

Preparation and characterization of sago/iota-carrageenan microgels as food thickener in texture modified food

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Abstract

An increasing number of older populations with swallowing difficulties need specialized diets with easy-to-chew and safe swallowing characteristics. Texture modification using a thickener is usually used to obtain the desired texture, which helps in lowering the risk of aspiration. Microgels usage to modify the texture and rheology of liquid and pureed foods has been an area of interest in the food industry for many years. Therefore, this study aimed to produce sago starch and ι-carrageenan based microgels with functionality as food thickeners. Different concentrations of ι-carrageenan (25%, 50% and 75%) were added into sago starch dispersion and then fabricated using ultrasound treatment followed by spray drying process. A commercial dysphagia thickener that is normally used at medical institutions and nursing homes was also included as a reference. Microgels with increasing ι-carrageenan concentration showed variation in behaviours during rheological and textural measurements. Rheological tests revealed that sago/ι-carrageenan microgels exhibited a predominant elastic behaviour, with $G' > G''$ within the frequency range. Texture profile analysis (TPA) suggested that the sample with 2.0 g of sago starch and 2.0 g ι-carrageenan, MS50, produced samples with similar hardness, springiness, and cohesiveness to the reference sample. Overall, sago/ι-carrageenan microgels have shown a potential to be used as an alternative thickener as it provides low adhesiveness and more elastic behaviour which is considered safe-to-swallow food, especially for elderly with swallowing difficulties.

1. Introduction

The world population is ageing quickly. It is estimated that approximately 2 billion people will be aged 60 and over by 2050. In many countries like Japan, Germany and Korea, approximately 15% of their populations will be over 80 years old, making the elderly the fastest-growing demographic group (Aguilera and Park, 2016). The ageing phenomenon imposes a challenge in feeding the elderly consumer and satisfying their special diet requirements (Leon *et al.*, 2016). For instance, ageing individuals experience increasing difficulties in masticating and swallowing oral contents due to anatomical and physiological alterations. Dysphagia or difficulty in transferring food, liquid, saliva, or medications from the mouth to the stomach is common among the elderly (Cichero *et al.*, 2013). Dysphagia increases dramatically during senescence and can cause malnutrition and morbidity by aspiration pneumonia (Teramoto *et al.*, 2008; Khan *et al.*, 2014).

Therefore, in order to maintain their nutritional intake, diet modification such as texture-modified food (TMF) is usually prescribed for frail old people as a compensatory method to treat dysphagia (Cichero, 2016; Funami, 2016; Painter *et al.*, 2017). TMF is a term that refers to foods with soft textures and/or reduced particle size as well as thickened liquids (drinks) aimed at the market segment of seniors with eating dysfunctions (Cichero, 2015). Food textures recommended for the elderly should be soft and moist; sticky and adhesive textures should be avoided as well as fibrous structures that are not easily disintegrated (Cichero, 2016). Foods with soft textures are preferred because they are disintegrated and mixed in the mouth by tongue-palate compression avoiding teeth mastication (Ishihara *et al.*, 2012). TMF include foods which are softened by processing, minced, pureed or liquidized as well as liquids that have been thickened to various extents (IDDSI, 2020). In addition, to prevent choking and

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aspiration into the lungs, a suitable thickener that can control the flow rate of thin liquids while swallowing is being investigated (Quinchia *et al.*, 2011; Moret-Tatay *et al.*, 2015).

Starch has been widely used in texture modification because of its ability to absorb water, causing an increase in viscosity. Clavé *et al.* (2006) reported that an increase in food viscosity can help to improve the safety during swallowing for patients with dysphagia. However, the use of starch as a thickening agent is often associated with its strong starchy flavour with some of them imparted off-flavours, and grainy texture, thus, reducing the acceptability of thickened drinks and foods (Moret-Tatay *et al.*, 2015). In addition, the usage of modified starch as a thickening agent has been shown to be unstable during swallowing, in which the thickened product could be broken down on contact with amylase-containing human saliva, resulting in reduced viscosity of the thickened food or drink. Human saliva contains α -amylase, an endoenzyme that catalyzes the hydrolysis of α -1,4-glycosidic bonds in starch to produce short oligosaccharides (Vallons *et al.*, 2015). Too thin food or drinks might have serious repercussions for patients who consume them which may lead to choking (Cichero, 2013).

Alternatively, microgels are used as texture modifiers, often incorporated into finished liquid foods such as juices, beverages and milk (Ellis and Jacquier, 2009). Microgels are small, soft, and stable particles that can be tailored to specific structures because of their vast range of forms, sizes, and textural characteristics (e.g., diameters from 1-100 μ m) (Nicolai, 2016). Microgels are commonly produced by direct gelling, frequently under shear, in a particle or fibre shape, or mechanically reducing bulk gel size. Microgel suspension is usually free-flowing, in contrast to bulk gels which often exhibit viscoelastic behaviour (Dickinson, 2015).

Microgels have been proposed as delivery systems for non-polar substances, such as vitamins, flavours, antimicrobials, and antioxidants, which can be distributed in tiny micelles or more functional liposomes (20 nm and a few hundred nm) in the aqueous phase, apart from their function as texture modifiers (Zhang *et al.*, 2015). Micron-sized hydrocolloid gel particles with a high water content (e.g., >95%) are extremely alluring to be used as structuring agents to consolidate dispersed phases as well as soup and sauce thickeners due to their soft texture and flowability (Raheem *et al.*, 2021).

The combination of sago starch and ι -carrageenan (ι -car) in microgel development is yet to be explored. Due to its mechanical and viscoelastic properties, blending of starch and ι -car has been found to be beneficial in

improving starch properties in terms of elasticity, cohesiveness, springiness and adhesiveness without any chemical modification (Tischer *et al.*, 2006; Al-Baarri *et al.*, 2018). In addition, carrageenans are not susceptible to the action of salivary α -amylase. Hence, thickeners incorporated with carrageenan are hypothesized to be less sensitive to amylase enzyme and will enhance the safety of swallowing by patients with dysphagia. This is because carrageenan is comprised of α -1,3 and β -1,4-glycosidic linkages, and does not contain α -1,4-glycosidic linkages, which can withstand the α -amylase reaction (McKim *et al.*, 2019). Therefore, the objective of this study was to develop and characterize microgels from sago starch and ι -carrageenan mixtures, in terms of rheological and textural properties.

2. Materials and methods

2.1 Materials

Sago starch was purchased from Sim Company Sdn. Bhd., Penang, Malaysia and ι -car was obtained from Modern-Lab Chemicals Sdn. Bhd., Penang, Malaysia. Commercial thickener (modified corn starch (MC), brand Valens Thixer), specifically designed for dysphagia was store-bought and used as a control sample.

2.2 Preparation of microgels

Ultrasonication of the sago/ ι -car mixtures was done in accordance with Azizi and Farahnaky (2016) method with several modifications, whereas spray drying was carried out according to the method described by Marín-Peñalver *et al.* (2021) with slight modifications. The composition of sample solutions is reported in Table 1. Briefly, the solutions were dissolved in deionized water followed by heat treatment at 90°C for 60 mins with magnetic stirring and subsequent cooling to 50°C. The samples were treated with ultrasounds for 10 mins at 20°C using an ultrasonicator, Model UP200S, Hielscher, Germany, at a frequency of 20 kHz and power 100 W. The ultrasound probe was immersed into the sample of approximately 2.5 cm. The polysaccharide mixtures (50°C) undergo spraydrying (SD) using a mini spray-dryer (model B-290, BUCHI UK Ltd, UK) with an

Table 1. Compositions of control and microgels samples.

| Samples | Sago (g) | ι -car (g) |
|------------------------------|----------|------------------|
| Native sago starch (NS) | 4.0 | - |
| Commercial ι -car (CI) | - | 4.0 |
| Microgel sago (MS) | 4.0 | - |
| Microgel ι -car (MI) | - | 4.0 |
| MS25 | 3.0 | 1.0 |
| MS50 | 2.0 | 2.0 |
| MS75 | 1.0 | 3.0 |

inlet temperature of 150°C, an aspirator at 100%, a pump at 50% and a Q-flow rate of 12.3 L min⁻¹. The microgels suspensions were continuously agitated with a magnetic stirrer, to avoid the deposition throughout the drying process. The spray-dried samples were stored in sealed plastic bags at room temperature before the analysis. For commercial thickener (TX), was prepared according to the manufacturer's instructions, where 3½ scoops of Thixer were used to achieve the characteristics of pureed food (Level 4) according to the International Dysphagia Diet Standardization Initiative (IDDSI) framework.

2.3 Particle size distribution

The particle size distribution was measured at 25°C using a particle size analyzer of Malvern Mastersizer 2000 (Malvern Instruments Ltd, UK). Each measurement was repeated three times. The mean particle size was expressed as D_[3,2], which represented the area-volume mean diameter.

2.4 Moisture content and water activity

The moisture content of the powder sample was measured by the oven drying method following AOAC 925.10 (Association of Official Analytical Collaboration (AOAC) International, 2000). The water activity (aw) was determined by using a digital aw meter (AquaLab Series 3, METER Group, Inc., USA) at 25°C. All the measurements were performed in triplicates.

2.5 Morphology of microgels

The microstructural characteristics of the samples were evaluated with a scanning electron microscope (SEM) (FEI Quanta FEG 650, Poland). Samples were coated with gold/palladium and observed under a voltage of 15 kV. Micrographs were taken at 1000× and selected images from at least three specimens per sample are reported.

2.6 Water solubility

The water solubility was measured by following the method of Zendeboodi *et al.* (2019) with some modifications. The aqueous suspension (1%) of all samples (100 mL) was heated at 95°C with constant stirring in a water bath for 1 h. The suspension was allowed to cool at 30°C for 30 mins. Then the samples were centrifuged for 10 mins at 3000×g. The supernatant was dried to constant weight in an air oven at 105°C. Water solubility was calculated by using Equation 1.

$$\text{Solubility (\%)} = \frac{\text{Weight of dried supernatant (g)}}{\text{Weight of dried sample (g)}} \times 100 \quad (1)$$

2.7 Rheological properties of microgels

A small amplitude of dynamic measurement and a

steady shear measurement were conducted using a controlled stress rheometer (AR1000, TA Instruments, New Castle, DE, USA) equipped with Rheology Advantage Data Analysis Program (TA, version V5.7.0). A 20 mm parallel plate with a gap of 1 mm was used. Stress sweeps were performed at 1 Hz to determine the linear viscoelastic region. Then, frequency sweeps were performed over the range between 0.1 to 100 rad s⁻¹ and the values of the storage or elastic modulus (G'), loss or viscous modulus (G'') and loss tangent ($\tan \delta = G''/G'$) were calculated using the equipment software. Native sago starch and commercial τ -car were not included in this section as the focus is directed towards the preparation and characterization of microgels.

2.8 Texture profile analysis of microgels

Texture profile analysis (TPA) was performed by following the method of Zhu (2015) with some modifications. Samples (around 20 g) of microgels were subjected to back extrusion cycles using a TA.XTplus Texture Analyzer (Stable Micro System Ltd, Goodling, Surrey, UK). Testing was done at a speed of 1 mm s⁻¹ and a downstroke to 20 mm from the bottom of the cell (approximately 50% of the initial height of microgels). At least three measurements were recorded for each type of sample. TPA results were used for calculating the hardness, adhesiveness, springiness, cohesiveness and gumminess of the samples. Native sago starch and commercial τ -car were not included in this section as the focus is directed towards the preparation and characterization of microgels.

2.9 Statistical analysis

Statistical analyses were conducted by using SPSS Statistics Desktop 26.0 (IBM Corporation, US). The results obtained were represented as the mean values of three individual replicates \pm standard deviation (SD). The significance of differences between means was estimated with post hoc one-way ANOVA using Tukey's test at a 5% probability level.

3. Results and discussion

3.1 Particle size and distribution analysis

The results of particle size and distribution analysis are presented in Table 2. The mean diameter of TX was 25.88 μm which was comparable to the findings by Lopez-Silva *et al.* (2019) on modified corn starch. The mean diameter of NS was 37.89 μm almost similar to the result reported by Okazaki (2018). The mean diameter of CI was 54.84 μm , as reported by Wullandari *et al.* (2021) that the particle size all fell below 100 μm in size.

The microgels' mean diameters were between 5.23

μm and $10.41 \mu\text{m}$ that were significantly smaller ($p < 0.05$) than the NS and CI. This result was almost similar to the findings of Joye and McClements (2014), who reported that generally particle diameters obtained by spray drying vary between $1 \mu\text{m}$ and $10 \mu\text{m}$. Moreover, the particle size reduction after being subjected to the cavitation effect of ultrasound was consistent with the studies reported by other researchers on starch and carrageenan (Zendeboodi et al., 2019; Tan et al., 2021).

Table 2. Particle size of control and microgels samples at different ι -car concentrations.

| Samples | d_{32} (μm) area-volume mean diameter |
|---------|--|
| NS | 37.89 ± 0.63^c |
| CI | 54.84 ± 0.99^f |
| MS | 7.10 ± 0.34^b |
| MI | 10.41 ± 0.51^c |
| MS25 | 6.89 ± 0.32^b |
| MS50 | 5.23 ± 0.24^a |
| MS75 | 6.36 ± 0.21^{ab} |
| TX | 25.88 ± 0.25^d |

Values are presented as mean \pm SD of triplicates. Values with different superscripts within the same column are statistically significantly different ($p < 0.05$).

The particle size of the microgels was significantly influenced by starch concentration. At constant power, large microgel particle sizes were obtained with MS25 compared to samples MS75. This feature coincided with results found by Gallant et al. (1972) who reported that the extent of damage on the granules by ultrasonication decreased with increasing starch concentration thus making its particle size larger.

The differences between the size of MS25, MS50 and MS75 also relied on their ι -car concentration. The presence of a high ι -car concentration led to a greater molecular weight than the sample with a low ι -car concentration. A low molecular weight seems to promote interactions between starch granules and carrageenans, as already mentioned by Huc et al. (2014.) However, MS75 did not interact much with starch granules at small concentrations. At higher concentrations, it seemed that MS50 carrageenan interacted to a better extent than MS25 carrageenan.

According to Engelen et al. (2005), concentration, dispersion medium and particle size were all important factors contributing to perceived grittiness. The other author, Lopez et al. (2016), reported that smaller particle sizes were considered less gritty than larger particles and, in turn, samples containing smaller particles were regarded as more pleasant. Therefore, in this regard, a reduction of particle size could be a potential strategy to improve the palatability of thickened food or beverage

products.

3.2 Moisture content and water activity

Table 3 presents the moisture content of all samples. Moisture content is a measurement of the total amount of water present in a sample. The moisture content of the samples analyzed ranged between 9.21% and 14.57% with CI showing the highest moisture content (14.57%), while MS25 was the lowest (9.21%). These moisture content values obtained from this study were less than 20% and were acceptable because it is allowed commercially up to 20 % of moisture in starch as raw material (Yousif et al., 2012). On the other hand, the high moisture content achieved for CI could be due to the fact that ι -car trapped more water in the interstitial spaces of the gel, which reduced the drying rate hence the final moisture content will be higher (Palanisamy et al., 2018; Pratama et al., 2018).

A significant increase ($p < 0.05$) in moisture content can be observed when 2.0 g and 3.0 g of ι -car were added to the sago starch (9.21% increase to 10.57%) The moisture content increases as the % of ι -car increases implying that ι -car increases the microgels' affinity to water. For hydrocolloids, hydrophobic properties are usually observed when intra-molecular bonds increase indicating that ι -car affects the network structure of the microgels.

Table 3. Moisture content of control and microgels samples at different concentrations of ι -car.

| Samples | Moisture (%) | aw |
|---------|--------------------|-------------------|
| NS | 13.59 ± 0.05^c | 0.32 ± 0.00^b |
| CI | 14.57 ± 0.04^g | 0.36 ± 0.00^c |
| MS | 9.24 ± 0.10^a | 0.27 ± 0.00^a |
| MI | 13.87 ± 0.07^f | 0.25 ± 0.01^a |
| MS25 | 9.21 ± 0.05^a | 0.31 ± 0.00^b |
| MS50 | 9.83 ± 0.07^b | 0.28 ± 0.00^a |
| MS75 | 10.57 ± 0.10^c | 0.26 ± 0.00^a |
| TX | 12.89 ± 0.08^d | 0.31 ± 0.00^b |

Values are presented as mean \pm SD of triplicates. Values with different superscripts within the same column are statistically significantly different ($p < 0.05$).

Water activity (aw) determines the ability of water in food to react with microorganisms. It measures the presence of water in food which is not bound to food molecules and which can support the growth of bacteria, yeasts and fungi. The higher the water activity, the higher the amount of free water in food, and hence the faster the microorganisms will be able to grow. All the aw values of the samples in this study varied from 0.25 - 0.36. This observation is in agreement with Larotonda et al. (2005) on carrageenan- rice starch blend in which the blends are becoming less hydrophilic as the proportion of

ι -car is increased. The significantly lower a_w values obtained by sago/ ι -car microgels indicated that microgels have greater water holding capacity than sago starch, which can help to retain water during the cooking or drying process.

According to Abdel-Haleem and Omran (2014), most of the microbial activity, fungi, and yeasts are inhibited when a_w was below 0.6, 0.7, and 0.8, respectively. In addition, Fernández-López *et al.* (2009) mentioned that the ideal range of a_w for low moisture food should be between 0.11 to 0.40. Food with low a_w is often desirable to consumers due to the perception that microorganisms are unable to grow in dry conditions, and thus the shelf life of food can be extended (Ijabadeniyi and Pillay, 2017).

3.3 Scanning electron microscopic of the microgels

Figure 1a presents SEM images of TX that were made from modified corn starch. Granules are irregular in shape with some pits compared to untreated corn starch and usually have smooth surfaces without cavities or fractures (Lopez *et al.*, 2019). From Figure 1b, NS, native sago starch, exhibited sphere or oval shapes with most of the particles intact, whereas some particles had truncated ends (Du *et al.*, 2020). The CI presents granules with irregular geometric, as shown in Figure 1c. The same characteristic is present in carrageenan hydrogels prepared in the work of Silva *et al.* (2020).

Figure 1d-h shows the microgel structures after the ultrasound and spray drying treatments. The microstructure of MS and MI (Figure 1d and 1e) shows that the dry samples obtained from ultrasonic treatment showed more disrupted granules when compared to their native state. These results suggested that ultrasonic treatment tended to cause substantial changes in the physical nature such as deformation and loss of granule structure (Manchun *et al.*, 2012).

The surface morphology of the sago/ ι -car microgels

(Figure 1f-h) shows degrees of granule homogeneity as the ι -car concentration increases. These observations can be due to the density differences between ι -car and sago starch. More homogenous features can be observed in Figure 1h which can be attributed to reduced retrogradation that is usually observed when starch, particularly amylose, is mixed with carrageenan where the ι -car relates to the steric hindrance to prevent amylose gelation (Funami *et al.*, 2008).

Furthermore, the increase of ι -car concentration facilitates the presence of smaller particles in the sago/ ι -car microgels due to greater disperse ability, electrophoretic mobility and zeta potential values (Sharma *et al.*, 2021). Van der Waals forces alter the interparticle forces, which resulted in the clumpy and agglomerative appearance observed in Figure 1f-h. The Van der Waals forces have a direct relation with an increase in the hygroscopicity of the microgels (Khushbu *et al.*, 2020). This might be due to an increase in surface area, which in turn exposes more of the hydrophilic groups of the samples to form a covalent/hydrogen bond with water. Due to their high hygroscopicity, sago/ ι -car microgels are hence appropriate for use as thickeners.

3.4 Solubility of microgels

Based on the results in Table 4, there were significant differences between all samples. The solubility of TX was 44.2% which is almost similar to the finding by Yousif *et al.* (2012), who obtained solubility of modified corn starch at 44.85%. The solubility of NS which is 36.4% was almost similar to the findings by Du *et al.* (2020), who reported that the sago starch exhibited solubility around 36.10% and 38.63%. The granular integrity was generally considered as the main factor affecting the solubility (Vamadevan and Bertoft, 2015). The sago starch granules have some pitting and channels, which is conducive to water infiltrating into the granules for starch hydration and leaching as observed by SEM (Figure 1b).

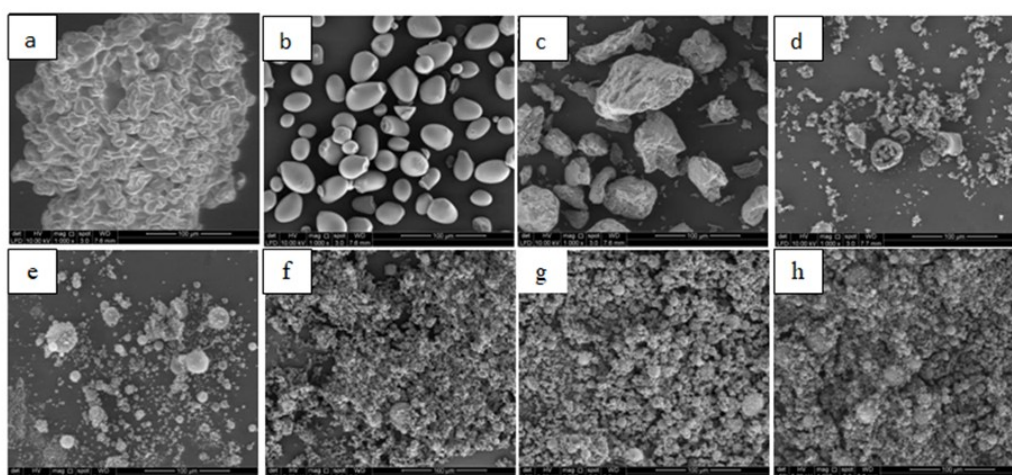


Figure 1. Micrographs of (a) TX; (b) NS; (c) CI; (d) MS; (e) MI; (f) MS25; (g) MS50 and (h) MS75. Magnification = 1000 \times .

Table 4. Solubility of control and microgel samples at different concentrations of ι -car.

| Samples | Solubility (%) |
|---------|------------------------|
| NS | 36.2±0.32 ^a |
| CI | 57.8±0.37 ^d |
| MS | 55.1±0.25 ^c |
| MI | 63.1±0.12 ^e |
| MS25 | 69.4±0.43 ^f |
| MS50 | 71.6±0.76 ^g |
| MS75 | 73.2±0.36 ^h |
| TX | 44.2±0.13 ^b |

Values are presented as mean±SD of triplicates. Values with different superscripts within the same column are statistically significantly different ($p < 0.05$).

Solubility increased dramatically after ultrasonication treatment of the sago starch and ι -car. For sago starch, the solubility increases significantly from 36.4% to 55.1%. This was due to the cavitation effects of ultrasound that disrupted the covalent bonds of crystalline structure and chains of sago starch. As a result, more water molecules may form hydrogen bonds with the free hydroxyl groups of amylose and amylopectin, increasing the solubility of starch (Tan *et al.*, 2021).

The solubility of the ι -car was determined as 57.8%. The ι -car forms a double helix conformation by hydrogen bonds in dispersion before heat or ultrasound treatment. A twist between residues of the anhydrous galactose molecule has three hydrogen bonds that stabilize the helix. Solubility of ι -car increased to 63.1% from 57.8% (MI) after being treated with ultrasonication. This may be attributed to the changing of the polymer conformation in ι -carrageenan, changing their tight crystalline structure into random coils where hydrophilic regions become more exposed to water molecules and solubility enhanced subsequently. The ultrasonic process has been confirmed to increase solubility in various kinds of starches (corn, potato, tapioca, and sweet potato) and polysaccharides (Iida *et al.*, 2008).

The solubility of sago/ ι -car microgels shows that the solubility of the microgels increases as the ι -car concentration increases. Maximum solubility (73.2%) was achieved with MS75 followed by MS50 (71.6%) and MS25 (69.4%), respectively. This can be attributed to the polyelectrolyte nature of carrageenan, which is more soluble in water than neutral hydrocolloids like starch because the negatively charged sulfate groups are hydrophilic. The high solubility of the sago/ ι -car microgels compared to the native sago starch would be beneficial in food/liquid thickener fabrication as they could facilitate starch gelatinization and gel formation and thus obtain a more stable thickening agent solution

(Tan *et al.*, 2021).

3.5 Rheological properties of microgels

Oscillatory measurements were carried out to investigate the physical properties of a sample, in terms of minor structural level changes, such as crosslinking, phase separation, and molecular aggregations, without damaging its structure (Mandala *et al.*, 2003). Storage modulus (G') and loss modulus (G'') are the important parameters to determine the viscoelastic behaviour of a sample during the application of stress. G' refers to the elastic energy stored in the sample while loss modulus (G'') refers to the energy that was dissipated from the structure and is sometimes called the viscous response (Saha and Bhattacharya, 2010). Higher values of G' than G'' showed the predominance of solid/elastic behaviour.

Based on Table 5, NS and TX showed high G' values but lower than CI. Starch is mainly made up of two main molecular components, namely amylose and amylopectin. There is a positive relationship between the viscoelastic properties of the sample (especially G') and the amylose content in starch (Sullivan *et al.*, 2010). This is because the linearity and extensive hydrogen bonding of amylose molecules require more energy to break these bonds (Sullivan *et al.*, 2010). Therefore, sago starch which contained high amylose, exhibited high G' values (Wattanachant *et al.*, 2002). On the other hand, the formation of a greater gel network from commercial thickener may also be caused by the modification process of starch involved in the production of commercial thickener. In contrast to starch, CI gel is more elastic and structured. For example, the storage modulus of CI is higher as classically observed in the literature (Hossain *et al.*, 1997). The weak gel-like behaviour of samples was characterized by a positive slope and the magnitude of G' was higher than G'' which is shown by a representative graph (Figure 2).

Table 5. Storage modulus (G'), loss modulus (G'') and $\tan \delta$ at 1 Hz frequency of all samples.

| Samples | G' (Pa) | G'' (Pa) | $\tan \delta$ |
|---------|---------------------------|--------------------------|------------------------|
| NS | 175.3±18.35 ^a | 57.3±12.72 ^a | 0.33±0.11 ^a |
| CI | 864.7±16.60 ^c | 144.9±14.36 ^b | 0.17±0.01 ^a |
| MS | 247.3 ±12.12 ^b | 75.27±10.03 ^a | 0.30±0.06 ^a |
| MI | 1185.0±15.55 ^f | 210.5±12.47 ^c | 0.18±0.01 ^a |
| MS25 | 207.0±12.39 ^{ab} | 55.37±14.09 ^a | 0.27±0.08 ^a |
| MS50 | 318.8±12.40 ^c | 69.87±9.88 ^a | 0.22±0.02 ^a |
| MS75 | 478.9±17.97 ^d | 71.84±11.32 ^a | 0.15±0.03 ^a |
| TX | 334.7±11.16 ^c | 63.21±11.33 ^a | 0.19±0.03 ^a |

Values are presented as mean±SD of triplicates. Values with different superscripts within the same column are statistically significantly different ($p < 0.05$).

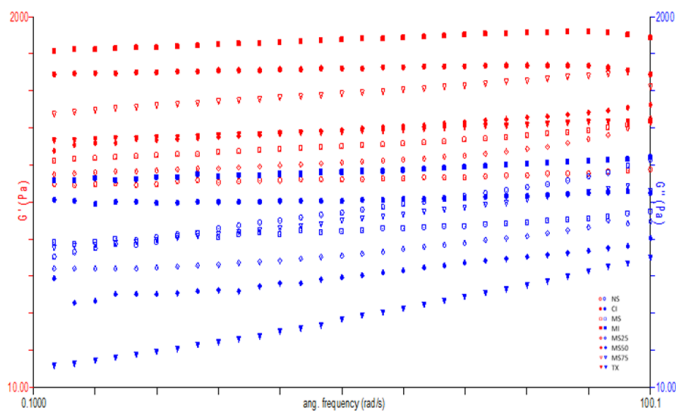


Figure 2. Plot of G' and G'' (Pa) against frequency (rad s^{-1}) of all samples including commercial thickener.

Furthermore, ultrasound increased the storage, G' , and loss, G'' , modulus of all samples which indicates that they have formed stronger gels after the treatment. This showed that sonication enhances the degradation of starch granules by cavitation forces and therefore granules become more permeable to water which leads to higher viscosity (Kaur and Gill, 2019). In addition, ultrasound irradiation disrupts carrageenan macromolecules and leads to full exposure and accessibility of the hydrophilic groups of polymers to water, producing a stable viscous system (Azizi and Farahnaky, 2016).

The contribution of ι -car on gel properties of sago/ ι -car microgels at various concentrations of ι -car was also studied. The changes in magnitudes of G' after the addition of ι -carrageenan were greater than changes in G'' . This means that the addition of different concentrations of ι -car largely influenced the elastic property of sago/ ι -car microgels. The G' values of sago/ ι -car microgels were higher than that of the gels composed of sago starch without ι -car. The G' values of MS75 were higher than those of MS50 and MS25. This could be due to the strong interaction between ι -car and sago starch. This was in agreement with the study of Jobling (2004) who reported that strong particle-particle interactions and the network structure in a stabilized form resulted in a larger value of G' value. Owing to the relatively high G' , the sago/ ι -car microgels could be structurally deformed during the eating process more easily compared with sago starch alone.

The trends of gel strength are illustrated by differences in loss tangent ($\text{Tan } \delta$) as shown in Table 5. The $\text{Tan } \delta$ value of <1 means the sample is more elastic than viscous whereas $\text{Tan } \delta > 0.1$ indicates the sample behaved like a weak gel (Torres et al., 2017). Besides, the strength of the gel is related to the $\text{Tan } \delta$, the lower the value of $\text{Tan } \delta$, the higher the strength of the gel. Since the $\text{Tan } \delta$ values were in the range of 0.1 - 1.0, all the samples were considered safe-swallow food meant

for dysphagia (Ishihara et al., 2011). The results revealed that there was slight variation for $\text{Tan } \delta$ after sonication and the addition of ι -car which showed lower $\text{Tan } \delta$ values compared to non-sonicated samples, NS and CI alone. The results of $\text{Tan } \delta$ were in accordance with the findings of G' and G'' obtained in this study. Therefore, it may be concluded that the sonicated samples with ι -car showed higher strength of gel behaviour.

3.6 Texture profile analysis

Mechanical properties of the developed microgel were determined using texture profile analysis (TPA). TPA is also known as the two-bite test as the sample was compressed twice using the suitable probe (Yusof et al., 2019). The results of the textural properties of microgels are presented in Figure 3.

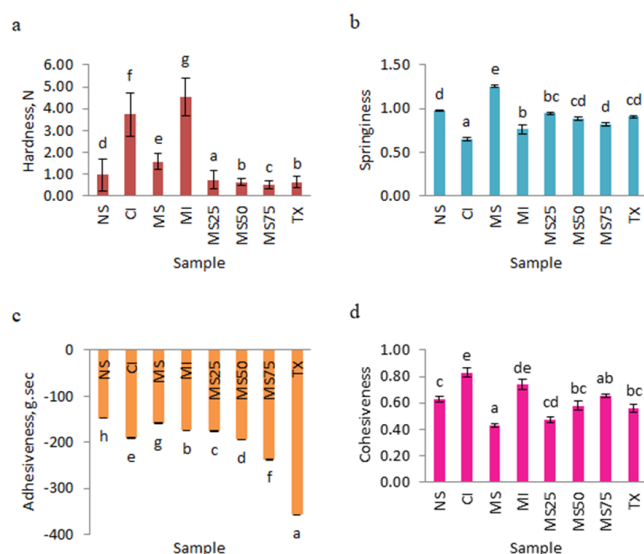


Figure 3. Texture profile analysis (TPA) parameters for MS, MI, MS75, MS50, MS25 and TX: (a) hardness, (b) springiness, (c) adhesiveness and (d) cohesiveness. The results are average of two independent experiments. Bars with different notations are statistically significantly different ($p < 0.05$).

3.6.1 Hardness

Hardness is the most significant parameter for TPA analysis. It is a factor that is used to evaluate the mouthfeel and is defined as the force required to attain a given deformation (Garrido et al., 2015).

The textural properties of the microgel samples including commercial thickener, TX, were determined using a texture analyzer and the results are presented in Figure 3a. The low values of hardness of TX corresponded to the amylose content of modified corn starch. Modified corn starch has a lower amylose content compared to sago starch which contributed to a decrease in the hardness of the sample.

The hardness of the MS was 1.55 N and was

significantly higher compared to the NS with 0.94 N. The increase in hardness of the sago microgels might be ascribed to maximum swelling of the starch granules due to partial disruption of the starch structure. Additionally, this might be due to mechanical vibration, and thermal and ultrasonic cavitation effects, the intramolecular hydrogen bonding of sago starch can be broken; thus, its molecular structure becomes loose and molecular winding nodes are reduced (Luo *et al.*, 2008). This result was in agreement with the studies conducted by Herceg *et al.* (2010) and Nie *et al.* (2019).

The hardness of CI was determined at 3.73 N. The gelation mechanism is still controversial, and the domain model has been suggested to explain gelation in terms of coil-to-helix transition followed by aggregation of domains of double helices α -car gels consisting of double helices with little or no aggregation, which renders them flexible and soft despite its high hardness value (Tischer *et al.*, 2006). Ultrasonication causes a significant increase in the gel strength of α -car to 4.51 N. These results are in accordance with solubility results.

On the other hand, the hardness of sago/ α -car microgel samples increased with increases in α -car concentrations but was lower than in other samples. MS50 with 0.63 N, had the most similar hardness with the commercial thickener, TX, with 0.61 N. The presence of α -car altered the gel structure of sago starch, in which the α -car (helix) interacts with leached amylose (helix) by hydrogen bonds, producing a stronger gel. Moreover, this can be attributed to the high concentration of α -car as they wind around each other to form double helical zones causing a more rigid network resulting in higher hardness (Chaplin, 2012). This observation was also noted in α -car and its interaction with other polysaccharides (Al-Baarri *et al.*, 2018).

3.6.2 Springiness

Springiness, also known as elasticity, is a perception of gel "rubberiness" in the mouth and is a measure of how much the gel structure is broken down by the initial compression (Leon *et al.*, 2018). High springiness gel is broken into a few large pieces under TPA compression whereas low springiness gel breaks into many small pieces.

According to Figure 3b, NS had a value of springiness higher than TX which was 0.98 and 0.91, respectively. This was attributed to the amylose contents of sago starch which was found higher than that of corn starch (Du *et al.*, 2020). The high amylose content caused the susceptibility to retrogradation and higher elasticity of sago starch pastes (Torres *et al.*, 2017), which is conducive to gelling. Furthermore, sago starch

is rated better to be used as an ingredient in pie filling ingredient and making pieces of bread, as it scored higher than cornstarch filling in terms of firmness, springiness, and overall evaluation (Hirao *et al.*, 2018). Springiness of CI was determined at 0.65 which is significantly lower than NS and TX ($p < 0.05$). Carrageenan is suitable to be used in TMF since it has less springy gel properties that will break down more easily during mastication (Leon *et al.*, 2018).

On the other hand, ultrasound increases the springiness of the gels. The springiness of MS and MI increased from 0.98 and 0.65 to 1.25 and 0.76, respectively. Cavities and microbubbles would be created in the solution as a result of the ultrasonic treatment. When microbubbles spread, they generate high energy that is subsequently transformed into high temperatures and pressures. In starch, this process led to polymer breakdown, such as amylopectin degraded into amylose, which is attributed to inducing more extensive intra and/or inter-molecular connections and results in a springier texture.

The springiness of sago/ α -car microgels was significantly ($p < 0.05$) affected by increasing α -car (from 1.0 g to 3.0 g). These results are in accordance with the hardness of gels so that along with decreases in the hardness, the gel springiness also decreases. From the results, it is obvious that the less springy gel structure imparted by sago/ α -car microgels compared to NS and CI alone would help to produce easy-to-swallow food.

3.6.3 Adhesiveness

Adhesiveness, also known as stickiness, indicates the work required to overcome the attractive forces between the surface of the food and the surface of the material with which the food comes into contact such as the tongue, teeth, and palate (Mutlu *et al.*, 2018). The area under the curve with a negative force that is obtained between cycles is referred to as adhesiveness.

The highest adhesiveness was recorded for TX with -357.92 g.s followed by CI and NS with -190.03 g.s and -148.03 g.s, respectively (Figure 3c). The high adhesiveness value of TX gels could be partially due to more amylopectin of modified corn starch. Amylopectin has higher adhesion and water absorption than amylose (Hunt *et al.*, 2009). Modified corn starch is rich in amylopectin, while sago starch is rich in amylose. Therefore, the TX has higher adhesion and water absorption than NS, which enhances the adhesiveness of the gel. This result was in agreement with Du *et al.* (2020) who reported that sago starch has a low adhesiveness compared to corn starch, potato starch and mung bean.

The adhesiveness in microgels was lower than in their original samples. This may be attributed to the degradation of leached starch. On the other hand, the addition of ι -car increased the adhesiveness of the sago/ ι -car microgels when its concentration increased from -175.84 g.s (1.0 g) to -194.09 g.s (2.0 g) and -237.58 g.s (3.0 g), respectively. Less adhesive food properties are desired in dysphagia diet management which indicates that it might need less time withdrawing from the palate and the teeth and was better for establishing a bolus that supported the swallowing (Steiner *et al.*, 2003). Adhesive foods are associated with an increased choking risk and require increased lingual effort to propel them into and through the pharynx (Suebsaen *et al.*, 2019). As a result, adhesive food textures should be avoided in frail elders and people who have difficulty swallowing (Suebsaen *et al.*, 2019).

3.6.4 Cohesiveness

The rate at which the material deforms under mechanical enforcement which is related to the strength of the internal structure and difficulty in breaking down the internal bonds is measured as cohesiveness (Hamedi *et al.*, 2018). Figure 3d shows that all samples possess almost similar cohesiveness values ranging from 0.43 to 0.74. MS50 with a value of 0.58 was the closest to the value of a commercial thickener, TX (0.56), which has been proven to be safe for dysphagia diet. The ultrasonication and addition ι -car affected gel cohesiveness significantly ($p < 0.05$). According to Zendeboodi *et al.* (2019), the cavitation impacts of ultrasound waves are responsible for improving the samples' texture integrity. In addition, Laustsen (2011) claimed that ι -car is known to perform an elastic and cohesive gel network. Therefore, the results suggest that the internal structures of all samples have higher integrities and may be more difficult to break during the first compression. Cohesive bolus will not disintegrate easily into pieces in the mouth, thus avoiding the scatter of small particles in the pharyngeal phase of swallowing, hence easy to swallow (Rao and Cooley, 1992).

4. Conclusion

Ultrasound and spray drying appeared to be effective in fabricating microgels below 100 μ m. Micrograph analysis has shown an obvious impact of ultrasound on the structure and size of starch and ι -car in which ultrasound treatment ruptures and mechanically damages their structures by the collapse of cavitation bubbles. The increase in solubility of sago starch, ι -car and the microgels is associated with the water absorption capacity. The higher facility for water entrance is due to

ultrasound disruption of granule which leads to a higher water uptake and retention. The dynamic rheological data for storage modulus (G') and loss modulus (G'') as a function of frequency at 37°C showed that all samples displayed weak gel-like behaviour. G' was higher than G'' throughout the frequency range, indicating that there was a predominance of the elastic over the viscous behaviour in all samples, as is typically observed for gelled structures. Values of $\tan \delta$ in the range of 0.1–1 have been suggested as the rheological criterion for safe-swallow foods destined for dysphagia patients.

The presence of ι -car in sago starch increased the strength of sago/ ι -car gels. The improvement in gel strength of sago/ ι -car was more profound for incorporating ι -car at higher concentrations. These characteristics suggest that sago/ ι -car microgels could contribute to forming a bolus with a weak gel consistency, easy to masticate and swallow. The results of textural measurement further confirm the rheological properties of sago/ ι -car as a thickener. In addition, this study also provides a strong basis to explore the potential use of sago/ ι -car gels in the development of TMF.

Conflict of 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.

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