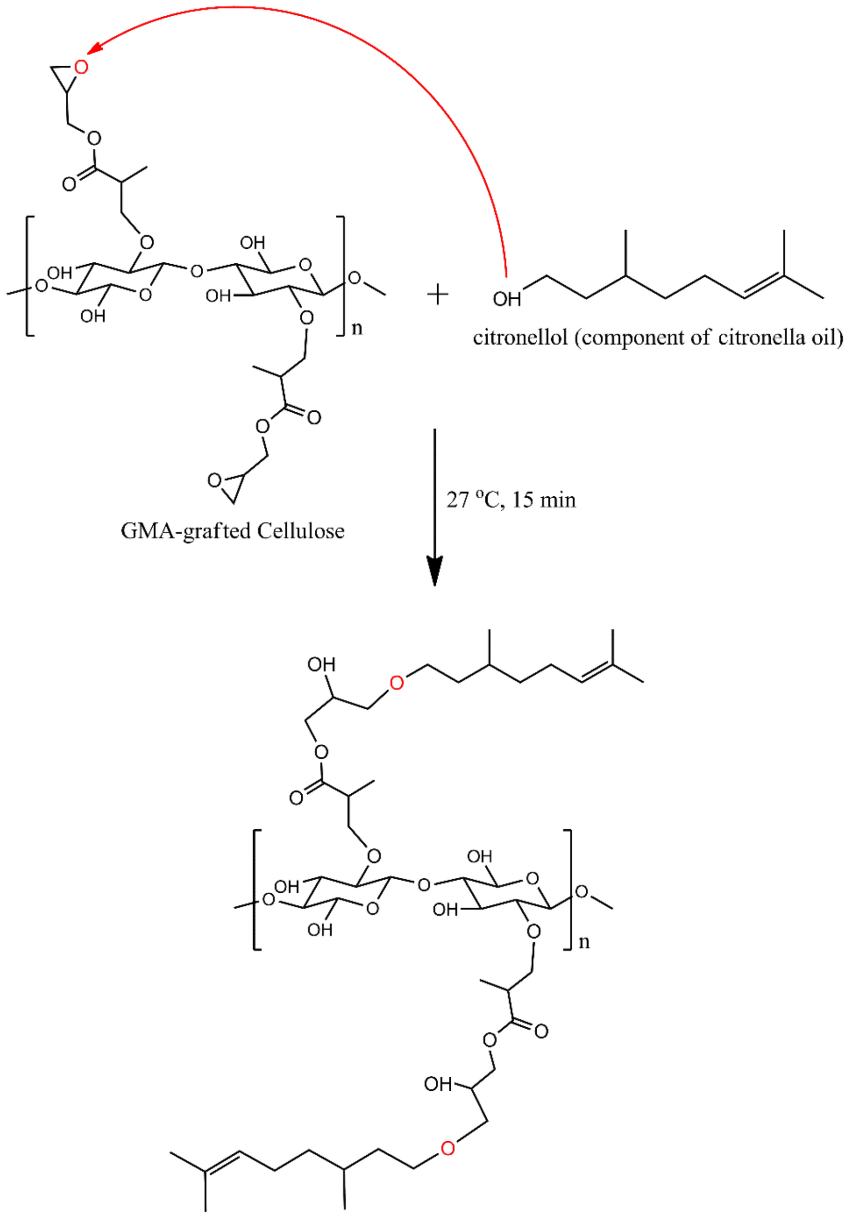

Fig. 4. Possible interaction pathway between citronellol (a component of citronella oil) and GMA-grafted cellulose.

addition of GMA monomers, which results in a more ordered film structure, is probably responsible for this increase in crystallinity.

In contrast, the cellulose-GMA film and the Graft-Cell/CitO1.2 film Fig. 5c show a comparable diffraction pattern, suggesting that the primary cellulose crystalline structure is still present. Nevertheless, diffraction peaks at $2\theta = 27.98^{\circ}$, 43.98°, and 64.32° show a modest decrease in peak intensity, and the CI drops to 14.89%. The interaction between the components of citronella oil and the GMA-grafted cellulose matrix, which somewhat disturbs the orderly arrangement of the polymer chains, is responsible for this reduction. The resultant film may become more flexible and elongated due to the slight drop in crystallinity.

TGA analysis

The TGA was used to systematically examine the heat-degradation behavior of cellulose-based films Fig. 6. The neat cellulose film exhibits two distinct weight-loss phases. The evaporation of physically adsorbed and chemically bonded water within the cellulose matrix is the first step, occurring between 30 and 100°C. During the second central stage, which lasts from 254.59°C to 386.07°C, the cellulose backbone breaks down due to depolymerization,

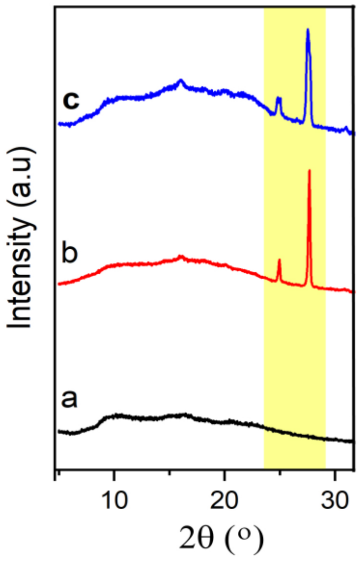

Fig. 5. The XRD patterns of: (a) Cellulose, (b) Grafted cellulose, and (c) GraftCell/CitO1.2 films.

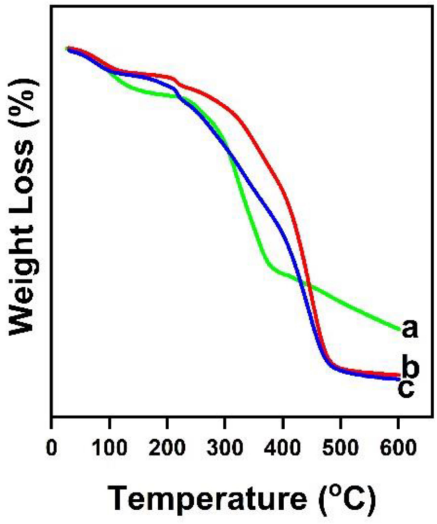

Fig. 6. Thermograms of: (a) Cellulose, (b) Grafted cellulose, and (c) GraftCell/CitO1.2 films.

dehydration, and glycosidic bond breaking. A substantial mass loss of about 81.21% is the outcome of this process, which is followed by progressive carbonization at 400°C.

The grafted cellulose film exhibits a modified degradation profile, with three distinct stages following GMA grafting. Moisture loss occurs in the first stage (30–100°C), and the breakdown of less stable grafted side chains occurs in the second stage (200–220°C). The basic degradation process, which includes the opening of the GMA monomer's oxirane ring and the subsequent disintegration of the cellulose backbone, is represented by the third stage (369.55–477.79°C). The grafted film has a higher initial degradation temperature than pure cellulose, indicating improved thermal stability. This enhancement arises from additional hydrogen bonding and intermolecular crosslinking introduced by GMA, which restricts molecular mobility and strengthens the polymer network. A similar trend was reported by Wang et al. (2023),[55] who found that GMA-modified sugarcane bagasse cellulose exhibited increased decomposition temperature due to the formation of strong hydrogen-bonded networks.

The GraftCell/CitO1.2 film, containing citronella oil incorporated into the grafted cellulose matrix, exhibits a three-step degradation profile similar to that of the grafted film, but with a slightly earlier onset of weight loss. The initial stage (30–100°C) corresponds to the evaporation of water, while the second stage (220–260°C) can be attributed to the evaporation and degradation of citronella oil components. The primary decomposition stage (324.57–477.42°C) involves the breakdown of the cellulose backbone and volatilization of residual organic constituents. The earlier onset temperature and slightly increased mass loss suggest that the presence of citronella oil reduces the film's structural compactness and lowers its thermal resistance, likely due to the oil's plasticizing effect, which weakens the hydrogen-bonded network within the polymer matrix. This interpretation aligns with the SEM observations Fig. 7d, which reveal a less-dense microstructure in the citronella-containing film.

In comparison to the pure cellulose film, both the grafted cellulose and GraftCell/CitO1.2 films show slower rates of decomposition, suggesting that GMA grafting greatly improves thermal stability. Although the stability of the citronella-containing film slightly decreases, this does not negate the overall benefit brought about by grafting. Rather, it represents a trade-off between lower network density and more flexibility. These results are consistent with XRD Fig. 5 and tensile test results Fig. 1, which demonstrate improved mechanical strength and crystallinity in the grafted systems.

Additionally, a similar finding was reported by Agusnar et al.[38], who discovered that patchouli oil-incorporated chitosan films demonstrated exceptional thermal resistance, with an initial degradation temperature ranging from 444.63°C to 492.20°C and a maximum degradation midpoint at 467.22°C, resulting in minimal weight loss (−6.730 mg). The idea that adding essential oils, like citronella, can alter the thermal breakdown behavior of biopolymer matrices by affecting their molecular packing and bonding interactions is supported by this earlier study. Citronella oil is therefore included in the

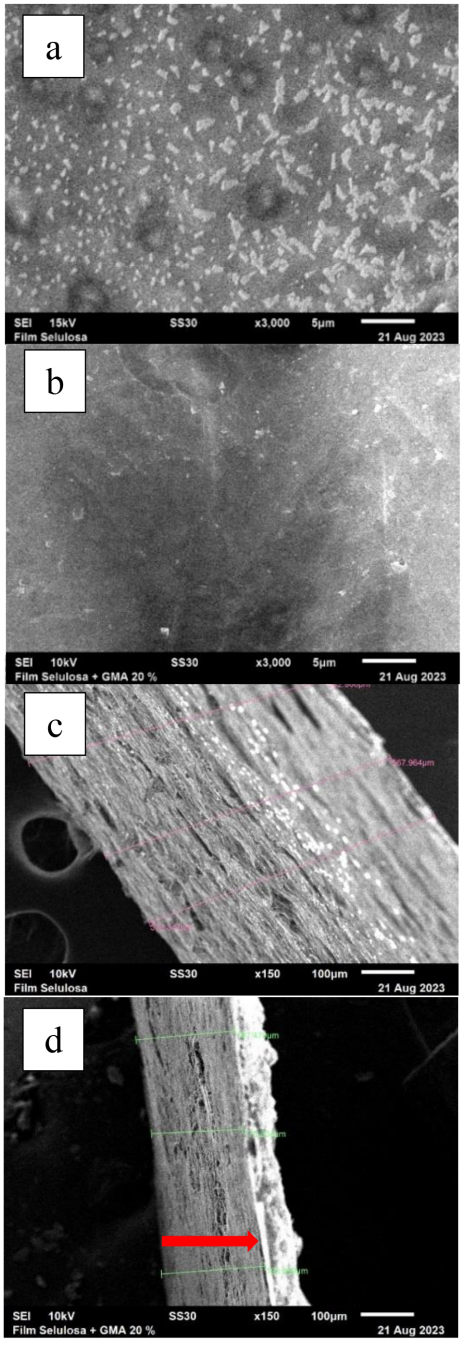

Fig. 7. SEM images of (a) The surface of the cellulose film, (b) The surface of the cellulose-g-GMA, (c) The cross-section of the cellulose film, and (d) The cross-section of the cellulose-g-GMA film.

GMA-grafted cellulose film to preserve a balance between functional flexibility and thermal endurance. This progression demonstrates that GMA grafting reinforces the cellulose network through chemical crosslinking[48], while citronella oil fine-tunes the film's degradation behavior by interacting with the polymer chains. The Tonset, Tmax, and char residue (%) values were not quantitatively analyzed in this study. The discussion is therefore based on qualitative interpretation of the TGA curves to describe the general degradation behavior of the films.

Scanning electron microscope (SEM)

The SEM images of cellulose film and GraftCell/CitO1.2 film (surface and cross section) are shown in Fig. 7. The surface of cellulose film Fig. 7a shows that cellulose polymers were arranged irregularly and relatively inhomogeneously, where some cellulose particles were still observed on the surface. This result was in line with XRD analysis Fig. 5, where cellulose film has an amorphous phase.

In contrast to cellulose film, GraftCell/CitO1.2 film shows a relatively homogenous surface, and cellulose particles were not observed in the surface Fig. 7b. The fibrils in the GraftCell/CitO1.2 film Fig. 7d were arranged denser than cellulose film Fig. 7c. This was probably due to the existence of GMA in cellulose polymer chains, which provides crosslinking formation. These results supported the results of the tensile test, where films with denser fibrils will produce higher tensile strength.

Film evaluations

The films produced were evaluated for their properties related to biodegradability, water absorption ratio, porosity, and percentage of swelling. A biodegradability investigation was performed for seven days, where the observations were made on the third, fifth, and seventh days. Based on Fig. 8, all films

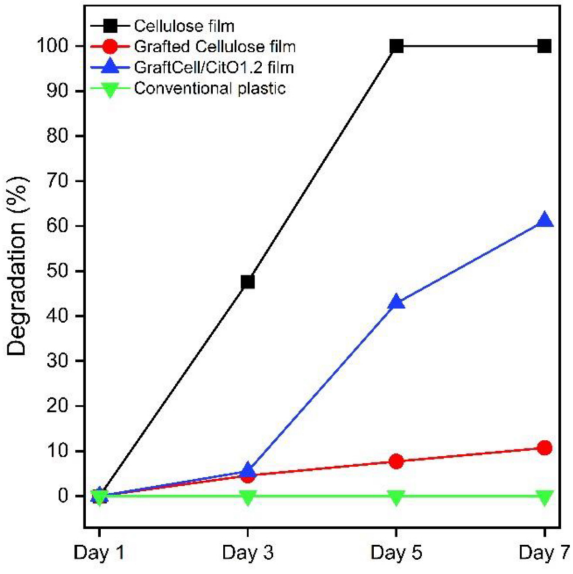

Fig. 8. Degradation rate of films compared with conventional plastic.

decreased in weight during the biodegradability test, but the weight of conventional plastic remained the same. Cellulose film showed a percentage of weight loss on the 5th was 100%. It indicated cellulose film was completely degraded on the 5th day. It may be due to the fact that cellulose film can be directly degraded by microorganisms in the soil.

As shown in Fig. 8, the degradation percentages on the third day for cellulose film, grafted cellulose film, and GraftCell/CitO1.2 film were 47.67%, 7.69%, and 5%, respectively. On the fifth day, the cellulose film was completely degraded, while the degradation percentages for cellulose grafted cellulose film and GraftCell/CitO1.2 film were 4.54% and 42.85%, respectively. On the seventh day, the degradation percentages for grafted cellulose film and GraftCell/CitO1.2 film were 10.71% and 61.11%, respectively. These results show that cellulose film experienced complete degradation on day 5 due to its poor hydrophobic properties, allowing it to absorb water easily and be easily degraded by soil microorganisms. However, GraftCell/CitO1.2 film showed a slower degradation because it had better hydrophobic properties and contained fewer hydroxyl groups than cellulose film. Moreover, citronella oil in GraftCell/CitO1.2 film has antimicrobial properties that slow down bacteria's breakdown of polymer chains.[56]

As shown in Fig. 9, the porosity, water absorption, and swelling properties of the films are highlighted compared to conventional plastics. The swelling percentage of grafted cellulose film and GraftCell/CitO1.2 film was significantly lower than that of cellulose film, indicating improved dimensional stability under moisture exposure. Conventional plastic exhibited no swelling due to its complete impermeability, but its non-biodegradable nature limits its environmental sustainability. In contrast, the controlled swelling observed in grafted cellulose film and GraftCell/CitO1.2 film demonstrates their adaptability to environmental conditions while maintaining biodegradability. The reduced swelling in grafted cellulose film and GraftCell/CitO1.2 film is attributed to the grafted GMA onto cellulose, which enhances cross-linking and minimizes free volume within the polymer matrix.[57] For GraftCell/CitO1.2 film, the addition of 1.2% CO further improved stability by introducing hydrophobicity and reinforcing the matrix structure. This dual modification contributed to a denser and more water-resistant film.

Comparable results have been reported in the literature, such as Yu et al.,[58] who demonstrated that incorporating 2.25% chitosan oligosaccharide (COS) reduced swelling to 46% in native bacterial cellulose, underscoring the effectiveness of cross-linking and hydrophobic additives in enhancing film properties. Similar findings were also reported by Biswas et al.[59] and Bazzaz et al.[60] who observed that embedding essential oils such as *Mentha pulegium* and eugenol in carboxymethyl cellulose (CMC) films decreased water absorption.[59,60] Another study reported that the presence of essential oils can reduce permeability to water, which directly correlates with reduced

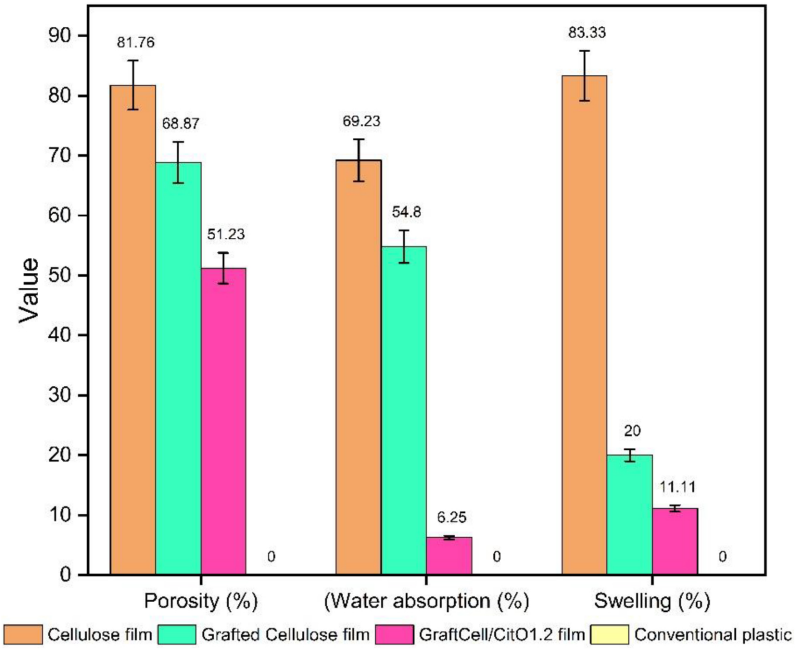

Fig. 9. Porosity, water absorption, and percentage of swelling of films and conventional plastic.

swelling and enhances the mechanical properties of the films.[61] These studies highlight the consistent impact of hydrophobic additives in reducing swelling and water uptake in biodegradable film matrices, supporting the modifications applied in the present study.

Fig. 9 shows that the porosity of the grafted cellulose film is significantly lower than that of the cellulose film, indicating a denser pore structure in the grafted cellulose film. Because fewer pores diminish water-penetration pathways, this structural feature is associated with reduced water absorption in grafted cellulose films. Notably, the porosity of the GraftCell/CitO1.2 film (51.23%) is even lower than that of cellulose and grafted cellulose films, indicating a more compact structure. On the other hand, conventional plastic is completely impermeable to water because it lacks porosity. The water absorption ratio, calculated as the percentage increase in the film's weight after water exposure, illustrates the film's resistance to water. Grafted cellulose film and GraftCell/CitO1.2 film show markedly lower water absorption than cellulose film, further confirming their superior water resistance. This property is crucial for assessing the suitability of these films as alternatives to conventional plastics for food packaging performance.

Previous studies reported that cellulose nanofibers (CNF) derived from spinifex grass, when surface-modified via grafting with epoxidized soybean oil (ESO), exhibited improved hydrophobicity. The water absorption capacity of ESO-grafted nano-paper was reduced by 40% compared to untreated nano-paper, highlighting the effectiveness of such grafting techniques in enhancing water resistance.[62] The cellulose film obtained in this work has a very high water absorption (69.23%) because cellulose has poor hydrophobic properties. Grafted cellulose film experienced a decrease in the water absorption (54.8%.) This shows that the GMA monomer bonded with cellulose is able to increase its hydrophobic properties so that the film is able to prevent the absorption of water from entering the polymer. The decrease in water solubility was caused by a decrease in the number of –OH bonds and the presence of aliphatic groups in the film when GMA was added, as demonstrated in FTIR analysis Fig. 2. This causes water molecule repulsion, so it is less able to absorb water on the film. Interestingly, the GraftCell/CitO1.2 film exhibited a reduction in water absorption percentage, i.e., 6.25%, which is eight times lower than that of the grafted cellulose film and eleven times lower than the cellulose film. These results indicate that the addition of citronella oil can decrease water absorption and enhance the hydrophobic properties of the film. Similar findings were reported by Imawan et al.[53] where the addition of citronella oil to chitosan/ascorbic acid films reduced water solubility from 24.58% to 20.41%. This decrease in water solubility is attributed to the reduction in –OH bonds and the presence of aliphatic groups in the film when citronella oil is incorporated. These structural changes create water molecule repulsion, reducing the film's capacity to absorb water.[63]

Performances of films as grapes packaging

The shelf-life of grapes packaged with the GraftCell/CitO1.2 film and conventional plastic was evaluated for 9 days to assess the films' effectiveness in maintaining fruit quality. As shown in Fig. 10, the grapes' emergence was seen on the seventh and ninth days. After seven days of storage, grapes wrapped in the GraftCell/CitO1.2 film showed a vibrant color and a fresh appearance. Those packaged in traditional plastic, on the other hand, had obvious surface wrinkles and color fading. The GraftCell/CitO1.2 film-coated grapes showed minor shrinkage and discoloration by day nine, but they were still aesthetically pleasing. Grapes wrapped in traditional plastic, on the other hand, showed noticeable discoloration, rotting, and extreme shrinking.

These results unequivocally show that the GraftCell/CitO1.2 biofilm effectively maintains fruit freshness and delays spoilage, suggesting its potential as a biodegradable packaging material. The citronella oil

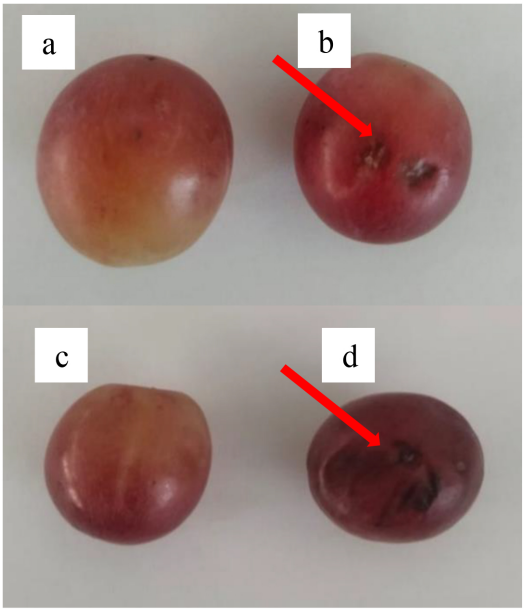

Fig. 10. Visual photograph of grapes packed using GraftCell/CitO1.2 film (a,c) and conventional plastic (b, d) after 7th (a, b) and 9th (c, d) day.

added to the grafted cellulose matrix, which has antibacterial and fungal activity, is probably responsible for this improved preservation capacity. Compared to traditional plastics, the components of essential oils (citronellal, geraniol, and citronellol) are known to damage microbial cell membranes and suppress metabolic activity, thereby slowing fruit deterioration. The incorporation of essential oils can improve surface hydrophobicity and antibacterial efficacy, as shown in a recent study.[64] However, matrix plasticization effects may also affect water vapor and gas permeability.

Mei[65] reported a similar outcome, finding that over an 18-day storage period, grapes coated with a cellulose-based film containing Chinese fir essential oil (CFEO) showed a lower decay percentage than those wrapped with commercial plastic. Additionally, Agusnar et al.[38] showed that adding essential oils (such as patchouli oil) to a biopolymer matrix enhanced the films' resistance to heat and microbes. Together, our findings imply that adding citronella oil to the GMA-grafted cellulose network improves both biodegradability and preservation performance. The addition of functional additives, such as citronella oil, to our GMA-grafted cellulose system likely improves preservation through combined antimicrobial, antioxidant, and light-barrier mechanisms, comparable to results from a recent study using carbon-dot-reinforced biopolymer films for fruit preservation.[66,67]

To maximize its use for perishable food packaging, more research on the antibacterial processes and release kinetics of citronella oil in the composite matrix is advised. Furthermore, to objectively verify the film's functional performance, future research will focus on conducting standardized barrier and microbiological tests (e.g., WVP, OTR, contact angle, TVC, and MIC). The observed preservation trends indicate that the GraftCell/CitO1.2 film effectively prevents visible decomposition compared to ordinary plastic. Even if the current evaluation was based on visual examination rather than quantitative measurement (e.g., mass loss, microbiological counts, hardness, or SSC/TA), standardized physicochemical and microbiological measures will be used in subsequent research to validate these results quantitatively.

Conclusion

A new cellulose film with superior properties has been developed by grafting cellulose with glycidyl methacrylate (GMA) prior to film production and incorporating citronella oil during its preparation. This innovative film demonstrates improved hydrophobicity, enhanced mechanical properties, and greater thermal stability. Additionally, it shows decreased water absorption, porosity, and swelling, demonstrating its superior performance as a food packaging performance. This film has enormous potential as a bioplastic substitute for traditional non-biodegradable plastics since it is both environmentally benign and biodegradable. The physicochemical characteristics of cellulose films have been successfully improved by the grafting method, opening the door for useful and sustainable packaging. While the developed films showed favorable mechanical and preservation behavior, future work will include standardized barrier analyses to quantitatively confirm packaging suitability.

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures in the manuscript are ours. Furthermore, any Figures and images that are not ours have been included with the necessary permission for republication, which is attached to the manuscript.
- No animal studies are present in the manuscript
- No human studies are present in the manuscript
- Ethical Clearance: The project was approved by the local ethical committee at Universitas Syiah Kuala.

Authors' contributions statement

R.R. writing – reviewing & editing, supervision, methodology, conceptualization, resources. F.F., and L.L. writing – reviewing & editing, supervision. S.S. writing – original draft, investigation, formal Analysis. A.A.K. writing – original draft, visualization, writing – reviewing & editing.

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تصنيع وتوصيف غشاء قائم على السليلوز المطعَّم بغليسيديل ميثاكريلات لأغراض تغليف الأغذية

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الخلاصة

لا يزال استخدام البلاستيك غير القابل للتحلل في تغليف الأغذية يثير العديد من المشكلات العملية والبيئية، مما عزز الاهتمام بالبدائل القابلة للتحلل الحيوي والمشتقة من الكتلة الحيوية. في هذه الدراسة، استُخدم السليلوز المستخلص من مخلفات جوز الهند مادةً مكوّنةً للأغشية. وقد جرى عزل السليلوز باستخدام مزيج من المعالجات الكيميائية والموجات فوق الصوتية، تلا ذلك تطعيمه بمركب غليسيديل ميثاكريلات (GMA). كما أُضيف زيت السترونيلا (CitO) بتراكيز مختلفة لإنتاج أغشية GraftCell/CitO0.15 وGraftCell/CitO0.6 وGraftCell/CitO1.2.. أكد تحليل مطيافية الأشعة تحت الحمراء بتحويل فورييه (FTIR) حدوث عملية التطعيم، في حين انعكست التغيرات البنيوية في ارتفاع مؤشر التبلور من 10.55% إلى 16.04%. كذلك أظهر التحليل الوزني الحراري (TGA) انتقال درجة حرارة بدء التحلل من 254.6°م(Cell) إلى 369.6°م(GraftCell) ، مع وصول درجة الحرارة العظمى للتحلل إلى 477.8°م، مما يشير إلى تحسن المقاومة الحرارية بعد التعديل. وقد تباينت الخواص الميكانيكية تبعًا لمحتوى زيت السترونيلا؛ إذ أظهر غشاء GraftCell/CitO1.2 مقاومة شد واستطالة عند الكسر بلغت 25.63 ميغاباسكال و21% على التوالي، وهي قيم تزيد بنحو ثلاثة أضعاف مقارنةً بغشاء السليلوز غير المعالج. كما أظهرت اختبارات مدة الحفظ أن حبات العنب المغلفة بغشاء GraftCell/CitO1.2 احتفظت بمظهر مقبول بصريًا لمدة تصل إلى 9 أيام، مقارنةً بخمسة أيام فقط عند استخدام التغليف البلاستيكي التقليدي، مما يدل على تحسن يقارب 80% في كفاءة الحفظ. وتبرز هذه النتائج الإمكانات الواعدة لغشاء GraftCell/CitO1.2 بوصفه مادة تغليف مستدامة وقابلة للتحلل الحيوي.

الكلمات المفتاحية: الغشاء القابل للتحلل الحيوي، السليلوز، زيت السترونيلا، غليسيديل ميثاكريلات (GMA)، التطعيم البوليمري.