

Development of Pectin-Pineapple Juice Films Incorporated with Ginger Essential Oil Nanoemulsion for Food Packaging Application

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ABSTRACT

Edible films loaded with essential oil nanoemulsions dissolve quickly in water and are completely biodegradable and compostable, making them an appropriate and sustainable alternative to single-use food packaging. This study aims to develop pectin-pineapple juice (PPJ) films with varying loadings of ginger essential oil nanoemulsion (GEONE), ranging from 5% to 20%, with a PPJ film without GEONE serving as a control. The effect of incorporating GEONE on the films' physical, mechanical, colour, and thermal properties, including enthalpy and moisture loss, was evaluated. The thickness of the films significantly increased ($p \leq 0.05$) from 91.0 μm to 112.0 μm when the loading of GEONE increased. The PPJ films' moisture content (11.57% to 10.33%) was insignificantly affected ($p > 0.05$) by the presence of GEONE. The water solubility of the PPJ film with 20% GEONE (83.16%) was lower than that of the control PPJ film (93.77%). The control film yielded the highest lightness (L^* value = 84.40) and intermediate yellowness (b^* value = -1.08). As GEONE was incorporated into the PPJ films, the lightness decreased while the b value, yellowness index, and colour differences increased. The tensile strength and Young's modulus values significantly decreased ($p \leq 0.05$) from 15.97 MPa to 9.54 MPa and from 398.92 MPa to 186.91 MPa, respectively, with increasing GEONE loading. It indicates that the GEONE nano-size droplets have plasticising effects, increasing the elongation at break of the PPJ films from 8.71% to 16.49%. Additionally, the differential scanning calorimetric curves demonstrated that films incorporated with

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increasing GEONe had a lower crystallisation enthalpy, improving the thermal stability of the PPJ-GEONe films. These findings revealed the potential of incorporating GEONe into pectin-based films, resulting in acceptable physical properties, flexibility, and thermal stability.

Keywords: Differential scanning calorimetry, ginger essential oil, nanoemulsion, pectin-based film, tensile properties

INTRODUCTION

Single-use plastics, including production, processing, distribution, and consumption, are widely used across the food chain system. This food packaging material is specifically designed for one-time use, without any reuse provisions, before being discarded or recycled (Foodprint, 2020). Malaysia is now ranked eighth among the world's top ten countries for the highest levels of mismanaged plastic waste (Zainal et al., 2023). In response, Malaysia introduced an ambitious roadmap in 2018 with the goal of eliminating single-use plastics by 2030 (Zainal et al., 2023). This initiative seeks to mitigate plastic mismanagement and promotes consumer education on reusable grocery bags, containers, and cutlery. Furthermore, there is a growing demand for food packaging manufacturers to develop biodegradable and compostable plastics as alternatives to single-use plastic food packaging. The biopolymer-based films, made from sustainable raw materials such as proteins, polysaccharides, and lipids, play a crucial role in facilitating this transition. These biopolymers are both environmentally friendly and edible and safe to consume along with packaged foods, offering a sustainable solution to the plastic waste problem.

Polysaccharides used to produce biodegradable and edible plastic films include starch, cellulose, chitosan, chitin, pullulan, and pectin. Industrially processed pectin obtained from apple pomace and citrus peels is also often used for this application (Dranca et al., 2021; Kute et al., 2020). Furthermore, sugar beet pulp and sunflower seed heads are sources with notable physicochemical qualities, characterised by their abundant pectin content and biomass availability (Adetunji et al., 2017). Pectin, a complex polysaccharide, comprises α -1,4-linked d-galacturonic acid units. The methylation of the carbonyl groups at the carbon-6 position within the pectin backbone is responsible for its gelling properties. Pectin can be categorised into two groups based on its degree of esterification (DE). High methoxyl pectin (HMP) is characterised by a methylation degree greater than 50%, whereas low methoxyl pectin (LMP) has a methylation degree of less than 50% (Blanco-Pérez et al., 2021; Marić et al., 2018). The DE might also differ based on the source of pectin. Pectin plays an important role in food manufacturing as a thickening agent, stabiliser, and emulsifier. Additionally, it is used to create edible coatings and films (Kumar et al., 2023). Pectin offers considerable potential as a bio-based packaging material due to its water solubility, excellent film-forming capability, and effective barrier against gases and aromatic compounds (Roy et al., 2023). However, films made from pectin are susceptible

to moisture due to the abundance of hydroxyl groups in their structure, leading to high permeability. They also exhibit limited thermal stability and are rigid and fragile. Combining pectin with other raw materials or subjecting it to modifications is a promising approach to overcome these drawbacks.

Different hydrophobic materials have been added to the pectin-based film as a food packaging material to improve its properties (e.g., physical, mechanical, and thermal). Essential oils (EO) are a prominent type of plant extract used in pectin-based film (Nastasi et al., 2022). Ginger essential oil (GEO), formally known as *Zingiber officinale*, is a highly valued aromatic oil derived from the tuber of the ginger plant. GEO is extracted through steam distillation, which captures the concentrated essence of the ginger plant. Due to its benefits, ginger is frequently used in food applications as a condiment, flavouring ingredient, and natural preservative. According to Noori et al. (2018), GEO has remarkable antimicrobial, antifungal, and antioxidant properties, which make it an excellent choice for food packaging applications. Unfortunately, most raw EOs are unstable and prone to oxidation in the presence of light, oxygen, and high temperatures (Fasihi et al., 2023). Furthermore, the hydrophobic characteristics of EOs can result in inadequate compatibility and phase separation when directly integrated into a water-soluble biopolymeric film-forming dispersion (Almasi et al., 2020). Poor compatibility between hydrophobic EO and hydrophilic pectin results in the film's not uniform structure and overall decreases its mechanical properties (Chen et al., 2023).

One potential strategy for addressing the aforementioned issues is to incorporate EO in nanoemulsion form. Several studies, including those conducted by Almasi et al. (2020), Candido et al. (2022), Jahromi et al. (2022), Norcino et al. (2020), and Shaaban et al. (2016) have investigated the potential of utilising essential oils such as pracaxi, marjoram, cinnamon, copaiba, and clove in the formulation of pectin-based films. The pharmaceutical, cosmetic, and food industries use the lipid-based colloidal delivery system to encapsulate essential oils such as emulsions. Encapsulating essential oils by emulsification is a feasible method to form a colloidal delivery system to stabilise the lipophilic functional materials (Fernandes et al., 2016). The nanoemulsion, featuring droplets smaller than 200 nm in diameter, represents a dispersion of an immiscible liquid in which lipid-based hydrophobic materials are dispersed within the water phase. This nanoemulsion is typically a stabilised non-ionic emulsifier, which is produced through a combination of high-speed homogenisation and ultrasonic crushing techniques. Nanoemulsions have several advantages over coarse emulsions, which have larger droplets measured in micrometres, including high stability and a larger surface area. The small droplets can better sustain the original structural arrangement of the film microstructure, reducing negative effects on film performance (Zhang et al., 2022). In addition, liquid lipid carriers such as edible oil (virgin coconut oil, corn, sesame, sweet almond, and black seed oil) have been widely

used in the formulation of lipid-based nanoemulsion to overcome micelles different storage conditions (Bashiri et al., 2020; Rosli et al., 2022).

Edible films have been incorporated with fruit juices such as grape, jambolan, pear, and purees of mango and pineapple (Chambi et al., 2020; Pimiento et al., 2023; Susmitha et al., 2021; Yıldırım-Yalçın et al., 2021). These fruit juices comprise carbohydrates (such as fructose, glucose, sorbitol, and sucrose), organic acids, amino acids, phenolic compounds, vitamins, and minerals (Salehi, 2020). Incorporating fruit-based products into edible films can improve their appearance and physical and mechanical properties and enhance their nutritional value. Simple sugars in fruit juice are known to have a plasticising effect on edible films, impacting their tensile strength, elongation at break, and water vapour permeability (Azeredo et al., 2016). Nonetheless, the amount of fruit juice added to edible films needs to be optimised to achieve the desired quality (Yıldırım-Yalçın et al., 2021).

Pineapple, or *Ananas comosus*, is a well-liked tropical fruit distinguished by its distinct aroma and sweet flavour. Malaysia has expanded its pineapple production cultivation area in recent years. In 2022, 537,231 tonnes of pineapple were produced in Malaysia (DOA, 2022). Post-harvest processing operations are crucial for preserving the quality, safety, and shelf life of processed pineapple products because pineapple is highly perishable (Abraham et al., 2023). Pineapple has attracted significant interest for its potential as a functional food and for various products made from pineapple (Ali et al., 2020). Commonly seen pineapple-based products in the market include drinks, jam, dehydrated slices, canned pineapple slices, and beverages. Both unsweetened and sweetened pineapple juice drinks are widely consumed in Malaysia. The amount of vitamins, minerals, and sugars in pineapple juice varies based on the variety and ripeness of the fruit. It has an especially high concentration of 9.2 to 93.8 mg of vitamin C per 100 mL (Kabasakalis, 2000). Significant levels of minerals such as potassium, magnesium, phosphorus, iron, and manganese are also present in pineapple juice. According to Statista (2024), Malaysia's pineapple juice sales revenue is predicted to increase by 1.52% yearly between 2024 and 2028. Thus, adding pineapple juice to biopolymers could offer new opportunities to diversify pineapple-based products beyond just drinking it as a beverage.

This study aimed to develop pectin-pineapple juice (PPJ) films containing ginger essential oil nanoemulsion (GEONe) with enhanced flexibility and thermal stability. The ginger essential oil was selected because it is commercially available, and its nanoemulsion form is a promising strategy to enhance the targeted properties effectively. Virgin coconut oil, which contains the health benefits of medium-chain triglycerides (MCT), was selected as a carrier oil in the nanoemulsion formulation (Ghani, Channip et al., 2018). MCT oils have lower interfacial tension and viscosity than long-chain triglyceride oils (e.g., soybean oil, safflower oil), resulting in the formation of smaller droplet sizes during the emulsification

process (Sampaio et al., 2022; Walker et al., 2017). The objectives of this study were: (1) to evaluate the effects of adding virgin coconut oil to the GEONE formulation to achieve a stable and homogenous nanoemulsion with nano droplet size and (2) to evaluate the effects of incorporating varying GEONE loadings (5% to 20%) into the PPJ films on the thickness, moisture content, water solubility, visual appearance, colour, tensile strength (TS), Young's modulus (YM), elongation at break (EAB), and thermal stability of the film in comparison to control PPJ film (absence of GEONE).

MATERIALS AND METHODS

Materials

The highly concentrated ginger essential oil (GEO), which is 100% pure, was purchased from Now Foods, Illinois, United States of America. Extra virgin coconut oil (VCO) was obtained from Country Farms Sdn. Bhd., Selangor, Malaysia. Polysorbate 80 (Tween 80), an emulsifier, was purchased from EvaChem, Selangor, Malaysia. Cold-pressed pineapple juice ($16.0 \pm 0.1^\circ$ Brix, $\text{pH } 4.1 \pm 0.1$) of the MD2 variety was purchased from Cafe Nanas Montel in Selangor, Malaysia, and included no additional sugar. Rapid set high methoxyl (HM) pectin powder (Product code: 1121) with a degree of esterification (DE) of 75%, extracted from citrus fruit peels, was obtained from Modernist Pantry, Eliot, ME 03903, United States of America. Edible beeswax was acquired from American Soy Organics, United States of America, while glyceryl monostearate with 99% purity was obtained from Eastchem Inc., United States of America. In this research, pectin was applied as the film matrix, and glycerin was employed as a plasticiser. Glyceryl monostearate served as the emulsifier, while beeswax was added to the mixture as a hydrophobic agent to improve the film's moisture resistance. Distilled water was produced using a water distiller (Favorit W4L Water Stills) from PLT Scientific Sdn. Bhd., Malaysia.

Preparation of Ginger Essential Oil Nanoemulsion (GEONE)

In a beaker, 5 mL of ginger essential oil (GEO) was added to 95 mL of distilled water and stirred using a high-speed stirrer (HS-30D, WiseStir[®]HS-D, DAIHAN Scientific, Kangwon-do, South Korea) at 700 rpm. Subsequently, two levels of virgin coconut oil (VCO) (1 mL and 3 mL) were added to the mixture, followed by 1.5 mL of polysorbate 80. Next, the mixture was stirred at 1400 rpm for 10 min to obtain a coarse emulsion. The coarse emulsion was subjected to ultrasonic emulsification using a Branson 450 ultrasonic emulsifier (Branson Ultrasonics Corp., United States of America) at 50% amplitude for 9 min to achieve a nanometer-sized droplet emulsion. Then, the ginger essential oil nanoemulsion (GEONE) was transferred to an amber glass bottle and stored at room temperature ($25 \pm 2^\circ\text{C}$) for future use.

Characterisation of Ginger Essential Oil Nanoemulsion (GEONe)

Droplet Size, Size Distribution, and Stability Measurement

The GEONe nano-size droplet diameter was indicated by the z-average value and polydispersity index (PDI) values yielded from the diameter distribution curve. These values were obtained using dynamic light scattering with a Zetasizer Nano ZS (Malvern Instruments Inc., Worcestershire, U.K.). The first measurements were taken when the emulsification reached 24 hours, followed by a second measurement on day 7 to assess the stability of the nanoemulsion during storage. The mean diameter on each day was measured twice, and the average mean diameter was reported.

Preparation of Pectin-pineapple Juice-GEONe Films

Four grams of pectin were added to 100 mL of $70 \pm 2^\circ\text{C}$ distilled water. Next, 20% (w/w of pectin) glycerin, 1% (w/w of pectin) beeswax, and 0.2% (w/w of pectin) of glyceryl monostearate were added to the pectin solution. Then, the pectin-based solution's temperature was raised to $90 \pm 2^\circ\text{C}$ and was continuously stirred at 100 rpm using a hot plate stirrer (MSH-20D, Daihan Scientific Co., Ltd., Wonju, South Korea) for 30 min. Once all the materials were dissolved completely, the pectin-based solution was cooled down to below 10°C in a chiller before subsequently adding 4% (v/v of distilled water) of pineapple juice (PJ) to the pectin-based solution and continuously mixing with a magnetic stirrer at 100 rpm for another 10 min until thoroughly mixed. Next, the ginger essential oil nanoemulsion (GEONe) was added at different loadings of 5%, 10%, 15%, and 20% (v/v of distilled water) and mixed under the same settings until GEONe was dispersed evenly. The concentrations were chosen following a research work reported by Ghoshal (2022). Ultrasonic degassing was performed on the film-forming solution to eliminate any entrapped air bubbles using an ultrasonic probe (Branson 450, Branson Ultrasonics Corp., United States) at 50% amplitude for 10 minutes with a pulse mode (59 sec. 'ON', 10 sec. 'OFF'). The pectin-based film without GEONe acts as a control film. Approximately 22 mL of each film solution was cast onto glass petri dishes with a 140 mm diameter and then dried at room temperature ($25 \pm 2^\circ\text{C}$) for 24 hours. Finally, the dried films were peeled off and conditioned in a humidity-controlled cabinet (DB-90s, VAMOS, Selangor, Malaysia) at 50% relative humidity for 48 hours before testing.

Characterisation of Pectin-pineapple Juice-GEONe Films

Film Thickness Measurement

The film's thickness was measured using a digital micrometre (Mitutoyo Corp. Kawasaki, Japan). The mean value was obtained by measuring three random locations on each film.

Moisture Content Measurement

The MX-50 Moisture Analyzer (Mettler Toledo, Greifensee, Switzerland) was employed to determine the film's moisture content. Prior to measurement, 2 g of the film was heated to 105°C until a constant weight was reached, following the preparation procedure as in AOAC International (1995). Measurement was taken twice from a separate film, and the average moisture content was recorded.

Water Solubility Measurement

The film's dissolvability in water was assessed through a water solubility test following a method procedure by Tulamandi et al. (2016), with the soaking condition underwent a minor modification.. The PPJ-GEONE and PJJ films were cut into square samples measuring 40 mm × 40 mm each. These films were oven-dried (model 30-1060, Memmert GmbH + Co. KG., Schwabach, Germany) at 105°C for 24 hours. The weight of the dried films was measured, representing the initial dry weight of the film (W_0). Subsequently, the dried films were soaked in 50 mL of distilled water and shaken under constant agitation at 100 rpm for 24 hours at $26 \pm 2^\circ\text{C}$ using a water bath shaker (BS-21, Alirantek (M) Sdn. Bhd., Selangor, Malaysia). Next, filter papers (Whatman No. 541) were pre-dried at 105°C for 24 hours before use. Then, the film-dissolved solution was filtered through the filter paper, and the residue of water-insoluble matter was pre-dried at 105°C for 24 hours to remove moisture until a constant weight (W_1) was obtained. All tests were performed in triplicate to obtain the mean values. Finally, the percentage of the film's water solubility was calculated using the following formula:

$$\text{Water solubility (\%)} = \frac{W_0 - W_1}{W_0} \times 100 \quad [1]$$

Colour Measurement

The colour parameters of the film samples L^* , a^* , and b^* were measured using a portable calorimeter (FRU WR10, Shenzhen Wave Optoelectronics Technology Co., Ltd., China). The colour parameters were based on the CIELAB colour scale for $L^* = 0$ (darkness) to $L^* = 100$ (lightness), $-a^*$ (greenness) to $+a^*$ (redness), and $-b^*$ (blueness) to $+b^*$ (yellowness). The colour parameter readings were taken at five random locations on each film, and the mean of the five readings was recorded. Next, the L^* , a^* , and b^* values were used to calculate the yellowness index (YI) and total colour difference (ΔE) of each film using Equations 2 and 3 (Ranjbaryan et al., 2019):

$$YI = \frac{142.86 \times b^*}{L^*} \quad [2]$$

$$\Delta E = \sqrt{(L^* - L_o^*)^2 + (a^* - a_o^*)^2 + (b^* - b_o^*)^2} \quad [3]$$

Tensile Test

The tensile main properties, including tensile strength (TS), Young's modulus (YM), and elongation at break (EAB), were obtained using a TA-XT_{plus} Texture Analyzer (Stable Micro Systems, Surrey, UK) following the ASTM standard method D882-18 (ASTM D882-18, 2018). The film samples were cut into 100 × 15 mm² rectangular strip dimensions. These film strips were securely mounted on the instrument's fixture grips with 50 mm between them. The tension of the film strip was carried out at a test speed of 0.5 mm/sec until a breaking point was reached. The tensile force (N) and the elongation distance (mm) were acquired to plot a graph of force against the deformation curve for data analysis. The mean value was taken from at least three replicates of separate films. The mean values for TS, YM, and EAB of the films were calculated using the following equations:

$$\text{Tensile Strength (MPa)} = \frac{F}{A} \quad [4]$$

where, F = The ultimate force at the failure point, N; A = Cross-sectional area of the film, mm²

$$\text{Young's modulus (MPa)} = \frac{\sigma}{\varepsilon} \quad [5]$$

where, σ = Tensile stress, MPa; ε = Tensile strain

$$\text{Elongation at break (\%)} = \frac{L_f - L_o}{L_o} \times 100 \quad [6]$$

where, L_f = Final length of the film at specimen break; L_o = Initial length of the film

Differential Scanning Calorimetry Analysis (DSC)

The film's thermal properties, namely enthalpy (ΔH) and moisture loss (%), were analysed from the endothermic peak areas observed in the DSC curves acquired using a differential scanning calorimeter (DSC 3, Mettler Toledo, Greinfensee, Switzerland). Approximately 10 mg of each film sample was placed and hermetically sealed in a standard aluminium pan and subjected to a temperature increase from -25°C to 200°C at a constant heating rate of 20°C/min. The chamber was purged with nitrogen gas flowing at 50 cm³/min. The acquired DSC curves were analysed using STARE Excellence software (Mettler Toledo, Greinfensee, Switzerland).

Statistical Analysis

The effect of VCO levels (0, 1, and 3 mL) and seven days of storage on droplet size and size distribution were assessed using a two-way analysis of variance (ANOVA). A one-way ANOVA was performed to evaluate the effect of five levels of GEONe addition (0%, 5%, 10%, 15%, and 20% GEONe) on PPJ films. The results were presented as mean values \pm standard deviation. Tukey's multiple comparison test with a p-value of 0.05 was employed to determine significant differences between mean values. The experimental data's mean and standard deviation calculations were performed using Microsoft Excel 2021 (Microsoft Corporation, Albuquerque, NM, USA). The error bar included in the graph represents the one standard deviation of the mean values.

RESULTS AND DISCUSSION

Droplet Size, Size Distribution, and Stability of Ginger Essential Oil Nanoemulsion (GEONe)

The measurement of ginger essential oil nanoemulsion (GEONe) droplet size (z-average value) and size distribution (polydispersity index) is important to evaluate its stability and impact on the pectin-based films' properties. Figures 1(a) and 1(b) depict the mean droplet size and polydispersity index (PDI) values for GEONe with and without virgin coconut oil (VCO). On day one, GEONe droplets without VCO measured 47.05 ± 2.62 nm (Figure 1(a)). Noori et al. (2018) reported a droplet size of 57.4 ± 2.7 nm for a freshly produced ginger essential oil (GEO) nanoemulsion using polysorbate 80 and water without a carrier oil. The nanoemulsion was prepared via ultrasonic emulsification with different setting parameters than the present study, such as power at 200 W and time for 5 min. Nevertheless, the present study has investigated the influence of VCO as the carrier oil on the droplet size and stability of the nanoemulsion. For this reason, during the oil phase preparation, VCO was mixed with GEO (lipophilic bioactive agent). VCO, which is rich in medium-chain triglycerides (MCT), has been used as a carrier oil in emulsions due to its safety and availability compared to long-chain triglycerides (e.g., corn oil) and short-chain triglycerides (e.g., tributyrin) (Jiang & Charcosset, 2022; Man & Marina, 2006; Nguyen & Diep, 2022). Walker et al. (2017) and Sampaio et al. (2022) reported that MCT oils exhibit lower interfacial tension and viscosity than long-chain triglyceride oils, forming smaller droplet sizes during emulsification. Adding 3 mL of VCO reduced droplet diameter significantly ($p \leq 0.05$) from 47.05 ± 2.62 nm (GEONe without VCO) to 24.6 ± 0.78 nm. Previous research by Walker et al. (2017) and Sampaio et al. (2022) found that combining low-viscosity carrier oils with high-viscosity bioactive oils reduced droplet size and promoted stable nanoemulsion.

GEONe without VCO showed a significant increase in droplet size over 7 days (from 47.05 ± 2.62 nm to 62.5 ± 0.21 nm (Figure 1(a))), while GEONe with VCO did not show

significant changes. These results indicate that the emulsions containing VCO as a carrier oil are more stable in short-term storage (7 days) than GEONE without VCO. Regarding the polydispersity index (PDI), both the GEONE without VCO and GEONE with 1 mL VCO yielded values above 0.4 (Figure 1(b)). However, the GEONE formulation with 3 mL VCO has a lower PDI value of 0.21, indicating a good droplet size distribution in the emulsion than the former formulations. Regardless of the amount of VCO added in the formulation (1 mL and 3 mL), the GEONE with VCO demonstrated no significant change in the 7-day storage, indicating short-term stability. The GEONE formulation with 3 mL VCO was chosen to fabricate films with small droplet sizes and a stable emulsion. This GEONE formulation was then incorporated into pectin-pineapple juice (PPJ) films at 5%, 10%, 15%, and 20% loadings.

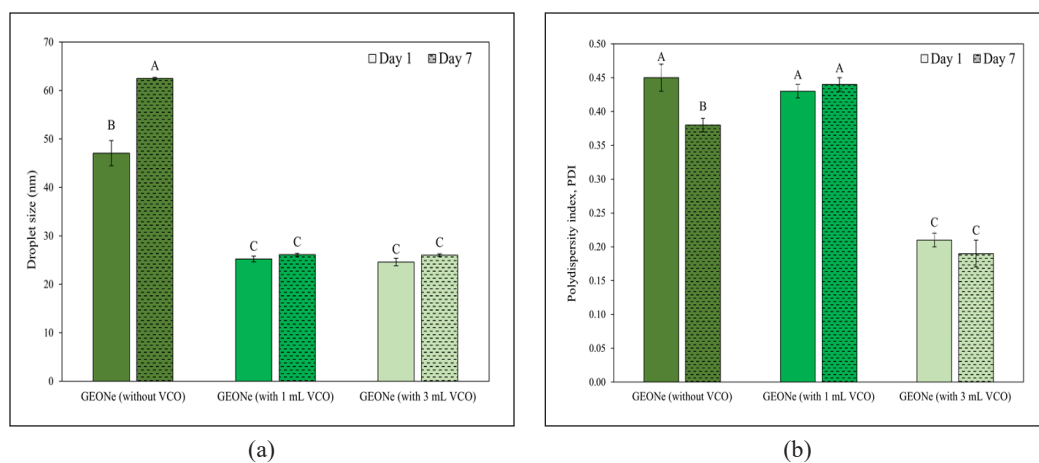


Figure 1. Droplet size (a) and polydispersity index (PDI) (b) of GEONE with varying levels of VCO, measured on Day 1 and Day 7. Different letters (A, B, and C) indicate significant variations in mean values when considering the interaction of both factors ($p \leq 0.05$)

Characterisation of Pectin-pineapple Juice-GEONE Films

Thickness, Moisture Content, and Water Solubility

Table 1 presents the thickness, moisture content, and water solubility of the pectin-pineapple juice-GEONE films. The thickness of the pectin-pineapple juice (PPJ) film was 91.0 ± 6.9 μm , but the addition of GEONE significantly ($p \leq 0.05$) increased the film's thickness. A study by Li et al. (2021) reported that low thickness formed a compact and ordered network. Nevertheless, an increase in films' thickness from 102.0 ± 0.9 μm to 112.0 ± 2.3 μm was recorded as the GEONE loading increased from 5% to 20%. All of the films have a thickness that is less than the 250 μm maximum standard thickness for edible film set by the Japanese Industrial Standard (1975). A similar increasing trend (55 ± 5 μm to 121 ± 1 μm) was also reported by Jahromi et al. (2022) when emulsified clove essential oil was

added to pectin-based films. Adding essential oil might weaken the interaction between biopolymer molecules and develop a discontinuous and non-compact matrix, increasing the thickness (Li et al., 2022; Wu et al., 2017). When equal volumes of the film-forming solution are applied for casting, the differences in thickness can be attributed to variations in the concentration of total solids present in the film-forming suspensions (Ghani, Barzegar et al., 2018; Kong et al., 2022). Furthermore, the nanoemulsion comprises a lipid phase, water, and surfactant; thus, they can contribute to a thicker film.

Table 1 displays the films' moisture content. The moisture content of PJJ films without GEONe was approximately 11.57%, while the moisture content with various GEONe loadings demonstrated insignificant change ($p > 0.05$). This result can be explained by a considerably low range of GEONe added to the films' solution in the present study.

In addition, Table 1 presents the film's water solubility. The PJJ film was highly soluble in water ($93.77\% \pm 1.07$). However, adding 20% GEONe to the film significantly reduces water solubility ($p \leq 0.05$) to $83.16\% \pm 5.28$, indicating a high water solubility. GEO's hydrophobic nature is expected to cause a decrease in water solubility. According to Nisar et al. (2018), cross-linking pectin with essential oils reduces the attraction between the polysaccharide polymer and water molecules. Consequently, the film has a low moisture content and is less water soluble.

Table 1

Thickness, moisture content, and water solubility of PPJ-GEONe films

Film samples	Thickness (μm)	Moisture content (%)	Solubility in water (%)
PPJ-0% GEONe (control)	91.0 ± 6.9^c	11.57 ± 1.13^a	93.77 ± 1.07^a
PPJ-5% GEONe	102.0 ± 0.9^b	11.49 ± 0.40^a	89.77 ± 0.66^{ab}
PPJ-10% GEONe	108.0 ± 0.6^{ab}	11.25 ± 2.67^a	86.27 ± 0.33^{ab}
PPJ-15% GEONe	108.0 ± 0.4^{ab}	10.98 ± 0.62^a	86.20 ± 0.49^{ab}
PPJ-20% GEONe	112.0 ± 2.3^a	10.33 ± 0.69^a	83.16 ± 5.28^b

¹The data is shown as the mean \pm standard deviation. Distinct letters within the same column signify statistically significant differences ($p < 0.05$)

Physical Appearance and Colour

Table 2 exhibits the photographic images of the films. All films were visually transparent and had a smooth surface. They were sufficiently flexible when peeling off by hand from petri dishes, indicating good strength and flexibility. Comparing the PPJ films containing various GEONe loadings to the PJJ films without GEONe, the former films exhibited better peel ability than the latter films. These findings are in line with a study by Jahromi et al. (2022), where adding clove essential oil emulsion to high methoxyl pectin-based films had improved peel ability and produced a smoother surface appearance than that of films without the clove essential oil emulsion. The films from the current study have a

mechanical peel ability comparable to the previous study, which makes them suitable for tensile testing (Figure 2(a) to (c)).

Table 2
Photographic images of PPJ-GEONE films

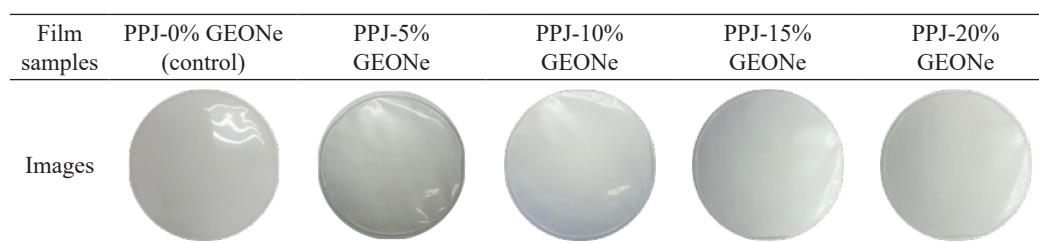


Table 3 presents the data acquired on the PPJ-GEONE films' surface colour parameters, including lightness (L^*), redness-green (a^* , blue-yellow b^*), total colour difference (ΔE), and yellowness index (YI). Adding different GEONE loadings to the films had a significant impact ($p \leq 0.05$) on all colour properties. The control film yields the highest lightness (84.40 ± 0.39) and intermediate yellowness (-1.08 ± 0.42) than the PPJ-GEONE films that were added with 15% and 20% GEONE nanoemulsions. The lightness and yellowness values of the PPJ-20% GEONE films were the lowest, with 81.14 ± 0.76 and 2.78 ± 0.97 , respectively. The yellowness in the films was anticipated due to the inherent yellow colour in the pineapple juice. However, PPJ films with 0% to 10% GEONE demonstrated no significant difference ($p > 0.05$) in their yellowness. Adding 15% and 20% GEONE in the films significantly increased the yellowness. Thus, it is speculated that sufficient loading of ginger essential oil in the films influenced the increased yellowness. According to Atarés et al. (2010), variations in colour parameters were influenced by the natural amber-yellow colour of essential oils or lipids. Nisar et al. (2018) reported a similar change in colour parameters in citrus pectin films added with clove bud essential oil.

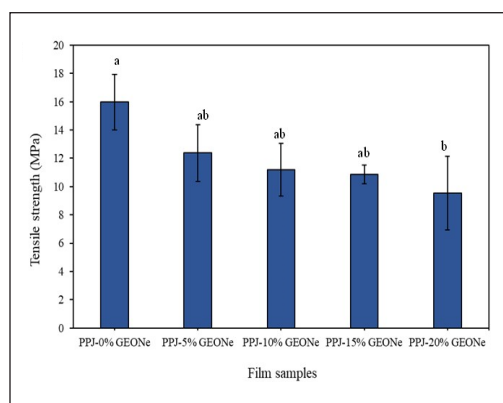
Table 3
Surface colour parameters (L^* , a^* , b^*), total colour difference (ΔE), and yellowness index (YI) values of PPJ-GEONE films

Film samples	L^*	a^*	b^*	YI	ΔE
PPJ-0% GEONE (control)	84.40 ± 0.39^a	1.25 ± 0.04^a	-1.08 ± 0.42^b	-1.82 ± 0.63^b	-
PPJ-5% GEONE	83.07 ± 0.40^b	1.06 ± 0.07^b	-0.77 ± 0.46^b	-1.33 ± 0.80^b	1.43 ± 0.48^b
PPJ-10% GEONE	82.90 ± 0.71^b	1.03 ± 0.07^b	-0.64 ± 0.78^b	-1.09 ± 1.15^b	1.67 ± 0.78^b
PPJ-15% GEONE	81.75 ± 0.37^c	0.73 ± 0.03^c	1.29 ± 0.54^a	2.26 ± 0.96^a	3.61 ± 0.61^a
PPJ-20% GEONE	81.14 ± 0.76^c	0.69 ± 0.05^c	1.58 ± 0.54^a	2.78 ± 0.97^a	4.22 ± 0.87^a

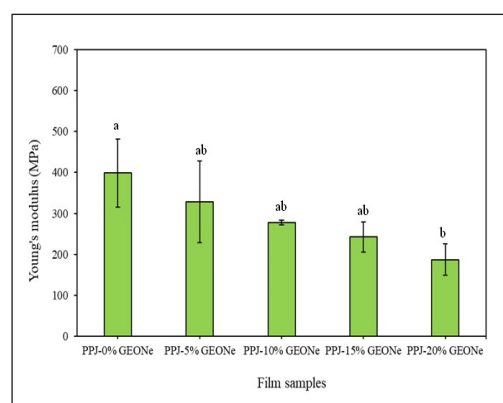
¹The data is shown as the mean \pm standard deviation. Distinct letters within the same column signify statistically significant differences ($p < 0.05$)

Tensile Properties

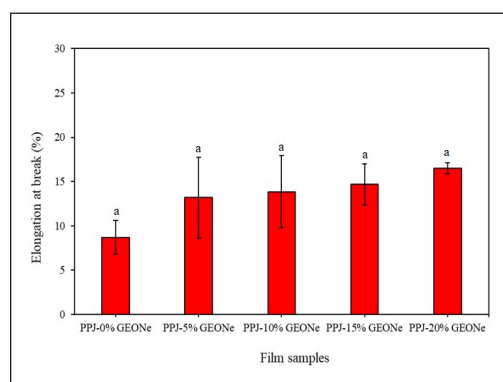
Figure 2 shows the tensile strength (TS), Young's modulus (YM), and elongation at break (EAB) of the PPJ-GEONE films. These are critical mechanical properties to assess the film's strength and flexibility in response to the applied tension force (Shen et al., 2021). The control film (PPJ-0%GEONE) yields TS, YM, and EAB values of 15.98 ± 1.96 MPa, 398.92 ± 83.35 MPa, and $8.71 \pm 1.88\%$, respectively. At the 20% GEONE loading in the PPJ film, the TS and the YM were significantly decreased ($p \leq 0.05$) to 9.54 ± 2.60 MPa (Figure 2 (a)) and 187 ± 38.58 -MPa (Figure 2 (b)), respectively. Conversely, the EAB of the PPJ-20%GEONE films did not change significantly ($p > 0.05$) when compared to the control film. JIS Z 1707 (1975) requires films used for food packaging to have a tensile strength of at least 0.39 MPa. The films produced in this study have tensile strengths that exceed the minimum requirement. However, according to JIS 1975, none of the films' EAB values meet the minimum elongation value requirement of 70%. Almasi et al. (2020) found that the incorporation of marjoram essential oil-loaded nanoemulsion (at 2.5% and 5% w/w of pectin) in the pectin-based films reduced TS from 3.95 ± 0.17 MPa to 1.87 ± 0.45 MPa, and YM from 648 ± 9.43 MPa to 619 ± 4.14 MPa, but had no significant effect on EAB. These findings can be attributed to the incorporation of an essential oil-loaded nanoemulsion, which reduces elasticity and increases plasticity. The ductility and flexibility of the PPJ-GEONE films are



(a)



(b)



(c)

Figure 2. The tensile strength (a), Young's modulus (b), and elongation at break (c) of PPJ-GEONE films. Note. Distinct letters (a, b, and c) show significant differences between means ($p \leq 0.05$) when comparing different percentages of GEONE addition

unaffected by the addition of GEONe (up to 20%). It should be noted that the TS and YM values reported in the current study are higher than those reported by Almasi et al. (2020), most likely due to the use of a higher loading range of GEONe (up to 20%) in the PPJ films. The GEONe droplets in PPJ films provided a plasticising effect, disrupting the hydrogen bonding in the pectin-based films, leading to reduced rigidity and resistance, while increasing the flexibility and segmental mobility of the polymer (Almasi et al., 2020; Otoni et al., 2016). According to Gahruie et al. (2017), the small droplet size of an emulsion can potentially reinforce the biopolymer network by promoting extensive hydrogen bonding (dipole-dipole interaction) between the polar (particularly hydroxyl) groups present along the polymer backbone and the polar head groups of surfactants located at the interface of dispersed nanodroplets in the film-forming solution. Recent studies have shown that adding essential oil-loaded nanoemulsions can reduce the TS of biopolymer-based films, such as pullulan-gelatin films (Shen et al., 2021), chitosan films (Chen et al., 2016), isolated soy protein films (Otoni et al., 2016), and whey protein isolate films (Ghadetaj et al., 2018). The reduced TS was attributed to the plasticising effect of the nanoemulsion droplets, which weakened intermolecular interactions between polymeric chains. In a separate study, Kowalonek et al. (2023) found that adding Tansy EO did not affect the EAB.

Differential Scanning Calorimetric (DSC) Curves

Figure 3 illustrates the DSC curves of pectin-pineapple juice (PPJ) films varied from 0% to 20% loadings of GEONe. These curves represent the thermally induced endothermic changes in the PPJ-GEONe films acquired at a temperature range of -25°C to 200°C. Adding the nanoemulsified ginger essential oil (GEONe) to the PPJ film reduced the enthalpy change (ΔH) values compared to the control film. Among the PPJ-GEONe films, the PPJ-5% GEONe film exhibited the highest ΔH , compared to the control film and PPJ film blended with 10%, 15%, and 20% GEONe. The ΔH of PPJ-GEONe film decreased from 143.01 J/g (PPJ-5% GEONe) to 125.80, 101.79, and 79.60 J/g with the increasing addition of GEONe, respectively. The decrease in the ΔH value of PPJ-GEONe film is also associated with a decrease in the percentage of moisture loss calculated by integrating the area under the endothermic peak in the DSC curves. The existence of hydrophobic ginger essential oil droplets with a high molecular weight disrupted intermolecular interactions within the molecular structure of the PPJ film. Consequently, the heat absorbed softens the structure of the PPJ films combined with GEONe was reduced, improving the film's capable of being extended or shaped below the melting temperature (Nisar et al., 2018; Tongnuanchan et al., 2015). Similar observations were reported by Jahromi et al. (2022) when clove essential oil, both emulsified and non-emulsified, was incorporated into high methoxyl pectin films, resulting in a general reduction in ΔH value.

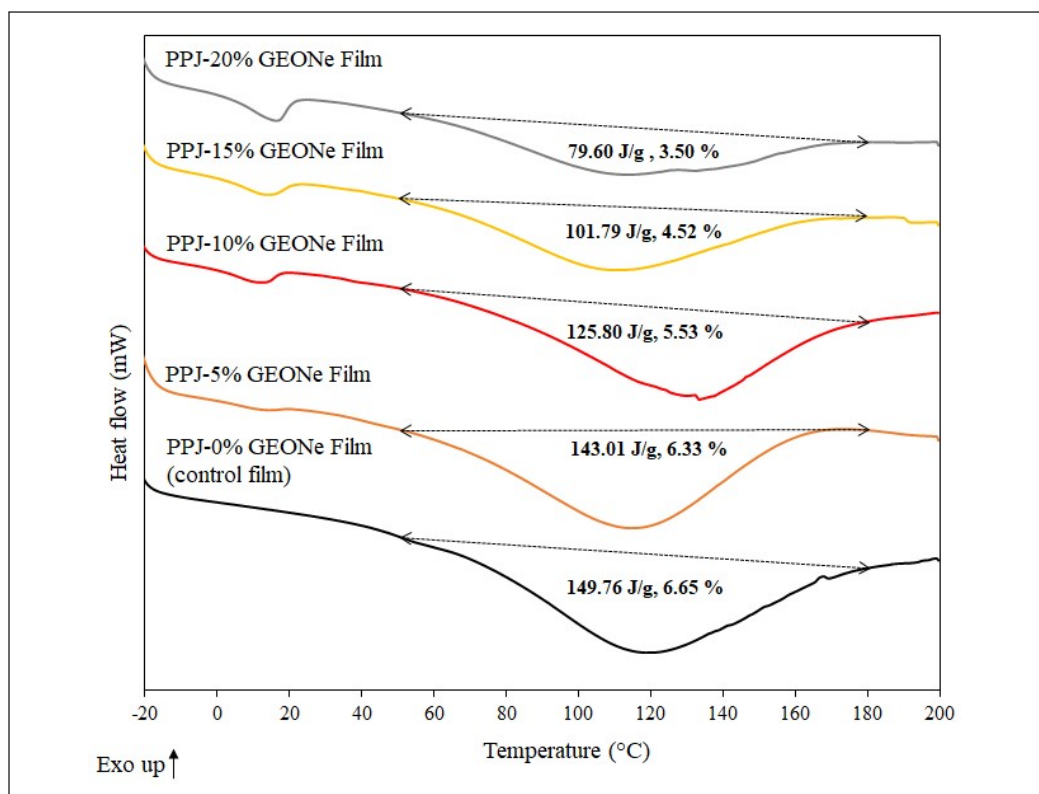


Figure 3. The differential scanning calorimetric (DSC) curves of PPJ-GEONE films
 Note. Enthalpy of crystallisation, ΔH (J/g); Moisture loss (%)

CONCLUSION

A stable nanoemulsion of ginger essential oil (GEONE) was successfully produced and incorporated at various loadings to pectin-pineapple juice (PPJ)-based films for improving the PPJ film's physical, mechanical, and thermal properties. The film's formulation consisting of 5 mL ginger essential oil, 3 mL virgin coconut oil, and 1.5 mL polysorbate 80, prepared by sonication at 50% amplitude for 9 min, yields the smallest droplet size with good stability as a nanoemulsion at day 7 (26.00 nm). As the loading of GEONE incorporated into the PPJ film increased, the film thickness significantly increased ($p \leq 0.05$) by 18.75% (PPJ-20% GEONE) compared to the control PPJ film (thickness = 91.0 μm). The moisture content decreased insignificantly ($p > 0.05$) from 11.57% to 10.33%, and the water solubility decreased significantly ($p \leq 0.05$) from 93.77% to 83.16% as the GEONE loading increased from 0% GEONE (control film) to 15% GEONE. The reduction in both moisture content and water solubility can be attributed to the cross-linking of pectin with essential oil, reducing the affinity of the pectin-based film towards water. All PPJ films blended with GEONE exhibited good flexibility, ease of peeling, and a

smooth surface appearance. The incorporation of GEONE resulted in decreased ($p \leq 0.05$) lightness but increased ($p \leq 0.05$) in the yellowness index and total colour differences ($p \leq 0.05$). Incorporating 20% GEONE into the PPJ film led to a significant decrease ($p \leq 0.05$) in the TS and YM to 9.54 ± 2.60 MPa and 187 ± 38.58 MPa, respectively. However, the elongation at break (EAB) of the PPJ-20%GEONE films did not show a statistically significant change ($p > 0.05$) compared to the control film. In terms of thermal properties, there is a decrease in the enthalpy change as the GEONE loading in the PPJ films increases from 5% to 20% (with a range of $\Delta H = 143.01$ J/g to 79.60 J/g). The PPJ-GEONE films have better physical, mechanical and thermal properties compared to the control film. In conclusion, the addition of GEONE alters the physical properties of the PPJ films, enhancing their flexibility and shaping ability, thereby making them potentially suitable for edible food packaging applications.

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