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Evaluation of sorbitan monolaurate concentration on carrageenan gel properties

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Abstract

Air fresheners are chemical products that have been used in the field of environmental sanitation for decades. The brands, materials and types of fragrance air fresheners have been developed to reduce the depression level amongst people. This study used seaweed from carrageenan to explore renewable resources from Malaysia as biopolymer. It is proven that the mild depression or stress can be released with help of aromatherapy substances, and one is aromatherapy scented gel. The objectives of this study were to formulate and characterize the scented gel from carrageenan and the stability of the scented gel. The gel was produced by mixing sorbitan monolaurate (surfactant) and carrageenan in water. Then, polyethylene glycol (PEG), alginic acid and essential oil were added into the mixture. The solution viscosity and shear stress for rheology properties were measured. The highest viscosity was the sample without sorbitan monolaurate which is 979.37 mPa·s while the highest shear stress was 0.50% (v/v) sorbitan monolaurate with 296.42 Pa. Moreover, for weight variation analysis, sample with 0.5% (v/v) sorbitan monolaurate showed the lowest residual loss which is 11.8% compared samples with higher concentration of sorbitan monolaurate. Bloom test demonstrated samples with 1.50, 2.00, and 2.50% (v/v) have high bloom when the gel strength increased at higher concentration of sorbitan monolaurate. Sorbitan monolaurate of 0.5% (v/v) of is a stable gel with high mass of residue. In conclusion, gel air freshener could be produced from carrageenan to replace other gelling material such as gelatin, xanthan gum and guar gum.

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1.0 Introduction

Freshener consists of two basic ingredients which are known as fragrances and solvents (Hutagaol, 2017). Water and oil are types of solvents used to produce air freshener. Since antiquity, fragrance compounds have been used to freshen up the air and mask odors. As an example, ancient Egyptians used musk and other natural materials to scent their tombs (Bratovčić, 2019). Variety of compounds, including numerous spices and floral extract, have been used for their pleasant aroma over the last 2000 years ago. However, in 1948, the first modern air freshener was introduced. The function was based on a military technology for dispensing insecticides and was adapted into pressurized spray using a chlorofluorocarbon (CFC) propellant. According to Masahiro et al. (2011), positive emotions elicited by certain fragrances have been shown to lower stress levels and improve overall mental outlook. Pleasant smell such as nostalgic scent can increase the comfort feeling and decrease the negative emotion in an individual (Herz, 2016). The gel air freshener can also act as an aromatherapy as it contains essential oil which functions as a fragrance. The existing gel freshener or scented gel are made from expensive materials such as gelatine, xanthan gum and gellan gum. A cheaper and more stable property of gel material is needed to replace the material from Malaysia local material.

Carrageenan is a substance extracted from red and purple seaweeds, consisting of a mixture of polysaccharides. Carrageenan is a vegan alternative to replace gelatine in some applications such as confectionery due to its gelling, thickening and stabilizing properties. Nonetheless, the main application is in dairy and meat products due to its strong binding to food proteins.

Malaysia is the world's third-largest producer of extracted carrageenan for the international market (Nor et al., 2019). Meanwhile, Southeast Asian countries accounting for 96.5% of total kappa-carrageenan production. The Philippines accounts for the highest production (55%), followed by Indonesia (38%) and Malaysia (2.5%) (Bono et al., 2011). Carrageenan refers to a class of sulphated galactans derived from marine red algae. Carrageenans are classified according to their primary structure. K-carrageenan is composed of alternating α (1-3)-d-galactose-4-sulphate and β (1-4)-3,6-anhydro-d-galactose (Michel et al., 1997). Compression techniques have been used extensively to study the rheological properties of kappa-carrageenan (Chen et al., 2002). The coil-helix transition and subsequent aggregation of double helices are thought to be involved in carrageenan gelation. Carrageenan is used not only as a texturing agent in the food, cosmetic, and personal care industries, but also in medical care, drug-controlled release, and encapsulation due to its high biodegradability and biocompatibility (Yang et al., 2018). The type and quantity of counter-ion present in the solution have a strong influence on the gelation mechanism (Ould Eleya, 2000). Carrageenan is the least sulfated and easiest to gel fraction (Arda et al., 2009). Carrageenan is widely used in the food industry as a gel or viscous agent, and it also has physiological effects such as anti-tumor activity (Yuguchi et al., 2002). In carrageenan/hydroxylpropyl methyl cellulose biocomposite, the formation of intermolecular hydrogen bonding forms and enhances the mechanical and thermal stability of the composite (Ramli et al., 2022). Carrageenan bioplastic film incorporated with microcrystalline cellulose improved the biocomposite matrix's tensile strength, elongation at break (EAB), moisture content and film appearance (Othman et al., 2021). Gum Arabic-Kappa carrageenan biocomposite films in hard capsule application was compatible to produce a homogeneous and viscous biocomposite solution with dipping ability to form hard capsule (Adam et al., 2020).

Sorbitan monolaurate is a non-ionic surfactant which contains sorbitol esterified with coconut oil-derived fatty acids (Bampidis et al., 2020). Because of its C12-alkyl chain and relatively high lipophilicity, sorbitan monolaurate was chosen as non-ionic surfactant for oil-in-water emulsion formation and stabilisation (López et al., 2000).

The aim of this study was to evaluate and characterize the formulated scented gel from carrageenan on the effect of different concentration of surfactant, which was sorbitan monolaurate on gel properties.

2.0 Methodology

2.1 Raw Material

Semi-refined carrageenan was purchased from TACARA, Sabah. Polyethylene glycol (PEG) (pH = 4.5–7.5), sorbitan monolaurate (GC ≥44.0%), sodium chloride (NaCl) (≥99.0%) and alginic acid were purchased from Merck, German. Food colouring was purchased from Star Brand meanwhile essential oil was obtained from Yubiso.

2.2 Scented gel solution preparation

The scented gel was prepared by mixing 7.5 g of powdered carrageenan into 150 mL deionised water at 70 °C. The solution was left stirred on the hot plate for 30 minutes. Sorbitan monolaurate was then mixed into the homogenous solution. After another 30 min, 3 mL, 3 g, and 0.2 g of PEG, alginic acid, and NaCl were added, respectively. The solution was left stirred for 3 hours. Colorant and essential oil were added15 min before the stirring process stopped.

2.3 Rheology analysis

The viscosity of carrageenan formulation solutions was determined using a cup and bob rotational measuring block Rheometer (Brookfield, Rheo 3000, USA) with the LCT 25 4000010 geometry. In triplicate, 16.5 mL of solution was programmed at 300 rpm with 100 MPoints at a constant temperature of 40 °C (Adam et al., 2020).

2.4 Weight variation analysis

The solution evaporation test of gel solution was performed by weighing the gel weight for 10 days. After 10 days of storage, scented gel weight loss and total weight loss were recorded and calculated. The weight reduction of scented gels was calculated by subtracting the weight of the gel at the time of weighing (M_n) from the total weight loss (M_4) and gel weight at initial time (M_0). The weight difference represents the amount of liquid which evaporated. Gravimetry was used to determine the percentage of total liquid evaporation and the percentage of residual gel weight, which were then calculated using Eq. (1) and (2):

$$\text{Total Liquid Evaporation (\%)} = \frac{M_n - M_{n-1}}{M_0} \times 100\% \quad (1)$$

$$\text{Residual Gel Weight (\%)} = \frac{M_n}{M_0} \times 100\% \quad (2)$$

where is M_n: the weight of gel at the time of weighing; M_{n-1} : weight of the gel in the previous week; M_0 : gel weight at initial time (Hutagaol, 2017).

2.5 Gel bloom strength test

The CT3 Texture Analyzer (Fig. 1) was used to conduct mechanical and bloom strength tests (Brookfield, Rheo 3000, USA). An average of at least three measurements was taken for each formulated solution. For this analysis, a flat-end probe (TA-10) with a diameter of 12.7 mm and a Brookfield bloom jar were used. Scented gel was placed on the texture analyzer platform and centred on the probe. The testing mode was set to 'compression', and the target option was set to 4.0 mm. The probe was pressed onto the gel at 1.0 mm/s and the breaking force was measured. (Hamdan et al., 2020).

3.0 Results and discussion

3.1 Rheology analysis

The decrease of average viscosity of sample without sorbitan monolaurate (0%), $C_{18}H_{34}O_6$ to 2.50% (v/v) of sorbitan monolaurate concentration used in the formulation is shown Fig. 2. The viscosity has reduced progressively over time (no results viscosity-time shown). Initially, viscosity of gel solution without sorbitan monolaurate was 979.37 mPa·s, then decreased to 586.71 mPa·s at 2% (v/v) but increased slightly to 633.29 mPa·s at 2.5% (v/v). This would be resulted from the interaction of $C_{18}H_{34}O_6$ with other chemical matrix in the gel solution such as carrageenan and alginic acid formulation of intermolecular interaction which should be further investigated in the gel matrix.

Fig. 1: Gel bloom test using texture analyser

Gel composite with 15 mM Na^+ has the highest viscosity. This behaviour may be due to the concentration of Na^+ which strengthened the hydrophobic interaction and the electrostatic interaction between WNPI (walnut protein isolate) and KC (kappa-carrageenan), as well as the high-molecular-weight polymers formed by the two may have caused higher apparent viscosity (Lei et al., 2022). Carrageenan extracted from different red algae species cultivated in Cam Rahn Bay, Vietnam indicated that kappa-carrageenan from *K. alvarezii* had larger viscosity value compared to other carrageenan samples (Bui et al., 2018) whereas semi-refined carrageenan used in this study was extracted from *Kappaphycus alvarezii*. Semi-refined carrageenan in addition of KOH at lower concentration for 30 minutes cooking time at 80 °C demonstrated a high gel viscosity of 1291.84 mPa·s (Bono et al., 2014). Kappa-carrageenan bio composite films in the addition of Arabic Gum demonstrated viscosity of 1058 mPa·s (Adam et al., 2020) which was slightly lower. Bioplastic solution in the incorporation of microcrystalline cellulose into refined carrageenan showed viscosity and shear stress of 293.90 mPa·s and 27.90 Pa, respectively (Adam et al., 2022). The viscosity range in this study was from 586 to 979 mPa·s. The viscosity of bio composite solution in the presence of hydroxyl propyl methyl cellulose (HPMC) in carrageenan hard capsule was 616.5 mPa·s (Ramli et al., 2022). Carrageenan bio composite solution with *Chlorella vulgaris* blending demonstrated the viscosity

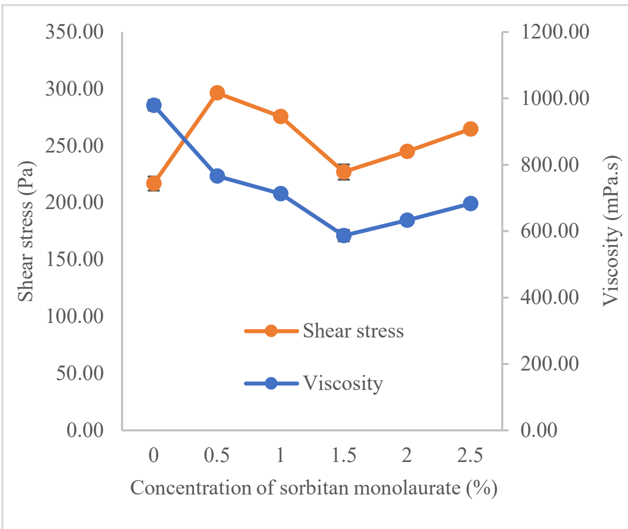

Fig. 2: Viscosity and shear stress at different concentrations of sorbitan monolaurate

of 245.29 mPa·s (Mat Yasin et al., 2022). The viscosity of carrageenan-based bio composite film formulation incorporated with microcrystalline cellulose (MCC) and carboxymethyl cellulose (CMC) were 0.982 mPa·s and 1.48 mPa·s, respectively (Hamdan et al., 2021).

3.2 *Weight variation analysis*

Fig. 3 and Table 1 summarised the residual loss of scented gel. The graph was plotted with the reading from day one until day ten. Residues at 0.5% (v/v) sorbitan monolaurate is 11.8%. The one with least residue 6.95%. was at 1.5% (v/v).

Kappa-carrageenan and non-ionic surfactants which is Tween-20 has a mechanism of interaction between the two species which allows for more effective shielding of the carrageenan helices (Fenton et al., 2021). Weight loss at 3% concentration of carrageenan with induction of lavender essential oil was 76% meanwhile at lower concentration of carrageenan, weight loss increased (Mat Amin & Abdul Hadi, 2020). In this study, constant concentration of 3% carrageenan was used and fixed for every sample at different concentration of sorbitan monolaurate. The weight loss of gel increased with increased concentration of sorbitan monolaurate. The influence of water-ethanol mixtures on 1% and 2% kappa-carrageenan gel in 100% concentration of ethanol demonstrated that weight loss was 67.6% and 59%, respectively (Sason & Nussinovitch, 2018). Carrageenan gel only lost significant weight when immersed in acetone solutions with concentrations greater than 50%. Losses were only moderate at lower acetone concentrations, ranging from 5 to 18%. At 10% acetone, the gel gained weight (Nussinovitch & Peleg, 1990).

Table 1: Residual gel weight for at different concentrations of sorbitan monolaurate

Sample (% (v/v))	Residual Gel Weight (After 10 days)
0	6.99
0.5	11.8
1	10.52
1.5	6.95
2	8.08
2.5	7.25

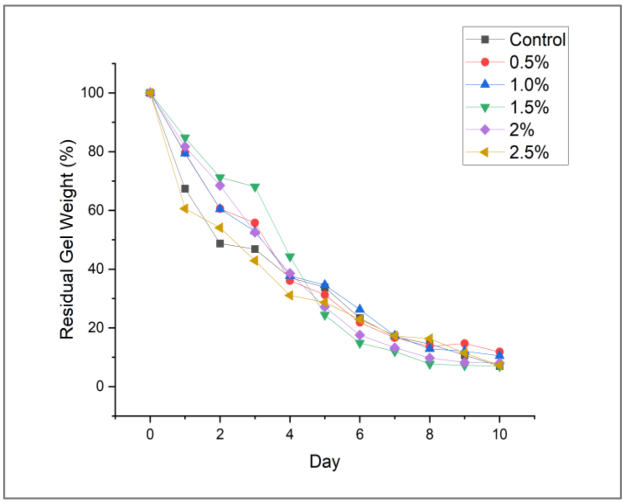

Fig. 3: Residual gel weight for 10 days at different concentrations of sorbitan monolaurate

3.3 *Gel bloom strength test*

Bloom number describes gel strength. The gel strength was low when bloom number was lower than 150 g (150–220 g represents a medium gel strength). Meanwhile 220–300 g bloom number indicates high gel strength (Johnston-Banks, 1990). In Fig. 3, control sample could be concluded to have low bloom as the obtained number of blooms were 83.29 g. Likewise, sample of 0.50% (v/v) produced a low bloom with only 104.69 g. Sample with 1.00% (v/v) also showed low bloom as the average bloom number was below than 150 g. This concentration was a turning point of the bloom strength from low to medium strength of the formulated scented gel. Meanwhile, samples with 1.50%, 2.00%, and 2.50% (v/v) were high bloom and could be concluded as strong gel due to high number of bloom strength. Increasing of NaCl concentration in soy protein isolate and secondary gums on Konjac gel resulted in the increased of gel strength (Akesowan, 2011).

Kappa-carrageenan in addition of surfactant either Tween-20 or Dehypon LS 36 formulation solution showed that as carrageenan concentration increased from 0.6% to 2% w/w, the gel strength decreased (Fenton et al., 2021). Meanwhile in this study, gel

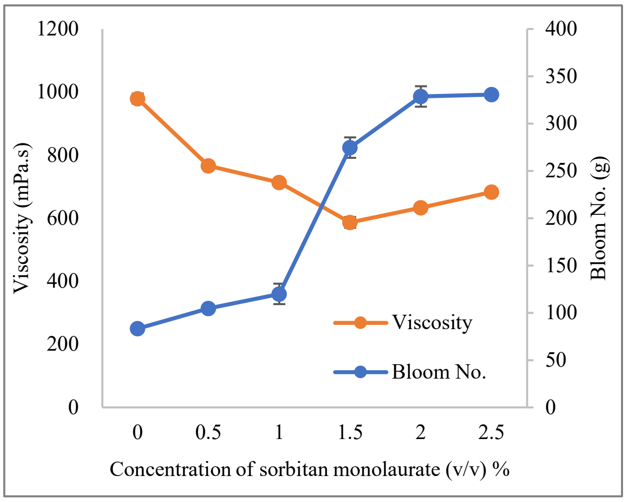

Fig. 4: Gel bloom strength and viscosity at different concentrations of sorbitan monolaurate

strength increased with increasing concentration of sorbitan monolaurate. This is probably because of the different chemical structure between Tween-20 and sorbitan monolaurate. Tween-20 or polyoxyethylene sorbitan monolaurate is a non-ionic surfactant that can protect proteins against surface induced damage by competing with proteins for adsorption sites on surfaces (Chou et al., 2005). Iota-carrageenan extract is another type of carrageenan; whereby using $Ca(OH)_2$, it has higher gel strength (~ 337.1 $g{\cdot}cm^{-1}$) as compared to being extracted with NaOH (Jiang et al., 2022). In this study, sodium chloride (NaCl) was also used in the formulation. The presence of Na^+ may probably affect the gel strength of the scented gel. Kappa carrageenan exhibited a synergistic effect with gelatine to alter and increase the gelling temperature, melting temperature and denaturation temperature of gelatin-based composite gels. Kappa carrageenan increased the gel strength from 1.68 to 4.13 times such as toughness and chewiness. On the contrary, the addition of konjac glucomannan resulted in gelatin composite gel network structure became denser and most ordered (Cheng et al, 2022).

4.0 Conclusions

In conclusion, the scented gel from carrageenan was successfully evaluated the stability of the gel formulation. The incorporation of sorbitan monolaurate in the scented gel matrix at different concentrations reduced the viscosity but increased the gel strength for the formulated scented gel. Meanwhile, highest residual gel weight at 0.5% (v/v) indicated that the gel was the most stable to maintain its structure up to 10 days compared to higher concentration of sorbitan monolaurate being added into the formulation. More extensive characterization of gel rheological property such as storage and loss modulus should be conducted to understand the gelling of the formulated matrix.

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