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Research article

Insights into physicochemical properties, optimization using response surface methodology, and green metrics evaluation of chitosan–cassava starch–essential oil edible films

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ABSTRACT

This study aimed to develop and evaluate biodegradable edible films composed of chitosan, cassava starch, glycerol, and essential oils via the solution-casting method. The optimal formulation for tensile strength was achieved using the Box-Behnken Design (BBD) with chitosan (1.5–2 g), starch (0.5–1 g), and glycerol (0.5–1.5 mL). The essential oil concentration was 0.5 mL. The incorporation of essential oils influenced film thickness, moisture content, swelling, solubility, optical properties, chemical, mechanical, and thermal properties. All films were fully degraded within 3 days. The green metric evaluation demonstrated good environmental performance, fulfill the majority of green chemistry principles. These findings suggest that the films hold strong potential for sustainable active packaging applications.

1. Introduction

Packaging materials are among the tools that ensure the quality, shelf life, and safety of products [1,2]. The use of plastic from petroleum that is not biodegradable, nor renewable [3], causes environmental pollution [4,5]. One strategy is applying edible films [6], which are safe for consumption [7], biodegradable [8], renewable [9], and environmentally friendly [10]. The manufacture of edible films has been widely studied to be developed and modified to obtain optimal conditions for their use. Edible film can enhance food stability by minimizing the transfer of gases, volatile compounds, moisture, and lipids between the food and its environment [3].

Materials for edible films must be abundant, inexpensive, and have good mechanical and protective properties [11]. After starch and cellulose, chitosan, a linear polysaccharide produced by deacetylation of chitin, is the second most common polymer found in nature [12–17]. It has a variety of distinctive characteristics and properties that portray a versatile material suitable for a broad range of applications [18]. Chitosan has functional groups that are easy to modify [19], can thicken the film. Chitosan functions as an antimicrobial agent [20,21] increasing UV resistance and mechanical properties of the film [22].

Starch is one of the abundant and biodegradable natural materials [23], has a large molecular weight, is cheap, has low degradability, and good biocompatibility. Starch has been widely studied as a selective polysaccharide coating with good protective properties [24]. Starch can form film layers due to its transparency and its amylose and amylopectin content [25]. Even so, starch-based films are subject to certain limitations, such as low mechanical strength, brittleness, and excessive water solubility. These drawbacks can be addressed by incorporating plasticizers or blending them with other polymers that offer superior mechanical performance, thereby improving the films' flexibility and structural integrity [26].

The use of Response Surface Methodology (RSM) in industry can balance safety, sustainability, and functionality and meet the growth requirements of consumers and regulatory bodies. Efficient, more environmentally friendly processes are considered in the industrial sector for sustainable preparation and ecological assessment [27–32]. The use of RSM can drive innovation and improve the environmental impact and overall quality of packaging solutions [33]. Optimization using RSM on chitosan edible film was studied by [34] and [35]. In

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addition, the chemical sustainability aspect can be studied using the green metrics value [36].

Previous studies have investigated chitosan-based edible films with individual additions such as cinnamon essential oil [37], starch [38], and glycerol plasticizer [39]. However, limited research has combined chitosan, cassava starch, glycerol, and various essential oils, specifically anise, cinnamon, and lime oil, into edible film formulation. Moreover, comprehensive evaluations covering both functional and sustainability-related properties are scarce. This study addresses these gaps by developing and optimizing a multifunctional edible film using RSM, followed by extensive characterization and evaluation of its physical, mechanical, chemical, and green metrics.

2. Materials and methods

2.1. Materials

Chitosan (DD: 97.4%; MW: 52.12 kDa) from Phy Edu Media. Starch was isolated from cassava from the Sukoharjo area. Glycerol and aquadest from CV. Agung Jaya Solo. Glacial acetic acid from Cipta Kimia. Anise oil, cinnamon oil, and lime oil from Happy Green.

2.2. Methods

2.2.1. Experimental design and edible film preparation

Design-Expert version 13.0.5 software (Stat-Ease, Inc.) was used to optimize the edible film formulation using Response Surface Methodology. In this work, chitosan (X_1), starch (X_2), and glycerol (X_3). As shown in Table 1, the independent variables were coded at three levels (-1 for low, 0 for center, and $+1$ for high). Fifteen experimental runs were performed to build the desired model. Tensile strength was used as the dependent variable.

2.2.2. Preparation of edible film enriched with essential oil

The optimum formulation consisted of 1.5 g chitosan, 0.75 g cassava starch, and 0.5 mL glycerol. The essential oils were anise oil (AEO), cinnamon oil (CEO), and lime oil (LEO). The addition of essential oils is based on past research [40], with modifications. Chitosan and starch were dissolved in acetic acid solution until gelatinized. The formed film solution was cooled to RT, then 0.5–1.5 mL of glycerol and essential oil were added to the mixture, which was stirred for 10 min. The mixture was poured into a petri dish and dried at 37.5 °C for 40 h.

2.2.3. Statistical analysis

The optimum formulation was predicted using response surface and contour plot analyses generated by the RSM model. The adjusted R (Adj-R^2) and R-squared (R^2) were used as benchmarks for validating the research model. The average differences in sample values were distinguished using variance analysis. Comparison of means was supposed to be significant at $p < 0.05$.

Table 1
The Box-Behnken Design for the optimization of the edible formulation.

Run	Materials			Coded Variables			Response (MPa)
	Chitosan (g)	Starch (g)	Glycerol (mL)	X1	X2	X3	
1	1.5	1	1	−1	+1	0	0.663
2	1.5	0.75	0.5	−1	0	−1	4.830
3	1.5	0.75	1.5	−1	0	+1	0.355
4	1.75	0.5	0.5	0	−1	−1	3.520
5	1.75	1	0.5	0	+1	−1	3.090
6	1.75	0.75	1	0	0	0	0.988
7	2	0.75	0.5	+1	0	−1	1.900
8	1.75	1	1.5	0	+1	+1	0.613
9	2	1	1.5	+1	0	+1	2.650
10	1.5	1	1.5	0	−1	+1	0.279
11	1.5	0.5	1	0	0	0	1.110
12	2	1	1	+1	+1	0	1.040
13	2	0.5	1	+1	−1	0	0.959
14	1.75	0.75	1	0	0	0	1.180
15	1.5	0.5	1	−1	−1	0	0.644

2.2.4. GC-MS analysis of essential oil

The main components of the essential oil were determined using a GC-MS Autosystem XL-Turbo Mass (PerkinElmer, USA). A capillary column (30 m x 0.25 mm×0.25 µm) coated with 5% phenyl methylpolysiloxane was used. The AEO analysis followed the method reported by Ghosh et al. [41]. Meanwhile, the identification of CEO and LEO referred to Zhao et al. [42] and Thonglem et al. [43], respectively. The resulting chromatograms were interpreted by contrasting the mass spectra of each peak with entries in the NIST library database integrated within the system.

2.2.5. Characterization of edible film

The thickness was measured using a digital thickness gauge. The film samples were measured for thickness in mm. Measurements were taken at 3 random points for each film sample, and the average value was calculated [44]. Characterization of moisture content (MC), swelling (S), and water solubility (WS) was carried out simultaneously, following the protocol by Istiqomah, Prasetyo, et al. [38].

The films were inspected for their morphology using a Microscope (Leitz Wetzlar, Germany) with 40x magnification and 0.12 NA. During observation, measurements were made using a mechanical stage with a built-in scale. A value of "140" was recorded on the stage scroll. The surface was also observed using a SEM (Zeiss Evo 10, Germany). First, the film was coated with palladium. The test run was with an acceleration voltage of 5 kV. The view was observed using 1000x magnification. The optical properties were measured based on Othman, Nordin, et al. [45]. The opacity value was calculated as UV-Vis absorbance at 600 nm divided by film thickness (mm). The mechanical properties were determined using a Universal Testing Machine (Zwick Roell 20, Germany) in accordance with ISO 527/ASTM D638. The tested film strip has a thickness of < 14 mm. The film was cut to 2 cm wide by 10 cm long. Testing was carried out in a rectangular shape without making a dog bone-like shape. Pre-load: 0,2 N, Speed, tensile modulus: 10 mm/min, test speed: 50 mm/min, and grip-to-grip separation at the start position: 100 mm.

Characterization of functional groups was carried out using ATR-FTIR (Agilent Cary 360). Samples were recorded with a mean of 20 scans per sample in the 4000–400 cm^{-1} range. The thermal properties were studied using the TGA-DTA (Linseis PT1600, Germany). The sample's weight loss was assessed as a function of temperature. The film was heated at a constant rate of 10 °C/min at a temperature range of 30–700 °C.

The biodegradability test of edible film refers to the research of Nigam et al. [46], Istiqomah et al. (2022a), and Siti Hajar Othman et al. [26], which was modified. The film was cut into 20 × 20 mm pieces, dried in an oven at 80 °C for 24 h, weighed, and then planted in the soil to a depth of ± 20 mm. Natural biodegradation can be determined by measuring the film's weight daily for 2 days. The film was cleaned with a brush to remove soil residue, then dried in an oven at 80 °C for 1 h to remove moisture. The biodegradability of the film was calculated from the percentage of weight loss and the degradation rate.

2.2.6. Evaluation of green metrics

The evaluation of sustainability aspects in the form of green metrics is based on previous research [36,47–49]. The E factor, Mass Intensity, Water Intensity, and Reaction Mass Efficiency were determined. Then, the greenness was visualized using a holistic Green Star.

3. Results and discussion

3.1. Fitting the tensile strength using RSM and statistical analysis

The Box-Behnken Design parameters generated for the optimization of the edible formulation are shown in Table 1.

A second-order quadratic polynomial, given in Eq. (1), was employed to describe the response to the selected parameter, which indicates the magnitude of response variation resulting from a unit change in the factor while other parameters remain constant. Based on Table 2, the *p*-value was used to determine whether each coefficient was statistically significant. The f and *p* values in the model were significant. The insignificant lack-of-fit results were obtained from the model transformation using the square root.

$$TS = (1.04 + 0.0744\,A + 0.0236B - 0.4594\,C + 0.0072AB + 0.4628AC + 0.0933BC + 0.0365A^2 - 0.1772B^2 + 0.3688\,C^2)^2 \quad (1)$$

The predicted and actual R2 values are shown in the Supplementary Data. The difference in values of more than 0.2 is due to the risk of overfitting. The optimum formulation with the highest tensile strength response was obtained from Run 2, with a desirability of 1. The predicted tensile strength was 4.913 MPa, and the actual data was 4.830 MPa. The difference between the predicted and actual values from the experiment was 0.083 with a percentage error value of 1.69%. It can be concluded that the model can be used to predict the next tensile strength response. This is also supported by the R^2 value, which explains 96.3% of the variability in tensile strength. The high adjusted R^2 value confirms the model's reliability. The Adeq Precision value shows a strong signal-to-noise ratio.

A *p*-value less than 0.05 was considered statistically significant. Although some factors showed a positive or negative coefficient in the regression equation, only those with significant *p*-values were interpreted as having a reliable effect on the response. Based on Table 2, the values of A (Chitosan), A^2, B (Starch), and B^2 have *p*-values of more than 0.05. These values indicate that A and B are not significant. Thus, the addition of chitosan and starch to the edible film does not greatly influence the tensile strength. On the other hand, the values of C (glycerol) and C^2 show significant *p*-values. Glycerol has an effect on the edible film. Excess glycerol can cause a strong nonlinearity in tensile strength.

The 3D plots of AB, BC, and AC are shown in Fig. 1. The AB value (Chitosan-Starch) with a p-value of 0.9315 and a coefficient of 0.0072 in Eq. (1) indicates that the interaction between chitosan-starch is not significant in increasing tensile strength. Interpretation of the 3D and contour plots of AB shows a flat or slightly curved response surface, with no strong peaks or valleys. Simultaneously increasing chitosan and cassava starch did not significantly affect tensile strength. The AB interaction is weak because the individual factors A and B are also insignificant.

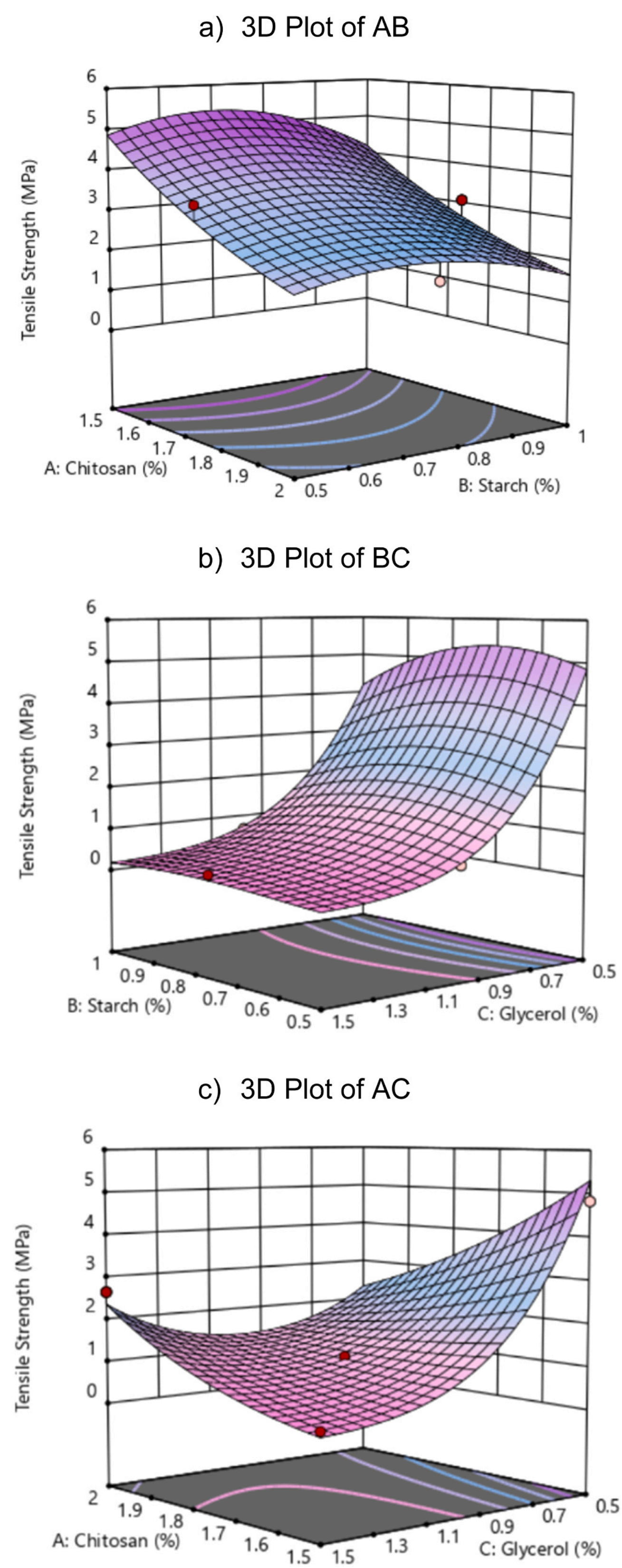

Fig. 1. 3D Plot of AB (a), BC (b), and AC (C).

The BC value (Starch-Glycerol) with a p-value of 0.2939 and a coefficient of 0.0933 in Eq. (1) indicates that the interaction between

Table 2
ANOVA and the coefficients of the quadratic model.

Source	Sum of Squares	df	Mean Square	F-value	*p*-value	
Model	3.29	9	0.3656	14.44	0.0045	significant
A	0.0443	1	0.0443	1.75	0.2433	
B	0.0045	1	0.0045	0.1758	0.6924	
C	1.69	1	1.69	66.70	0.0004	
AB	0.0002	1	0.0002	0.0082	0.9315	
AC	0.8569	1	0.8569	33.85	0.0021	
BC	0.0348	1	0.0348	1.37	0.2939	
A^2	0.0049	1	0.0049	0.1944	0.6777	
B^2	0.1159	1	0.1159	4.58	0.0854	
C^2	0.5023	1	0.5023	19.84	0.0067	
Residual	0.1266	5	0.0253			
Lack of Fit	0.1222	3	0.0407	18.60	0.0514	not significant
Pure Error	0.0044	2	0.0022			
Cor Total	3.42	14				

starch-glycerol has a slight effect on increasing tensile strength. Interpretation of the 3D plot and contour plot of BC shows a slight curvature in the contour line, but it does not provide a strong effect. Since the C value is very significant, the effect is mostly due to glycerol. The optimal region will mostly align with glycerol concentration rather than the level of cassava starch added.

The AC value (Chitosan-Glycerol) with a p-value of 0.0021 and a coefficient of 0.4628 in Eq. (1) indicates a strong interaction between chitosan-glycerol. Interpretation of the 3D and contour plots of AC shows curvature with visible peaks and valleys. This indicates the optimal range of chitosan and glycerol. The optimal area is most clearly obtained in the AC plot because of the significant C and AC. The addition of chitosan and the reduction of glycerol can improve tensile strength. Glycerol increases polymer chain mobility and reduces intermolecular interactions, particularly hydrogen bonding between chitosan and starch molecules. As a result, the film matrix becomes more flexible but mechanically weaker, leading to decreased tensile strength at higher glycerol concentrations.

The optimum formulation with the highest tensile strength response was obtained from Run 2, with a desirability of 1. The prediction value of tensile strength was 4.913, and the actual value was 4.830 MPa. The gap between the predicted and actual values from the experiment of 0.083, it can be concluded that the model can be used to predict the next tensile strength response. This is also supported by the R^2 value, which explains 96.3% of the variability in tensile strength. The high adjusted R^2 value confirms the reliability of the model. The Adeq Precision value shows a strong signal-to-noise ratio.

The use of chitosan, cassava starch, and glycerol was in accordance with the Run 2 (EF) formula. The edible film enriched with essential oil obtained was 9 samples with the formulation in Table 3. The resulting edible film was then tested for tensile strength, and the optimal formula was determined.

Based on Table 3, the tensile strength of the edible film tended to decrease as the concentration of essential oil increased. This may be attributed to the easier incorporation of essential oil into the biopolymer matrix, thereby reducing intra- and intermolecular interactions [50]. Based on these results, further characterization was carried out on the edible films EF, EF/AEO, EF/CEO, and EF/LEO, each containing 0.5 mL of essential oil.

3.2. The main component of essential oils

The chemical composition of the EO incorporated into the edible films was identified using GC-MS. These three dominant compounds for each EO were used to further interpret their effects on film characteristics. The three main components were selected based on the relative abundance (% area) and the highest similarity index (SI). The AEO was rich in trans-anethole (82.85%; SI: 924), followed by linalool (4.15%; SI: 961) and α-pinene (3.25%; SI: 934).

Trans-anethole has a strong odor, is water-soluble, is highly volatile, has antimicrobial properties, and is low in physicochemical stability [51]. The compound exhibits a sweet, herbaceous odor and a sweetness nearly 10 times that of conventional sugar. It is regarded as food-grade and has been affirmed as safe by FEMA and the FDA [52]. The log P value of trans-anethole is 3.3.

The CEO showed cinnamaldehyde (70.53%; SI: 928), cinnamyl ester (8.38%; SI: 961), and eugenol (7.56%; SI: 950) as major compounds. Meanwhile, the major compounds of LEO are limonene (52.40%; SI: 894), γ-terpinene (19.33%; SI: 908), and β-pinene (13.81%; SI: 906). Cinnamaldehyde, a yellowish oily liquid, is one of the most popular EOs. It has high volatility, is unstable [53], is a hydrophobic aromatic aldehyde with low molecular weight, and exhibits high antibacterial activities. It's considered safe by the Joint FAO/WHO Expert Committee on Food Additives [54]. The log P value of cinnamaldehyde is 1.9. Limonene is a colorless liquid having a lemon-like odor [55]. Limonene-enriched EO to show substantial inhibitory effects against prevalent foodborne pathogens. It is generally recognized as safe (GRAS) [56] and has a log P value of 3.4. All the log P values were obtained from the PubChem database. Detailed retention times, peak areas, and compound identifications of the essential oils are presented in Supplementary Information.

3.3. Physicochemical properties of edible film

The physicochemical properties of edible film are shown in Table 4. All edible films produced have a maximum thickness of 0.250 mm [57]. The inclusion of EO into edible film can significantly affect the film's thickness, depending on its chemical characteristics and interactions with the film-forming matrix. The addition of LEO increased film thickness, which may be associated with its relatively high log P value and greater hydrophobicity, both of which limit its compatibility with chitosan/starch. This leads to phase separation and a looser structure, thereby increasing the film's thickness. In contrast, the EF/CEO film is thinner than the EF film. This could be attributed to the low log P of cinnamaldehyde. This value allows the CEO to form hydrogen bonds with the hydrophilic polymers, resulting in a more compact matrix and thinner film.

The EF/AEO showed similar MC with EF. This behavior may be attributed to the trans-anethole, which can interact with the film matrix, enhancing cohesion and reducing water affinity and molecular mobility. The CEO and LEO increased the MC. Those changes could be attributed to the natural oily character of CEO and LEO [38].

Water will be absorbed by the film and make the film smooth and swell [58]. Both AEO and CEO contributed to a reduction in swelling properties. This may be attributed to the ability of AEO and CEO to integrate well with the polymer matrix, enhancing compactness and reducing water uptake. It can also be shown based on the film's thickness. Interestingly, EF/LEO exhibited slightly higher swelling values. This behavior may be associated with structural heterogeneity and reduced compatibility between lime essential oil and the hydrophilic polymer matrix, which could facilitate water penetration into certain regions of the film.

The main component of CEO, cinnamaldehyde, has a low log P, making it more hydrophilic. It facilitates water uptake and increases the

Table 3
The tensile Strength of Edible Film Enriched with Essential Oil.

Materials	Volume (mL)	Tensile Strength (MPa)
EF/AEO	0.5	4.41
	1	3
	1.5	2.94
EF/CEO	0.5	10.9
	1	3.24
	1.5	2.19
EF/LEO	0.5	12.2
	1	6.27
	1.5	2.39

Table 4
Physicochemical properties of edible film.

Material	Thickness (mm)	MC (%)	Swelling (%)	WS (%)	EAB (%)	YM (MPa)
EF	0.1067 ± 0.0058	13.6364 ± 0.0003	98.2805 ± 0.0006	54.5918 ± 0.0013	35	8.27
EF/AEO	0.0733 ± 0.0058	13.3333 ± 0.0006	95.5857 ± 0.0009	54.4776 ± 0.0003	33	8.7
EF/CEO	0.0867 ± 0.0058	16.1417 ± 0.0006	98.0903 ± 0.0003	73.9496 ± 0.0013	25	32.4
EF/LEO	0.0867 ± 0.0058	20.5223 ± 0.0006	98.8229 ± 0.0003	46.8750 ± 0.0003	14	11.6

All measurements were repeated three times.

water solubility. In contrast, the main component of LEO, limonene, has a high log P, whichs decrease in WS. The observed reduction can be explained by the hydrophobic properties of the essential oil. The addition of oil diminished the number of hydroxyl groups and the presence of aliphatic components in the film, producing a less soluble matrix. Consequently, water molecules were repelled, limiting their penetration and dissolution within the film [59]. Nevertheless, further studies on film–water interactions are required to confirm the underlying mechanism.

The addition of essential oils to the edible film causes a decrease in the EAB value of the edible film. This can be obtained from the appearance of additional functional groups in essential oils. These functional groups cause deformation at the polymer position and cause a less stable structure [38]. Elongation decreases with increasing tensile strength due to the large number of hydrogen bonds between starch chains, which cause high tensile strength, thereby reducing molecular mobility in the starch layer [60].

An increase in the tensile strength value indicates an increase in film stiffness and an increase in the Young's modulus value [61]. These results are comparable to the tensile strength test. The addition of essential oils to edible films increases the Young's modulus, strength, and stiffness, and provides an elastic effect on the film. These results are in accordance with the previous studies [62,63]. The morphology of edible film is shown in Fig. 2. The edible film without essential oil has an uneven, coarse pattern. This attributed to the dehydration of moisture during the film preparation [64]. In contrast, the addition of EOs showed a homogenous, smooth pattern in the film's surface. This result aligns with previous studies [40,65–67].

Films incorporated with EO showed numerous small patches. This could be attributed to moisture disturbance in the film. Even though this study used the same concentration of each EO, the film's pattern is slightly different. This could be the different components of EOs [64]. The EF/AEO film displayed the most uniform film. This indicated that AEO could interact well with the EF. This is consistent with the thickness, MC, S, and WS reported in this study. The dispersion of oil droplets was absent on the edible film without essential oils, whereas the edible film enriched with EOs exhibited the presence of numerous droplets [68]. The addition of essential oils into the film matrix caused some gaps and holes. This may be due to the hydrophobic nature of the essential oils [69].

The results of the color and opacity tests are shown in Table 5. The addition of CEO and LEO decreased the a* value. The decreased a* value indicates that the film becomes greenish. Meanwhile, the addition of AEO did not significantly affect the EF film. The films also slightly yellow, which was confirmed by the tiny positive b* values. The addition of EOs increased the b* value. The highest b* value was observed from EF/CEO, followed by EF/LEO and EF/AEO. These results could be attributed to the type of essential oil, which showed different original colors [70] when observed visually.

The lightness (L*) value reflects the brightness of the film surface, with higher values indicating a lighter appearance. Adding EOs decreases the lightness value, making the film slightly darker. This decrease can be caused by an increase in diffuse reflectance due to light scattering in EO, which reduces the film's whiteness index [70]. The total color difference (ΔE) was measured to assess the overall color variation between the films. All the EOs increased the ΔE. The differences between each ΔE are small and not noticeable visually.

Opacity is measured based on light absorbance at 600 nm by the film. The thickness of the film affects the light absorption [71]. The droplet of EOs in the film's matrix blocks the light passage. This addition contributed to greater film opacity, thereby improving its ability to block ultraviolet (UV) radiation [70].

The visual appearance of the edible film is shown in Table 6. The film changed after being planted for 3 days with the planting medium. The film experienced deterioration in terms of size, appearance, and color changes. This indicates a change in biological properties that indicates degradation on the surface. On the 3rd day, no residue could be recovered, indicating complete degradation. This could be attributed to the biodegradability properties of chitosan [72] and starch [23].

The decomposition process in the soil causes the films to become hard, brittle, and easily broken, eventually decreasing in size. The film color turns black [63]. Microbial activity during burial causes changes in film weight. All films decreased in weight during the test period. Soil microbes can utilize the hydrophilic properties of chitosan and starch to proliferate and break down biopolymers with hydrolytic enzymes [58]. However, all films experienced different weight changes and degradation rates.

ATR FTIR characterization aims to determine the functional groups in the material. ATR FTIR spectra of chitosan, cassava starch, and edible film are shown in Fig. 3. The spectrum of chitosan is comparable to previous findings as reported [68,69]. A broad absorption band at approximately 3260 cm^{-1} represented O–H stretching, which overlapped with NH stretching in the same region. The C═O stretching (amide I) and NH bending (amide II), in chitosan is affiliated with a peak at 1585 cm^{-1}, and a small peak near 1630 cm^{-1}, respectively.

The broad band at 3288 cm^{-1} in the cassava starch spectrum is referred to as O–H stretching. The band at 2939 cm^{-1} is associated with symmetric and asymmetric C–H stretching. The band at 1639 cm^{-1} indicates C—O stretching (amide I). The peak at 1339 cm^{-1} was attributed to C–H bending vibrations of polysaccharides and to the stretching deformation of C–O–C linkages. The band at 1144 cm^{-1} is associated with C–O, C–C, and C–O–H stretching vibrations. The band at 991 cm^{-1} was assigned to C–O–H bending vibration [73].

Physical versus chemical interactions when two or more substances are mixed are observed as changes in characteristic spectral peaks [74]. The enhanced band in the region between 3200 and 3300 cm^{-1} could be attributed to the reduced extension of free OH or NH groups due to hydrogen bonding between the O–H groups of chitosan, cassava starch, and glycerol. This can strengthen the film structure. In general, these results are in agreement with several previous reports [38].

The absorption band in the range of 2900–3000 cm^{-1} is related to the aliphatic C–H group of cassava starch and glycerol. The band indicates the presence of both materials in the film matrix. The incorporation of cassava starch shifted the amino peak of chitosan from 1585 to 1639 cm^{-1} , indicating molecular interactions, most likely hydrogen bonding, between the hydroxyl (–OH) groups of cassava starch and the amino (–NH) groups of chitosan [74]. This shift indicates a stronger hydrogen bond and has the potential to increase the compactness of the film and affect its mechanical properties.

The weight loss versus temperature curve of edible films is shown in Fig. 4. All samples showed the first degradation at 45–131 $^{\circ}C$. This degradation can be caused by unbound water, residual solvents, and evaporation of small molecules [75]. The addition of essential oils causes the presence of hydrophobic compounds that delay the evaporation of water and other components. The second stage of degradation at 150–330 $^{\circ}C$, which is possible due to the evaporation of glycerol, amino groups of chitosan, and organic acids. The final degradation occurs at a temperature of 580–700 $^{\circ}C$, where the biopolymer is degraded. Overall, the addition of essential oils makes the film more stable [76].

3.4. Evaluation of green metrics

Evaluation of green metrics in edible film preparation was carried out using calculations of E-factor, mass intensity (MI), RME, and water intensity (WI). The raw materials are biodegradable and renewable. The edible film production involved mechanical homogenization, heat treatment for gelatinization, and chemical treatment using acetic acid. This is because chitosan is insoluble in water [77]. The results obtained are shown in Table 7. The E-factor measures the efficiency of a reaction by accounting for waste formation, though water is generally excluded from this evaluation. The result shows the E-factor from all films is close to zero. This indicates that edible film production is environmentally

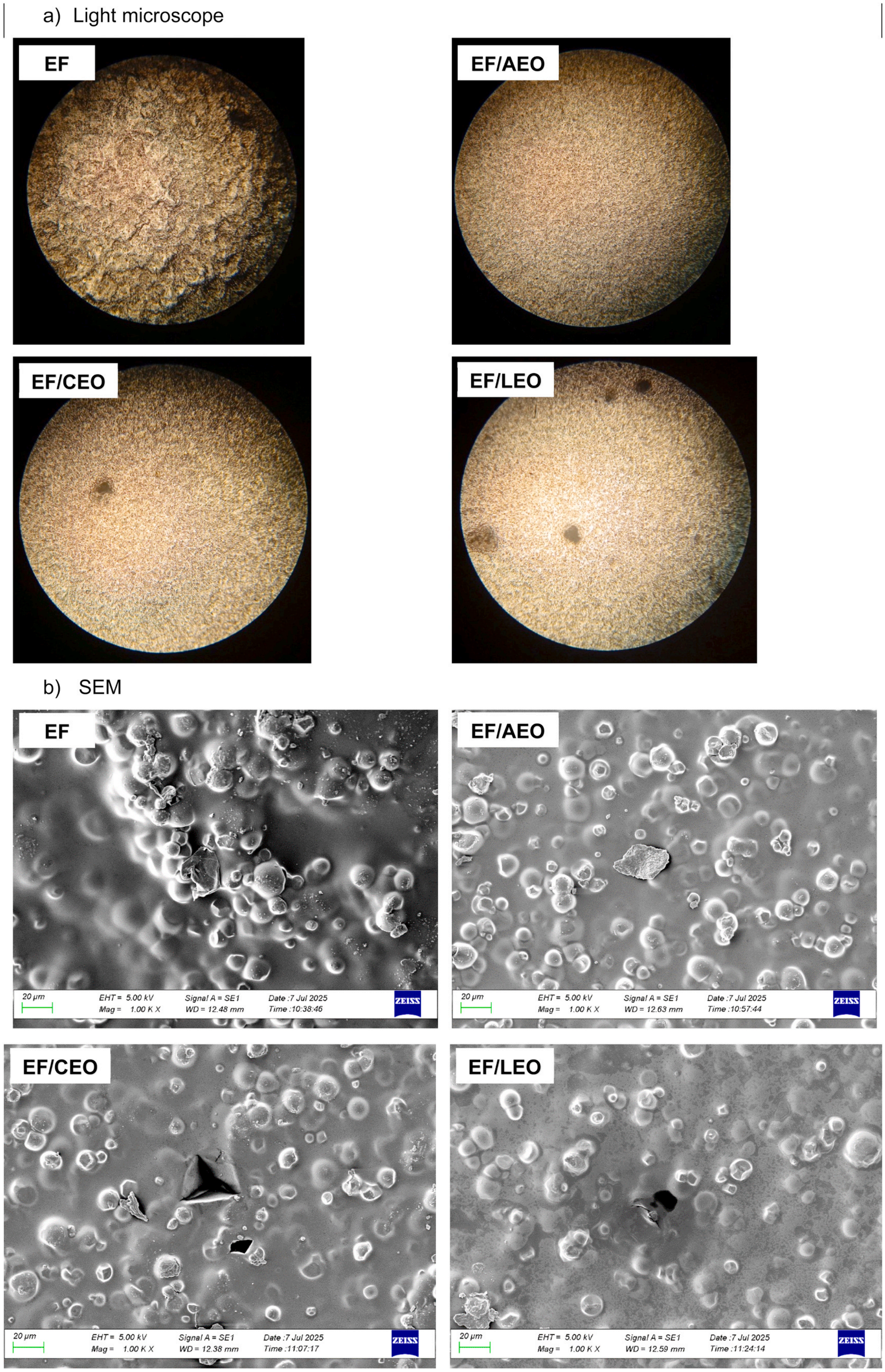

Fig. 2. The morphology of edible film using a light microscope (a) and SEM (b).

Table 5
Optical and biodegradability properties of edible film.

Material	a*	b*	L*	ΔE	Opacity (A_{600}/mm)	Weight Loss (%)		Degradation Rate (%)	
						Day 1	Day 2	Day 1	Day 2
EF	−0.1100 ± 0.0001	1.2100 ± 0.0001	93.7033 ± 0.0058	5.8067 ± 0.0058	0.8166 ± 0.0544	42.5532 ± 0.0003	64.8148 ± 0.0003	0.0133 ± 0.0001	0.0058 ± 0.0003
EF/AEO	−0.0700 ± 0.0001	1.5500 ± 0.0001	92.7733 ± 0.1960	6.7900 ± 0.1908	0.9350 ± 0.0683	13.7097 ± 0.0006	68.2243 ± 0.0001	0.0057 ± 0.0006	0.0122 ± 0.0006
EF/CEO	−0.3300 ± 0.0001	2.0800 ± 0.0141	92.2367 ± 0.0058	7.4400 ± 0.0001	1.1938 ± 0.0940	59.2105 ± 0.0003	22.5806 ± 0.0003	0.0150 ± 0.0003	0.0012 ± 0.0003
EF/LEO	−0.4067 ± 0.0058	0.8950 ± 0.0071	84.2267 ± 0.0058	15.1933 ± 0.0058	1.7837 ± 0.0966	21.5054 ± 0.0001	34.2466 ± 0.0001	0.0067 ± 0.0012	0.0042 ± 0.0006

All measurements were repeated three times.

Table 6
Visual appearance of edible film during biodegradation test.

Day	Edible Film			
	EF	EF/AEO	EF/CEO	EF/LEO
0				
1				
2				

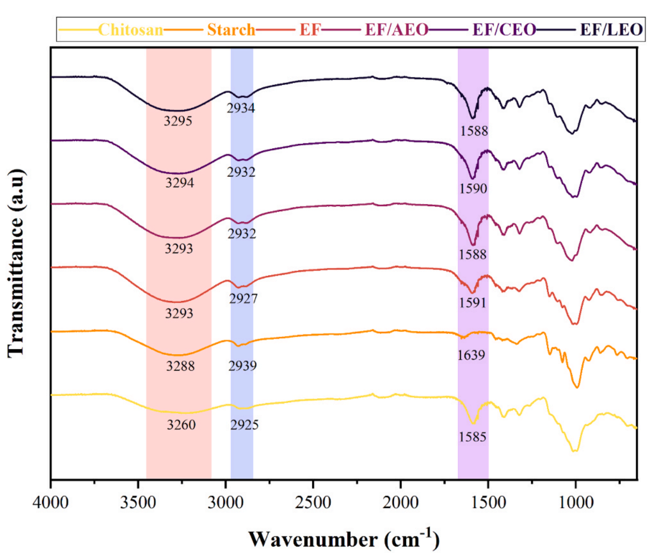

Fig. 3. ATR FTIR spectra of edible film.

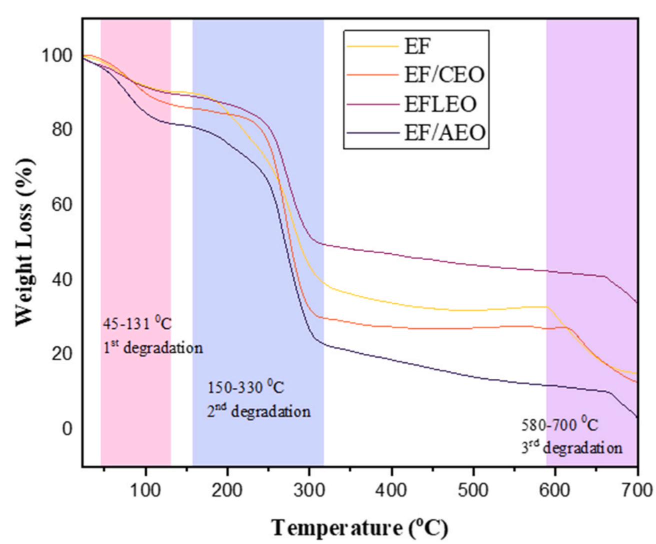

Fig. 4. TGA curve of edible film.

friendly [48]. This outcome aligns with the primary tenet of green chemistry, which prioritizes waste prevention over treatment or disposal after its generation [78]. Additionally, the almost-zero E Factor value means that for every gram of product made, there is just under a gram of waste.

The MI also excluded water. Constable and Curzons stated that the ideal MI should be close to 1. The MI results of this edible film are in the range of 2–3. Based on these results, only 2–3 g of raw material are needed to make every 1 g of product. This value shows material efficiency and is connected to the first and fifth principles. The RME value of edible film is between 35% and 42%. Higher RME values correspond to enhanced environmental performance of the edible film production process [48]. The RME value is directly related to the second principle. The higher the RME value, the fewer atoms are wasted. Meanwhile, the

Table 7
The green metric of edible film.

Material	E factor	MI (g/g product)	WI (g/g product)	RME (%)
EF	0.0337	2.7374	75.804	36.5315
EF/AEO	0.0354	3.0609	83.1933	32.6699
EF/CEO	0.0337	2.8389	78.1991	35.2254
EF/LEO	0.0272	2.1616	68.3702	46.2620

WI value of the edible film ranges from 68 to 83. Although water is a safe solvent (principle 5), this value is quite large and requires energy for evaporation. Indirectly, the WI value affects the 6th principle (energy efficiency).

Visualization of these values against the principles of green chemistry is shown in Fig. 5. The scale used is between 1 and 3. Scale 1 indicates excellent greenness, value 2 indicates moderate greenness, and 3 indicates poor greenness [49]. Based on the plotting results, excellent greenness was obtained in accordance with the principles P1, P3, P4, P5, P6, P7, P8, P10, and P12. The scale on P1 depends on the low value of the E factor. Scale 1 on P3, P4, and P12 is related to the use of GRAS materials. The use of 1% acetic acid indicates a safe solvent with a scale of 1 on P5. The drying process of the edible film at 37.5 °C produces a P6 scale of 1.

All materials used are renewable on P7. In addition, no products are produced, and the scale is 1 on P8. The materials and products of the edible film produced can be degraded and produce a scale of 1 on P10. Meanwhile, moderate greenness is associated with P2 and P9. This is because the RME value (35–42%) is associated with P2, and the absence of catalysts used is associated with P9. Meanwhile, in P11, greenness is poor. This is because there is no real-time analysis in the measurement. The assessment of the green chemistry principles was based on qualitative evaluation.

4. Conclusion

The optimal formula for the best tensile strength was obtained with chitosan (1.5 g), cassava starch (0.75 g), glycerol (0.5 mL), and essential oil (0.5 mL). The main components of AEO were trans-anethole, linalool, and α-pinene. The major components of the CEO were cinnamaldehyde, cinnamyl ester, and eugenol. Meanwhile, for LEO, there are limonene, γ-terpinene, and β-pinene.

The thickness of all films is < 0.25 mm. The essential oils affect moisture content, swelling, and water solubility, which differ among the EOs. The addition of EOs smooths the morphology of the films. Color and optical properties were also altered by the addition of EOs, which make the film less transparent and have a higher opacity value. The addition of EOs decreased the EAB and increased the YM.

The EOs also influence the functional group of edible films. All the edible films were fully degraded on the 3rd day of the biodegradation test. The qualitative evaluation of green metrics was fulfilled 9 of the 12 green chemistry principles. The E factor was close to zero, and the mass intensity was about 2–3. The RME was in the range of 30–50%, while the water intensity was about 68–83. Based on the holistic green star, the excellent greenness of edible film was obtained against the principles of P1, P3, P4, P5, P6, P7, P8, P10, and P12.

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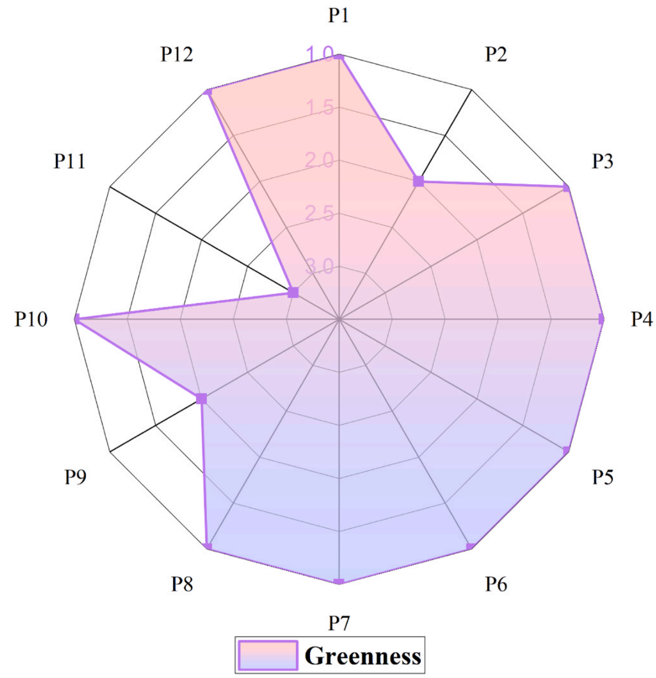

Fig. 5. Greenstar of edible film.

CRediT authorship contribution statement

Sabella Vegasty: Writing – original draft, Visualization, Software, Methodology, Conceptualization. **Maulidan Firdaus:** Writing – review & editing, Validation, Supervision, Software. **Triana Kusumaningsih:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. **Siti Hajar Othman:** Validation, Supervision, Funding acquisition.

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.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.nxmate.2026.102651.

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