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Development, characterization and applications of chitosan-based biodegradable films enriched with essential oils for active food packaging

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ABSTRACT

The present study was undertaken to develop and characterize chitosan-based biodegradable films incorporated with essential oils for active food packaging applications. Chitosan films were prepared using 1.0 and 1.5 per cent chitosan solutions incorporated with different concentrations (0.1, 0.2 and 0.3%) of lemongrass, tea tree, thyme and basil essential oils. The prepared films were evaluated for physicochemical properties, barrier properties, oxygen transmission rate and biodegradability. Film thickness ranged from 0.92 to 0.98 mm and increased slightly with increasing essential oil concentration. Incorporation of essential oils reduced swelling index, film solubility and water vapour permeability while improving water resistance due to enhanced hydrophobic interactions within the film matrix. Among the treatments, films incorporated with lemongrass oil showed superior water resistance, whereas, tea tree oil-incorporated films exhibited the lowest oxygen transmission rate among the EO-based films. Water vapour permeability was lowest in films containing 0.3 per cent tea tree oil and lemongrass oil. All films showed noticeable biodegradation within 30 days under soil burial conditions, confirming their eco-friendly and biodegradable nature. The results demonstrated that incorporation of essential oils improved the functional and barrier properties of chitosan films and enhanced their suitability as active packaging materials for food preservation and shelf life extension.

Keywords: Active food packaging; Biodegradable films; Chitosan; Essential oils; Oxygen transmission rate; Water vapour permeability; Biodegradability; Food preservation

INTRODUCTION

The demand for sustainable food packaging solutions has increased significantly due to rising environmental concerns and growing consumer preference for eco-friendly products (Hussain et al 2024). Chitosan derived from chitin is regarded as a highly biocompatible biopolymer because of its biodegradability bioadhesive properties and bioactivity which enable its wide range of applications (Jimenez-Gomez and Cecilia 2020). However its high hydrophilicity limits its application in food packaging. Essential oils (EOs) are incorporated into packaging materials to inhibit microbial spoilage and extend the shelf life of products due to their hydrophobic nature and the presence of bioactive compounds such as terpenes and phenolics. Some of the most commonly used EOs in packaging systems are extracted from jasmine, rosemary, peppermint, cinnamon, oregano, thyme, cumin, eucalyptus, rosewood, clove, tea tree, palmarosa, geranium, lavender, lemongrass, mandarin, bergamot and lemon (Carpena et al 2021).

This study aims to develop and characterize chitosan-based films incorporated with EOs and to evaluate their physicochemical antimicrobial and application potential in food preservation.

MATERIAL and METHODS

Film preparation

Chitosan solutions (1% and 1.5%) were prepared using 1 per cent acetic acid. EOs (lemongrass oil: LGO, tea tree oil: TTO, thyme oil: TO, basil oil: BO) were added at concentrations of 0.1, 0.2 and 0.3 per cent. Tween 80 (0.05%) was used as emulsifier and glycerol (0.1%) as plasticizer. Films were cast and dried for 24-48 hours. Plate 1 shows chitosan films with and without incorporation of EOs.

Physical property analysis

Film thickness: The thickness of the sample was determined using a screw gauge equipment. Measurements were repeated in 5 different regions of each sample and then an average value was calculated.

Swelling index: Films were weighed and subsequently immersed in water for 10 minutes (Anon 2005a). The mass of the swollen films was measured after the surface water was removed with filter paper. The swelling index (SI) of the film was calculated.

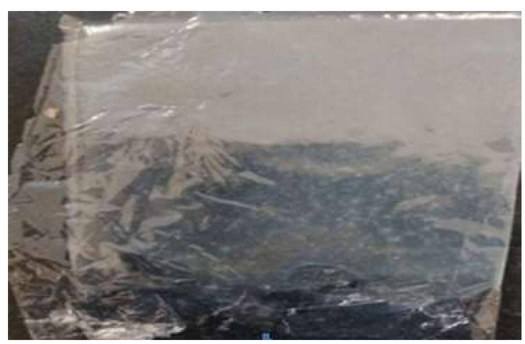
Water resistance: Water resistance was determined gravimetrically by immersing weighed pieces of chitosan film in 50 ml of distilled water by a constant using magnetic stirrer for 15 min at room temperature. Subsequently, the solutions were filtered at room temperature using Whatmann Number 1 filter paper. The chitosan films and the wet filters were dried at 110°C until constant weight. Water resistance was calculated.

Solubility: Solubility was assessed via immersion and drying. A modified method (Anon 2005a) was used to measure film solubility. Film was weighed at 50 mg and and dried at 110°C in a hot air oven for 24 h and then weighed for initial dry weight. The films were placed in glass beaker with 50 ml of distilled water and shaken gently by using centrifuge at 25°C for 24 h. The solution was filtered through Whatman Number 1 filter paper to recover the remaining undissolved films. The remaining pieces of film after immersion were dried at 110°C to constant weight (final dry weight). Solubility was calculated in water.

Barrier properties

Water vapour permeability (WVP): Water vapour permeability was determined by the method described by Anon (2005b). Fifty ml of water was measured and taken in 100 ml beakers. Each beaker was covered

Chitosan film



Chitosan + Basil oil film

Chitosan + Lemon grass oil film

Chitosan + Thyme oil film

Plate 1. Chitosan films with and without incorporation of essential oils

with different chitosan (CH)- and EO-based packaging materials along with the control high-density polyethylene (HDPE). The beakers covered with different packaging materials were kept at room temperature for 24 h and the loss in water volume was measured to determine the water vapour permeability of the packaging materials.

Water vapour permeability (WVP) is an important parameter commonly considered in food packaging as it involves sorption diffusion and adsorption processes. WVP should be as low as possible because it retards moisture transfer between the food and the surrounding environment.

Oxygen transmission rate (OTR): The OTR is the quantity of oxygen gas passing through a unit area of the film surface per unit time under specified oxygen partial pressure temperature and relative humidity conditions. The transfer of oxygen from the environment to food greatly affects food quality and shelf life. Oxygen causes food deterioration through lipid and vitamin oxidation leading to changes in sensory and nutritional quality. Therefore, OTR is an important parameter as oxygen influences the rates of oxidation and respiration. OTR through chitosan films was determined using a volumetric permeability cell (Custom Scientific Instruments, New Jersey) according to the ASTM D-1434 procedure. Testing was carried out at 27 ± 1°C and 65 per cent relative humidity. Films of size 10 cm × 5 cm were used for the determination of oxygen transmission rate following the methodology of Chinga-Carrasco et al (2012). The mean ± SE values of oxygen transmission rate ($g/mPa/s/m^2$) were highest in chitosan film followed by films blended with EOs, whereas, the lowest OTR value was recorded in polyvinyl alcohol (PVA) film.

Biodegradability test: The biodegradability of antimicrobial chitosan-based films was studied as per the procedure suggested by Li et al (2019). Biodegradable films were buried in soil for 30 days. The film samples were cut into rectangular pieces (2 cm × 3 cm) dried at 60°C to constant weight (w_0) and buried at a depth of 5 cm below the soil surface. The soil was watered every alternate day to maintain moisture content.

After 15 and 30 days the buried films were removed, cleaned, washed and dried at 60°C until constant dry weight was obtained. The percentage of film degradation was calculated using the formula given below:

$$Is\ (\%) = (w - wi)/w \times 100$$

where Is = Degradation percentage, w = Initial weight of the ûlms, wi = Final weight of the ûlms

RESULTS and DISCUSSION

Physicochemical properties: The physicochemical properties of chitosan films incorporated with EOs are presented in Table 1. Film thickness increased slightly with increasing EO concentration and ranged from 0.92 to 0.98 mm. All films remained transparent. The swelling index generally decreased with the incorporation of EOs due to the increase in hydrophobicity which helps in maintaining the quality of packaged food products during storage. Among the treatment basil oil at 0.1 per cent concentration with 1.5 per cent chitosan showed the lowest swelling index (100%), whereas, the highest swelling index was observed in lemongrass oil incorporated films. Three-factor ANOVA revealed significant differences among treatments for swelling index. Among the EOs, basil oil was found to be most effective in reducing swelling index followed by tea tree oil, thyme oil and lemongrass oil. Among the two chitosan concentrations 1.5 per cent chitosan generally showed lower swelling index than 1.0 per cent chitosan. It was also observed that chitosan films incorporated with tea tree oil and basil oil showed a decreasing trend in swelling index with increasing essential oil concentration from 0.1 to 0.3 per cent.

EO incorporation significantly improved water resistance and reduced film solubility. At 0.3 per cent EO concentration, the films showed lower solubility values due to enhanced hydrophobic interactions and cross-linking between chitosan and EOs. Among the different EOs, lemongrass oil-incorporated chitosan films exhibited higher water resistance followed by thyme oil, basil oil and tea tree oil. Water resistance increased with increasing EO concentration in both 1.0 and 1.5 per cent chitosan films as compared to chitosan films alone.

Among the treatments, films incorporated with lemongrass oil showed superior water resistance over other EO treatments. The reduction in film solubility and swelling index could be attributed to increased cross-linking interactions between chitosan and hydrophobic EOs. The water solubility values obtained

Table 1. Physicochemical properties of chitosan films incorporated with essential oils

Oil concentration (%)	Tea tree oil (chitosan %)		Lemongrass oil (chitosan %)		Thyme oil (chitosan %)		Basil oil (chitosan %)	
	1.0	1.5	1.0	1.5	1.0	1.5	1.0	1.5
Film thickness (mm)								
0.1	0.94	0.95	0.92	0.96	0.95	0.96	0.96	0.97
0.2	0.94	0.95	0.93	0.97	0.95	0.96	0.96	0.97
0.3	0.95	0.96	0.94	0.97	0.96	0.98	0.97	0.97
Swelling index (%)								
0.1	130	140	160	120	115	120	110	100
0.2	120	158	160	130	130	145	125	115
0.3	114	160	130	140	140	160	135	125
Water resistance (%)								
0.1	18	20	16	28	15	18	16	18
0.2	20	26	26	36	18	28	20	25
0.3	30	30	32	40	24	32	26	34
Film solubility (%)								
0.1	14.6	16.6	12.5	19.0	10.5	11.5	11.0	10.0
0.2	10.8	11.6	10	11.1	9.5	10.0	10.0	9.5
0.3	6.5	8.5	9.5	4.4	8.5	9.5	8.5	8.0
Water vapour permeability (%)								
0.1	1.25	1.25	2.5	1.25	1.15	1.15	1.15	1.25
0.2	1.25	1.25	1.25	0.625	1.0	1.15	0.95	1.0
0.3	0.625	0.625	0.625	0.625	0.95	1.0	0.75	1.0

Property	SED	$CD_{0.05}$	$CD_{0.01}$
Film thickness (mm)	0.758	1.3495	1.8772
Swelling index (%)	0.74365	1.48359	1.97025
Water resistance (%)	0.7765	1.3435	1.9905
Film solubility (%)	0.7656	1.4125	1.9898
Water vapour permeability (%)	0.0074	0.01479	0.01965

for the chitosan film combinations were in agreement with results reported in earlier studies. Water vapour permeability (WVP) decreased markedly with EO incorporation. The lowest WVP value (0.625%) was observed in films containing tea tree oil and lemongrass oil at 0.3 per cent concentration, indicating improved moisture barrier properties desirable for food packaging applications.

Three-factor ANOVA revealed significant differences among treatments. Among the EOs, basil oil was most effective in reducing swelling index, whereas, lemongrass oil showed superior performance in improving water resistance. Tea tree oil-incorporated films recorded the lowest film solubility values at 0.3 per cent EO concentration with 1.5 per cent chitosan. Among the two chitosan concentrations, 1.5 per cent chitosan generally performed better than 1.0 per cent chitosan for most of the physicochemical parameters analysed. Overall, 1.5 per cent chitosan incorporated with 0.3 per cent lemongrass oil performed best for water resistance and WVP, whereas, 0.1 per cent thyme oil incorporated in 1.0 per cent chitosan showed comparatively poorer performance.

Gas transmission property of films: The oxygen transmission rate (OTR) of different film formulations is presented in Table 2. Significant differences were observed among the treatments with respect to oxygen barrier properties. Pure chitosan film (1% chitosan) exhibited the highest OTR value (6.55 ± 0.07 g/mPa/s/m²), indicating comparatively poor resistance to oxygen transmission. Incorporation of polyvinyl alcohol (PVA) and EOs reduced the OTR values of the films, thereby, improving their oxygen barrier properties. Among the EO-incorporated films, the lowest OTR was recorded in films containing tea tree oil (5.25 ± 0.03 g/mPa/s/m²), followed by basil oil (5.47 ± 0.02 g/

Table 2. Gas (oxygen transmission rate) transmission property of films

Treatment	Oxygen transmission rate ($g/mPa/s/m^2$)
1% chitosan	6.55 ± 0.07
1% chitosan + 5% PVA + 0.3% tea tree oil	5.25 ± 0.03
1% chitosan + 5% PVA + 0.3% lemon grass oil	5.96 ± 0.09
1% chitosan + 5% PVA + 0.3% basil oil	5.47 ± 0.02
1% chitosan + 5% PVA + 0.3% thyme oil	5.99 ± 0.03
5% PVA	3.45 ± 0.12

$mPa/s/m^2$), indicating better oxygen barrier efficiency of these films. Films incorporated with lemongrass oil and thyme oil showed relatively higher OTR values of 5.96 ± 0.09 and 5.99 ± 0.03 $g/mPa/s/m^2$ respectively. The lowest overall OTR value was observed in 5 per cent PVA film (3.45 ± 0.12 $g/mPa/s/m^2$), suggesting superior oxygen barrier properties compared to chitosan-based films. The reduction in oxygen transmission in EO-incorporated films may be attributed to the formation of a more compact and hydrophobic film matrix, which restricts oxygen diffusion through the packaging material. Improved oxygen barrier properties are highly desirable in food packaging as they help reduce oxidative deterioration and extend the shelf life of food products.

Biodegradability: The biodegradation behaviour of chitosan, PVA and chitosan-PVA composite films incorporated with EOs is presented in Fig. 1. All the films exhibited noticeable degradation within 30 days of soil burial, indicating their biodegradable nature and suitability for sustainable food packaging applications. Among the different films, pure chitosan films showed the fastest degradation rate, which may be attributed to the natural biodegradable characteristics of chitosan and its susceptibility to microbial action in soil.

In contrast, films containing PVA degraded comparatively slowly due to the stronger intermolecular interactions and comparatively stable polymer network formed in the blends. The incorporation of EOs slightly reduced the degradation rate of the films, possibly because of the hydrophobic nature and antimicrobial properties of EOs, which may inhibit microbial activity responsible for degradation. However, all chitosan-PVA composite films still showed substantial degradation during the experimental period. The results confirm that chitosan-based composite films are environmentally friendly and capable of undergoing biodegradation under natural soil conditions. Such biodegradable packaging materials can help reduce the environmental problems associated with conventional synthetic plastic packaging.

Moalla et al (2021) developed active food packaging films based on chitosan enriched with *Artemisia campestris* essential oil (ACEO). The study evaluated the effect of incorporating *A campestris* on the physical, mechanical, thermal and antioxidant properties of the films. The results revealed that the addition of ACHE and ACEO improved the water resistance of chitosan films. Similarly, Zehra et al (2022) developed chitosan (CH)-based biodegradable films enriched with thyme essential oil (TEO) along with additives such as zinc oxide (ZnO), polyethylene glycol (PEG), nanoclay (NC) and calcium chloride ($CaCl_2$). The incorporation of TEO and these additives significantly reduced water vapour transmission rate and improved tensile strength, while maintaining water solubility and biodegradability of the films. The developed films were reported to be eco-friendly and effective in extending the shelf life of collard greens under refrigerated storage conditions.

Yashaswini and Iyer (2019) prepared chitosan-based films containing turmeric, clove and ginger along with gelatin and glycerol as a plasticizer to obtain transparent and stretchable active packaging films. The thickness of chitosan films incorporated with combined cinnamon and ginger EOs ranged from 0.068 to 0.105 mm. The films exhibited nearly 70 per cent transparency when analysed using a spectrophotometer, which was mainly influenced by film thickness and colour. EOs are widely incorporated into biodegradable films because of their antibacterial (Jing et al 2019) and antioxidant properties that contribute to food preservation (de Oliveira et al 2019). In another study, Al-Harrasi et al (2022) developed and characterized chitosan (CS) and porphyran (POR)-based films loaded with ginger essential oil (GEO).

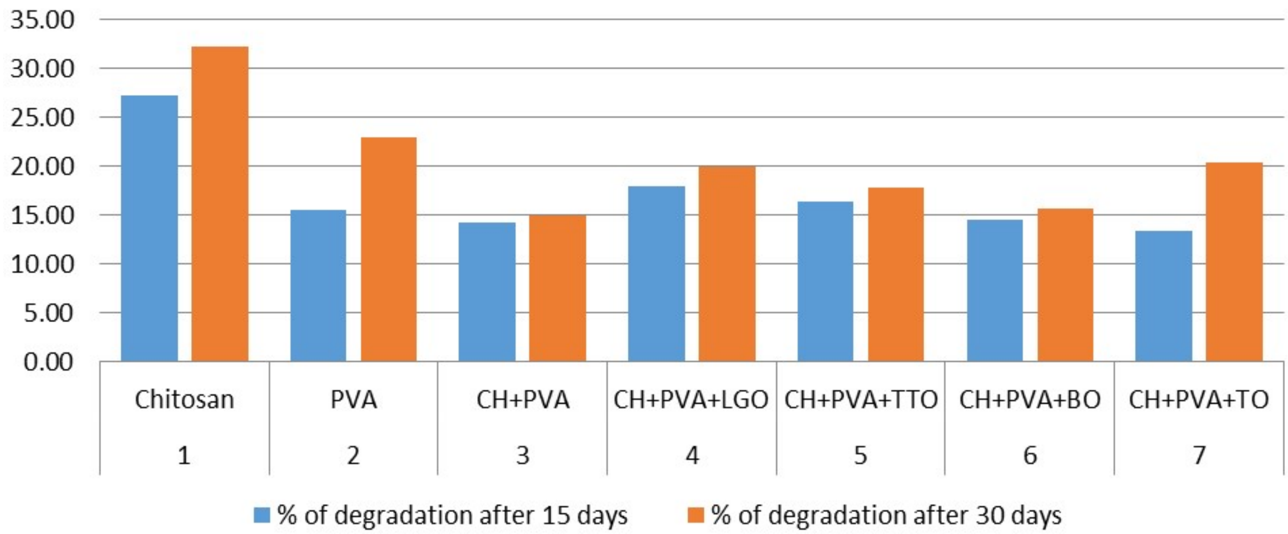


Fig 1. Comparative biodegradation of chitosan, PVA and chitosan-PVA composite films incorporated with essential oils over 15 and 30 days

The incorporation of GEO substantially improved the barrier, thermal, optical and mechanical properties of the films and reduced moisture content, swelling index and thickness. However, the addition of GEO slightly altered the film colour towards yellowness. Shen and Kamdem (2015) developed active biodegradable chitosan films containing 10 to 30 per cent citronella essential oil (CEO) and cedarwood oil (CWO) using the casting and solvent evaporation method. The incorporated oils significantly influenced the physical, mechanical and thermal properties of the films. The addition of CEO and CWO markedly reduced water vapour permeability, with nearly 63 per cent reduction observed in films containing 30 per cent CEO. Such biodegradable films offer promising opportunities for the development of active food packaging materials with enhanced preservation potential.

CONCLUSION

Chitosan-based biodegradable films incorporated with essential oils (EOs) demonstrated improved physicochemical and barrier properties suitable for active food packaging applications. Incorporation of EOs enhanced water resistance and reduced swelling index, film solubility and water vapour permeability due to increased hydrophobic interactions within the film matrix. Among the different treatments, lemongrass oil-incorporated films showed superior water resistance and moisture barrier properties, while tea tree oil-incorporated films exhibited lower oxygen transmission rate among the EO-treated films. The films also showed substantial biodegradation under soil burial conditions within 30 days, confirming their environmentally friendly nature. Overall, incorporation of EOs significantly improved the functional properties of chitosan films and indicated their potential application as sustainable and biodegradable packaging materials for extending the shelf life and quality of food products.

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