Siam orange (*Citrus nobilis* L.) nectar characteristics with variations in stabilizer and sucrose level


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**Abstract**

The Siam orange (*Citrus nobilis* L.) has low economic value because of its sour taste and unattractive appearance. However, processing the fruit into nectar can increase its value. Nectar production requires the addition of sugar and the appropriate stabilizer for consumer acceptance. This research aimed to study the ideal combination of sucrose and stabilizer in Siam orange nectar production. A factorial completely randomized design was employed with two factors, the concentration of sucrose (10%, 15%, and 20%) and the type of stabilizer (CMC, carrageenan, pectin). As more sucrose was added to the juice, the moisture content decreased and the volume of soluble solids increased. The type of stabilizer only affected nectar pH and viscosity. CMC produced the highest nectar pH, followed by carrageenan and pectin. However, based on the results of the analysis of all parameters using the effectiveness index, carrageenan was the best stabilizer. Orange nectar with 20% sucrose and 0.5% carrageenan contained 24.2% total soluble solids, 76.6% moisture content, 7.9% reducing sugar, 23.5% vitamin C, and 3.38% crude fibre, with a viscosity of 109 cP and pH of 3.7. The product’s sensory characteristics were an orange colour, a sweet, citrus aroma, and a slightly thick texture. Additional research on the storage stability of this nectar formulation is necessary.

1. **Introduction**

There is high potential genetic diversity in Indonesia’s citrus fruits, and the country’s orange production is substantial, reaching 2,510,442 tons in 2018 (Badan Pusat Statistik, 2020). One variety of orange grown in Indonesia is the Siam orange (*Citrus nobilis* L.), which constitutes approximately 80–85% of citrus production in Indonesia (Hanif and Zamzami, 2012). However, they have a sour taste, and their greenish or yellowish skin colour is unevenly distributed. Currently, consumers tend to prefer sweet oranges (*Citrus sinensis*) for direct consumption, demonstrating less interest in Siam oranges, particularly sour ones (Sadeli and Utami, 2013). The compositions and properties of oranges vary by type (De-Carvalho et al., 2020), and orange fruits can be easily damaged, making their shelf life relatively short (Arshad et al., 2019). These factors indicate that appropriate technology is needed to increase the economic value of Siam oranges, one method of which is to process the oranges into nectar (Lozano et al., 2020).

Nectars are fermented or unfermented products obtained by adding water to fruit juice, concentrated fruit juice, fruit puree, or concentrated fruit puree, or a mixture of these that conform to the specifications and may contain up to 20% added sugar (De Sousa et al., 2010). Depending on the fruit type, the minimum fruit content in fruit nectar is approximately 25 to 50% (Krumreich et al., 2018), but nectar can be 10–99% fruit pulp or juice (Najafabadi et al., 2020). Highly acidic and fibrous fruits are often transformed into nectar. Adding water makes the nectar more drinkable. Nectar production may also involve heating and filtration processes, possibly affecting the concentration of bioactive compounds. Nectar turbidity is associated with colloidal suspensions in varying amounts, and the solid content is usually 5 to 20% (w/w) (Krumreich et al., 2018). The same kind of fruit with different compositions require different and precise nectar formulations (Bahlol et al., 2018).

The production of fruit nectar has been studied for multiple fruits, such as mango (Lozano et al., 2020), soursop (Tran et al., 2020), guava (Krumreich et al., 2018), peach (Nedić-Tiban et al., 2003), and mixed fruits.
(Bahlol et al., 2018). Furthermore, Stella et al. (2011) investigated the antioxidant activity of commercial orange nectar of Citrus reticulata, and Matiashe et al. (2014) developed nectar from lemons (Citrus limon). However, the production and composition of nectar from Siam orange (Citrus nobilis L.) have not been investigated, and this research is necessary to increase the fruit’s economic value.

Adding sucrose to nectar increases the sweetness and enhances the flavour (Nedić-Tiban et al., 2003). Sucrose is a sweetener that is colourless and soluble in water. It can play an important role in increasing the acceptance of certain foods (Brochier et al., 2019), and its addition will help develop a product with the desired properties. For example, working with guava, Khan et al. (2014) found that different concentrations of sucrose produced guava bars with different characteristics. Furthermore, Ferrarezi et al. (2013) stated that consumers’ desire to buy orange juice and nectar is influenced by sensory properties, in addition to price and labelling. Hoffmann et al. (2017) developed Butia fruit nectar using a xanthan gum stabilizer and variable amounts of added sucrose, and the authors found that samples with 14°Bx sucrose had the best sensory characteristics, including colour, aroma, flavour, sweetness, and overall acceptability.

Stabilizers increase the nectar’s viscosity, physical consistency, and stability (Hoffmann et al., 2017). In the production of orange nectar, carboxymethylcellulose (CMC), carrageenan, and pectin can be used, and different stabilizers are appropriate depending on the nectar produced. CMC as sodium carboxymethyl cellulose salt provides good shape, consistency, and texture. It also acts as a water binder, thickener, emulsion stabilizer, and gum texture (Akkarachaneeyakorn and Tinrat, 2015). In the production of soursop and mango nectars, CMC is used as a stabilizer and produces nectar with greater viscosity than CMC in the production of dragon fruit velva (Basito et al., 2018). Pectin is a type of α-D-galacturonic acid carbohydrate biopolymer that contains methyl ester and can be extracted from fruit flesh and skin with an acidic solvent. It is often used as a stabilizer in foodstuffs, jelly ingredients, and films (Krumreich et al., 2018). When preparing guava nectar, pectin has demonstrated good viscosity generation and vitamin C retention during storage.

The objectives of this study were to examine Siam orange nectar’s physical, chemical, and sensory properties using different sugar concentrations, determine the physical, chemical, and sensory effects of an added stabilizer and determine the combination of sucrose concentration and stabilizer that produces the best Siam orange nectar.

2. Materials and methods

2.1 Materials

Siam oranges (Citrus nobilis L.) were purchased from a local market in Purwokerto, Indonesia. The stabilizers tested were carrageenan (kappa carrageenan, produced by CV Karagen Indonesia), CMC (Butterfly brand, purchased from Intisari, Purwokerto, Indonesia), and powdered pectin (low methoxy pectin, product of PT Chemindo Ekatama).

2.2 Experimental design

The research used a factorial completely randomized design with two factors: sucrose concentration (at 10%, 15%, and 20%) and three types of stabilizers (CMC, carrageenan, and pectin). The treatments were arranged factorially to obtain nine treatment combinations, and each treatment combination was repeated three times to obtain 27 experimental units. The data were analyzed by ANOVA (F-test). If the ANOVA revealed a significant effect, Duncan’s multiple range test (α = 5%) was performed.

2.3 Production of nectar

The orange nectar was produced according to Hoffmann et al. (2017), with modifications to the ingredient proportions and stabilizer type. The nectar was made by mixing water and pulp (1:4), sucrose, and stabilizer. The amount of sucrose added was 15%, 20%, or 25% (w/v). The stabilizer used was carrageenan, CMC, or pectin. Nectar samples (150 mL) were pasteurized in a 250 mL bottle for three minutes at 100±5°C. The nectar was cooled at 30°C until it reached 28°C, then analyzed.

2.4 Nectar sample analyses

Moisture content, reducing sugar content, soluble solids, crude fibre, vitamin C, viscosity, and pH were determined. Moisture content was determined by the oven method (AOAC, 2005). Soluble solids were measured using a refractometer (AOAC, 2005) and the pH was measured using a pH meter (AOAC, 2005). Reducing sugars were analyzed using the Nelson–Somogyi method, and viscosity was measured according to Steele et al. (2014). The crude fibre was analyzed using the gravimetric method (AOAC, 2005).

Total soluble solids (TSS) were measured with a
refractometer after calibration. Several drops of each sample were placed on a blue prism and the TSS (°Bx) read. The refractometer was calibrated after each sample. The reducing sugar was measured in 2 g of sample diluted with distilled water to 100 mL. From the solution, 1 mL was taken and then diluted with distilled water to 10 mL. To 1 mL of this second solution, 1 mL of Nelson’s reagent was added. The solution was heated in a water bath for 20 mins. The sample was cooled by holding the flask under running water, 1 mL of arsenomolybdate was added, the sample was shaken using a vortex, and 7 mL of distilled water was added. The absorbance was measured using a spectrophotometer at a wavelength of 540 nm. Reducing sugar content can be measured by converting the absorbance with a standard curve.

Viscosity was measured using a viscometer (Steele et al., 2014). A spindle rod was installed on a viscometer with a number that corresponded to the type of sample. The spindle rod was inserted into the sample in a glass beaker. The viscometer was turned on, and the viscosity was read after 5 mins using the viscometer scale. All values were converted to viscosity according to the speed and spindle used.

The crude fibre was analyzed by the gravimetric method (AOAC, 2005). To 2 g of sample in a 250 mL Erlenmeyer flask, 50 mL of 1.25% H₂SO₄ solution was added. The sample was heated for 30 mins, and 50 mL of a 3.25% NaOH solution was added. The sample was reheated for 30 mins in an 80°C water bath. The suspension was filtered through filter paper that had been oven-dried and weighed. The residue left in the filter paper was washed with 15 mL of hot 3.25% H₂SO₄ and 15 mL of boiling distilled water, then washed again with 15 mL of hot 96% ethanol. Sample washing was continued until the washing water was not acidic. The filter paper was dried in an oven at 110°C to a constant weight, cooled in a desiccator, and weighed. Crude fibre content was calculated based on the ratio between the dried residue and the initial sample weight.

The pH was measured using a pH meter (model Do700, Extech Instruments, Shanghai, China). Before use, the pH meter was calibrated using buffer solutions of pH 7 and pH 4. Then, the pH meter electrode was dipped into a sample of orange nectar until the value stabilized.

Vitamin C was determined using the titration method based on the reduction of the indicator 2,6-dichlorophenol. The sample (5 g) was added to a 100 mL volumetric flask, and distilled water was added to 100 mL. The filtrate was homogenized, filtered, and 25 mL was added to an Erlenmeyer flask with 1 mL of a 1% starch solution. The filtrate was then titrated with a standard 0.01 N iodine solution until a colour change occurred.

The sensory evaluation utilized the Meillgard method (De Sousa et al., 2010). Sensory analysis was performed, using test scores for taste, colour, aroma, thickness, and preference parameters on a scale of 1 to 5. The scales for taste were 1 = bitter and 5 = very sweet; for colour, 1 = light yellow and 5 = very orange; for aroma, 1 = not strong and 5 = very strong; for viscosity, 1 = not thick and 5 = very thick; for personal preferences, 1 = dislike and 5 = like very much. Twenty trained panellists were employed, and the Friedman test was used to analyze the organoleptic results. If there was an effect, a multiple comparison test with α = 5% was conducted. The orange nectar sensory test variables based on a numeric scale can be viewed in Table 1.

3. Results and discussion

3.1 Total soluble solids

The concentration of sucrose significantly affected the TSS of orange nectar. The greater the quantity of sucrose added, the higher the nectar TSS. The highest soluble solid value was 23.4°Bx, with the addition of 20% sucrose (Figure 1). Sucrose contains high TSS. According to Krumreich et al. (2018), the solid content of sucrose is 95%, and the more sucrose added, the greater the TSS. This result is consistent with the increase detected in sucrose content as the TSS in orange nectar increased (Stella et al., 2011).

The interaction between the sucrose concentration and the type of stabilizer had a significant effect on TSS (Figure 2). The highest TSS values were found with the addition of 20% sucrose and carrageenan as stabilizers. Carrageenan has higher TSS than CMC and pectin due to its high protein content of 3.4%, while CMC has no protein (Septianti et al., 2019). The 20% sucrose in this study provided higher TSS than the 10% and 15%, and

<table>
<thead>
<tr>
<th>Numeric Scale</th>
<th>Colour</th>
<th>Taste</th>
<th>Aroma (citrus flavour)</th>
<th>Viscosity</th>
<th>Preference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Light yellow</td>
<td>Not sweet</td>
<td>No citrus flavour</td>
<td>Thin</td>
<td>Dislike</td>
</tr>
<tr>
<td>2</td>
<td>Dark yellow</td>
<td>Little sweet</td>
<td>Little</td>
<td>Little thick</td>
<td>Like</td>
</tr>
<tr>
<td>3</td>
<td>Slightly orange</td>
<td>Slightly sweet</td>
<td>Slightly</td>
<td>Slightly thick</td>
<td>Slightly like</td>
</tr>
<tr>
<td>4</td>
<td>Orange</td>
<td>Sweet</td>
<td>Citrus flavour</td>
<td>Thick</td>
<td>Like</td>
</tr>
<tr>
<td>5</td>
<td>Very orange</td>
<td>Very sweet</td>
<td>Very strong</td>
<td>Very thick</td>
<td>Very like</td>
</tr>
</tbody>
</table>

Table 1. Numeric scale of attribute
in combination with carrageenan as a stabilizer, it produced orange nectar with the highest TSS, at 24.1°Bx. With 10% sucrose added with CMC, carrageenan, and pectin stabilizers, the nectar showed no significant differences in TSS, at 16.3, 16.4, and 16.8°Bx, respectively (Figure 1).

As free water was bound by a stabilizer, TSS increased. As more particles bind to stabilizers, the TSS increases, and the precipitate decreases. In the presence of a stabilizer, the suspended particles will be trapped in the system and will not settle under the influence of gravity (Krumreich et al., 2018). Prasetijo et al. (2017) found that the higher the sucrose concentration in ripe fruit, the higher the total dissolved solids. Sucrose and pectin play a role in increasing the total dissolved solids content.

The orange nectar in this study had TSS values of 16.3–24.1°Bx (Figure 2). The TSS of these nectars were higher than those found for lime (13–13.2°Bx), guava (14.1–16.2°Bx), and mango (11.94–12.45°Bx) nectars (Matiashe et al., 2014; Krumreich et al., 2018; Lozano et al., 2020). However, the TSS of orange nectar was lower than mango nectar (72–75.4°Bx) (Khan et al., 2014), as mangoes have more total solids than oranges.

### 3.2 pH

The concentration of sucrose did not affect the pH of the orange nectar significantly. The average pH values of the orange nectars with 10%, 15%, and 20% added sucrose were the same (3.7). This is because sucrose has a neutral pH, so it does not affect the pH when added to orange nectar. These results are consistent with Khan et al. (2014), who found that adding sucrose did not affect nectar pH.

The stabilizer type had an impact on the pH of the orange nectar, and nectars with CMC as a stabilizer had the highest pH (3.9), followed by carrageenan (3.7) and pectin (3.6), as shown in Figure 3. As CMC is an alkaline stabilizer, the orange nectar with added CMC will have a higher pH than those with carrageenan and pectin. Khan et al. (2014) noted that the pH of guava bars with the addition of CMC was slightly higher than that of those with added pectin. Our results are also consistent with Prasetyo (2014), in which the addition of CMC in the manufacture of red guava fruit drinks had a pH ranging from 5.3 to 5.9. Furthermore, adding 0.5% CMC to cashew syrup resulted in a pH of 5.34 (Manoi 2006). According to Simamomar and Rossi (2017), adding 0.5% pectin in the manufacture of mango apple (Sonneratia caseolaris) jam resulted in a pH of 3.20. Therefore, applying pectin to citrus fruit nectar will result in a more acidic pH than citrus nectar with CMC or carrageenan.

The addition of CMC will cause the pH to increase because CMC is a salt of a strong base and a weak acid, so the resulting solution will have a high pH (Marchelina et al., 2020). In addition, there are large amounts of hydrocolloids in CMC to increase the pH (Malaka et al., 2017). The addition of pectin will increase the acidity so that the pH decreases. This process occurs because, during nectar production, pectin is hydrolyzed into pectinate and pectic acid so that the more pectin is added, the higher the acid produced and the lower the pH (Tran et al., 2020).
The interaction between the stabilizer type and sucrose concentration did not significantly affect the pH of the nectar. The pH of the citrus nectar in this study was 3.6–3.9, which was lower than the 4.2 found by Matiashe et al. (2014), who studied lime nectar production.

The pH of a food product is extremely important, as it limits the growth of disease and spoilage bacteria. According to Xiang-Ng and Kuppusamy (2019), heat treatment also promotes the stability of ascorbic acid because this vitamin is more stable at acidic pH. There is no pH standard for orange nectar, although the standard range for orange juice, according to the FDA Center for Food Safety and Applied Nutrition (2008), is 3.3 to 4.19. Based on this FDA standard, the pH of orange nectar from 3.6–3.9 would meet FDA standards.

3.3 Viscosity

The type of stabilizer had a significant impact on the viscosity of the orange nectar (Figure 4). CMC produced the highest viscosity (195 cP), followed by carrageen (119 cP) and pectin (26 cP). The high viscosity of orange nectar with added CMC is due to the dispersal of CMC in the fluid phase, which binds large amounts of water and forms a gel framework, preventing the free movement of water molecules (Akkarachaneeyakorn and Tinrat, 2015). According to Utomo et al. (2014), the viscosity of many water molecules is increased because the crosslinks formed by the helical arrangement and their interactions are trapped within the three-dimensional structure. The water previously free to move outside the granules can no longer do so because it is absorbed and bound to the CMC granules, increasing viscosity (Siskawardhani et al., 2013).

The addition of carrageenan will cause decreased stability, especially at high temperatures. Carrageenan is capable of forming a gel of polymer chains that form a three-dimensional mesh. This network captures or mobilizes water therein and forms a strong and rigid structure (Sharma et al., 2017). However, the tissue formed by carrageenan is weaker than CMC, so the viscosity of nectar added with carrageenan was lower than the viscosity of nectar added with CMC. According to Penjumras et al. (2019), adding more CMC rather than carrageenan to the dough will increase its viscosity.

Compared to other stabilizers, pectin produced the lowest orange nectar viscosity. Khushbu and Sunil (2018) stated that mayonnaise had a lower viscosity with added pectin than CMC. Similarly, Tran et al. (2020) found that adding pectin in low concentrations resulted in a reduction in sour sop nectar viscosity.

The interaction between sucrose concentration and stabilizer type did not significantly influence the viscosity of the orange nectar. According to the guidelines of the National Dysphagia Diet (Steele et al. 2014), the viscosity of orange nectar should be 51–350 cP. The viscosity of orange nectar produced in this study was 21.7–221.7 cP (Figure 5). Orange nectar with pectin stabilizer does not fulfil these requirements, as its viscosity is less than 50 cP, while orange nectar with carrageenan and CMC stabilizers fulfilled the dysphagia diet criterion.

3.4 Reducing sugar content

Sucrose concentration and stabilizer type, and the interaction between them, did not affect sugar reduction significantly. Figure 6 shows that the reducing sugar content was 5.7–11.1%. No studies have investigated the reducing sugar levels of nectar, but Nuraeni et al. (2019) found an 8–10% lower sugar content for fruit juice. Sugar reduction in citrus nectar is similar to that in guava bars, at 839–1138 (Khan et al., 2014).

3.5 Vitamin C content

Sugar concentration and type of stabilizer had no significant effect on the orange nectar ascorbic acid levels, which were 21.1–28.2 mg/100 mL (Figure 7).
According to Wariyah (2010), Siam oranges contain 20–60 mg/100 mL of vitamin C. The proportion of juice to the water in the manufacture of orange nectar is the same (4:1), so the levels of vitamin C could be compared.

Krumreich et al. (2018) found that ascorbic acid was higher in guava nectar (5.2%), and Kumalasari (2015) found that it was 9.7% in papaya-pineapple juice. Khan et al. (2014) also stated that the type of stabilizer did not affect ascorbic acid levels.

3.6 Crude fibre content

The analysis of variance showed that the type of stabilizer had a significant effect on the crude fibre content of citrus nectar. Moreover, the sucrose concentration and the interaction between sucrose and stabilizer had no significant effect on the crude fibre content of citrus nectar.

The crude fibre content of orange nectar with added pectin was 3.27%, carrageenan 3.17%, and CMC 2.57%. The differences are due to the different characteristics of each type of stabilizer. These results are consistent with Herlina (2020) that adding 0.5% carrageenan to fruit leather of Chrysophyllum cainito resulted in higher fibre content (6.81%) than adding 0.5% CMC (5.16%). Mangrove apple jam with 0.5% pectin had a crude fibre content of 2.08%, according to Simamomar and Rossi (2017).

Fibre is composed of carbohydrates that cannot be hydrolyzed by strong acids or bases under controlled conditions (Palafox-Carlos et al., 2011). Anderson et al. (2009) reported that there are two fibre classes, namely water-soluble fibre and insoluble fibre. Soluble fibre includes pectin, gum, and carrageenan. Meanwhile, the fibres that are not soluble in water are cellulose, hemicellulose, and lignin. Penjumras et al. (2019) stated that CMC contains water-soluble fibre, which will increase the fibre content in the product. However, the total dietary fibre content of CMC is 74 g/100 g, which is less than that of carrageenan (83.62 g/100 g) (Muzaifa 2006). In this study, the product with the highest crude fibre content was the product with added pectin. Pectin is a soluble dietary fibre that the higher the concentration of pectin added, the higher the soluble fibre content (Dickinson 2009).

3.7 Sensory evaluation

Sensory testing of six products was performed on those nectar products with added CMC and carrageenan. Nectar with pectin as a stabilizer was not tested because it did not comply with viscosity standards.

Colour is an important element that helps to determine the acceptance level of a specific food. If a product has good nutritional value, good taste, and attractive colour, it will interest the public (Akkarachaneeyakorn and Tinrat, 2015). In this case, the orange nectar colour scored 3.84–4.24, which means the nectar had a yellow colour, and the difference between treatments was not significant. The colour of oranges results from the presence of beta carotene.

Taste is a major parameter in consumer food selection since it is a quality attribute capable of determining the level of consumer acceptance of products (Chakraborty et al., 2020). Different perceptions of food products are also determined by taste. The taste of the orange nectar had a score of 3.24 (slightly sweet)–4.4 (sweet), which was influenced by the combination of stabilizer type and sucrose concentration. As shown in Figure 8, the highest taste score was for the nectar with the addition of 20% sucrose and carrageenan stabilizer. The addition of a high amount of sucrose and carrageenan increased the sweetness. Similarly, Marzelly et al. (2018) found that adding sugar and carrageenan increased the sweetness of fruit leather.

Aroma is one of the sensory parameters used by consumers when selecting food (Tuan-Azlan et al., 2020). Most people will first smell the product before tasting it. There was no significant difference in the
aroma of the orange nectar between the treatments, with scores of 2.60–3.24, indicating a mild citrus aroma. Marzelly et al. (2018) proposed that sugar and carrageenan form a network of matrices that traps and produces the characteristic citrus aroma.

Sensory texture tests were performed on the product viscosity. Texture considerably influences the images of foods and is sometimes more important than flavour (Chakraborty et al., 2020). The sensory score for the viscosity of the orange nectar was 2.00–3.56, differing significantly by treatment effect. The highest viscosity (3.56) was in the nectar with the addition of 20% sucrose and CMC stabilizer. The sensory viscosity test results are consistent with viscometer results. The high viscosity of orange nectar with CMC was due to the dispersed CMC during the fluid phase, which binds large quantities of water and creates a gel-frame, preventing water molecules from moving freely (Akkarachaneeyakorn and Tinrat, 2015).

The panellists’ overall taste verdict was the final evaluation, combining the parameters of colour, aroma, taste, and texture. The preference score for citrus nectar was 2.72–3.36, influenced by the sucrose concentration and the type of stabilizer. The highest preference (3.36) was for the orange nectar with 20% saccharose and carrageenan stabilizer (Figure 8).

Figure 8. Sensory properties of orange nectar on various stabilizers and sucrose levels (A: 10% sucrose and CMC; B: 10% sucrose and carrageenan; C: 15% sucrose and CMC; D: 15% sucrose and carrageenan; E: 20% sucrose and CMC; F: 20% sucrose and carrageenan).

4. Conclusion

Squeezed oranges can be processed into nectar with the addition of sucrose and a stabilizer. The more sucrose added to the nectar, the lower the moisture content and the more soluble solids produced. The type of stabilizer had a major impact on the pH and viscosity of orange nectar, and the best stabilizer was found to be carrageenan. The preferred citrus nectar was made with the addition of 20% sucrose and carrageenan. The favoured product had the sweetest taste, an orange colour was slightly dense, and had a slightly orange aroma. The preferred nectar had a moisture content of 76.6%, total dissolved solids of 24.2%, pH of 3.7, a viscosity of 109 cP, vitamin C of 23.5%, reducing sugar of 7.9%, and crude fibre of 3.38%. Squeezed oranges of low economic value can be made into orange nectar by adding 20% sucrose and 1% carrageenan. The processing of squeezed oranges into citrus nectar is expected to increase its shelf-life and economic value. More research on its stability during storage is needed.

Conflict of interest

The authors declare no conflict of interest.

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