

PHYSICOCHEMICAL AND SENSORY PROFILES OF VANILLA ICE MILK IN INDUSTRIAL AGEING TANKS USING TWO DIFFERENT AGITATOR SPEEDS

Muhamad Rizal, Siti Aminah, Muhammad F. Kurniawan, Aminullah*

*Djuanda University, Faculty of Engineering and Halal Food Science,
Department of Food Technology and Nutrition, Jl. Tol Ciawi No. 01,
Ciawi, Bogor, Indonesia*

*Corresponding author: aminullah@unida.ac.id

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Abstract: Ice milk is one of the low-fat ice cream-based dairy products which requires good both physicochemical and organoleptic profiles. The objective of the research was to study the effect of ageing time and agitation speed in industrial-scale on physicochemical and sensory properties of vanilla ice milk. This research using factorial completely randomized design consisted of an ageing time of 4, 24, and 48 hours and an agitation speed of 35 rpm and 45 rpm with three replications. Fat globule diameter, viscosity, phase separation, total solids, fat content, and sensory profile using different from control tests and quantitative descriptive analysis (QDA®) were analyzed in this research. The results showed that the ageing time, and the agitation speed did not have a significant effect on viscosity, total solids, and fat content of ice milk, but have little effect on fat globule diameter and phase separation. Ageing at 48 hours and agitation speed of 45 rpm slightly decreased the separation phase in ice milk. In addition, the overall attribute of 4 hours aged ice milk with a speed of 35 rpm was significantly different from 4 hours aged ice milk of 45 rpm. Based on the QDA®, the difference was only in the coarse texture attribute from ten attributes. This minor difference reflected that ice milk in these industrial tanks resulted in uniform and consistent product quality.

Keywords: *ageing time, agitation speed, low-fat ice cream, industrial scale, physicochemical stability, sensory analysis*

INTRODUCTION

Low-fat ice cream like ice milk is now becoming increasingly popular in the community because of its low-fat content compared to full fat ice cream. However, there are challenges for the ice cream industry to achieve the same creamy texture as the full-fat version. With less fat, the ice cream usually contains more water, so it has a larger ice crystal [1]. Smet *et al.* [2] said that in particular, fat contributes significantly to the properties of ice cream during mixing and freezing, which can increase the melting resistance and texture smoothness characteristics. Ice milk was similar to ice cream; however, the texture was not dry and contained a little milk fat [3]. Ice milk was a frozen product containing 2 - 7 % fat, no less than 11 % of total solids (TS), and no less than 155.8 kg·m⁻³ of TS. Fat substitutes such as protein (whey protein) or carbohydrates (modified starch) were generally added to ice milk for improving the quality of low-fat ice cream [4].

Goff [5] described the various steps in the process of making ice cream, including pasteurizing, homogenizing, ageing, freezing, and hardening the ice cream, which contributes to the development of ice cream structures. One of them is the application of a minimum ageing time of four hours or more at 2 - 4 °C between mixing and freezing processes. This treatment allows the hydration of milk proteins and stabilizers, crystallization of fat clots, and rearrangement of the membrane, production of smoother textures which impact to better quality products. Mixes that were not aged will result in weak resistance, very wet when extruded, and show variable mixing ability. Alkalin *et al.* [6] confirmed at the pilot plant scale that the ageing process improved the softness of shape and texture, resistance to fast melting rate, and the case of whipping whole ice cream products, where ageing process longer than 4 hours can give better results with a high-fat mixture. In addition, the stirring speed in the tank during the ageing process is expected not to have a detrimental effect on the physical-chemical and organoleptic characteristics of ice milk. Clarke [3] stated that the mix was maintained at 0 – 4 °C, and it was stirred gently with minimum agitation for a specific time to avoid heating from the mix. Research on industrial scale ice milk has not been well documented which is very interesting so that this research can make a significant contribution to the ice cream industry related to product quality both from physicochemical and sensory properties.

This study aims to study the effect of ageing time and agitation speed in two industrial-scale storage tanks of 4000 L on the physicochemical stability of ice milk such as fat globules, viscosity, fat content, total solids, and phase separation. In addition, sensory analysis is also performed on ice milk using the Quantitative Descriptive Analysis (QDA®) tool with several parameters.

MATERIALS AND METHODS

Materials

The materials used in this study were mix ice milk, aquades, fiberglass paper pad of #200150 (CEM Corporation), neusal solution, immersion oil, and water. Then, the tools were 4000 L ageing tank, microwave moisture analyzer type HClO₃ (Mettler Toledo),

centrifuge type SuperVario-N (Funke - Dr. N. Gerber Labortechnik GmbH), milk butyrometer 12 % (Funke - Dr. N. Gerber Labortechnik GmbH), analytical balance type ME-T (Mettler Toledo), water bath type WBU 45 (Mettmert), hot plate, and stirrer, viscometer type Brookfield LV DV-I Prime (Ametek Brookfield). and spindle set, microscope type BA210 (Motic) and Motic Image Plus application version 2.0 ML, slide and cover slide, stainless steel cup, plastic cup, beaker glass, stirring rod, volumetric flask, measuring cup, dropper, and other glassware.

Methods

This research has consisted of two stages, namely the first stage of making ice milk and the second stage of the primary research, which can be seen in Figure 1.

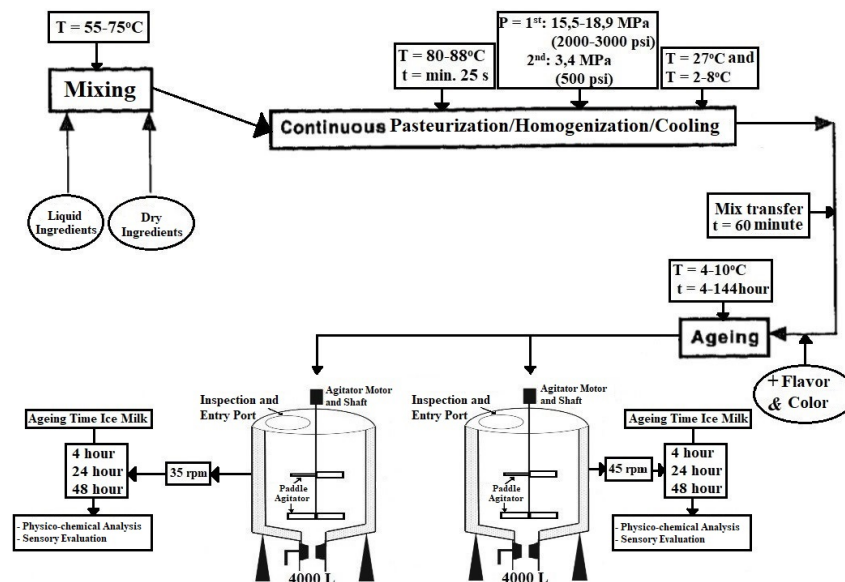


Figure 1. Process diagram of ice milk research on an industrial scale (modification of Goff [5])

Making ice milk

At this stage, the mixing of liquid (water and fat) and solids (milk solid non-fat, sweetener, stabilizer, and emulsifier) ingredients was based on the specifications of the company. Then, it was pasteurized using high-temperature short time technique, followed by two stages of homogenization, then cooled to room temperature (27 °C) and continued to cold temperature (2 - 8 °C), then the mixture was transferred to the ageing tank of 4000 L while the heat-sensitive ingredients (flavor and color) were added. The mixture temperature was maintained at 4 - 10 °C within a specified period and speed of agitation.

Primary research

At this stage, treatment was carried out using two factors, namely ageing times of 4 hours, 24 hours, and 48 hours (A1, A2, and A3) and agitation speeds of 35 and 45 revolutions per minute (rpm) (B1 and B2) for ice milk samples by applying factorial completely randomized design with three replications. Then the physicochemical properties of viscosity, total solid, fat content, phase separation, and fat globule were analyzed. In addition, the sensory analysis of difference from control test and the quantitative descriptive analysis with an unstructured scale, if there was a difference in the Different From Control (DFC) test, was conducted in the research.

Product Analysis

Viscosity analysis

First, the mixture temperature was measured and then heated in a water bath until it reached a temperature of 25 ± 2 °C. Mixture viscosity was measured using Brookfield LV DV-I Prime Viscometer with spindle no. S61 at a speed of 60 rpm for 20 seconds. The experimental data were adjusted to the law model in which the actual viscosity μ_{app} at shear stresses of 20 s^{-1} [7].

Total solid analysis

Total solid analysis on ice milk was using microwave method [8]. The fiberglass paper pad of #200150 media sample (CEM Corporation) was inserted into an opened Microwave Moisture Analyzer (MMA) Mettler Toledo. Then the cover was closed and held until the pad dryer has changed to zero settings (internal balance). Then, the sample was applied little by little as much as ± 0.5 gram on pad paper. The MMA has closed again where it was analyzed at a temperature of ≥ 100 °C for 5 minutes.

Fat content

Fat content analysis in this research was using Gerber-Neusal method [9]. Preparation of Neusal solution was carried out by weighing 60 g of trisodium citrate, and 60 g of sodium salicylate in a 1 L glass cup, and then distilled water of 320 mL was added. The solution was homogenized and heated in a water bath at 90 °C until dissolved. Then, it was cooled at room temperature. After that, 30 g isoamyl alcohol and 14 g tween-80 were added, then homogenized. Then, 240 mL of 99 % alcohol and 280 mL of distilled water containing 0.2 g of methylene blue were added and homogenized again. Furthermore, distilled water was added until 1 L in volume and homogenized again.

Ice milk was weighed ± 5 grams into a cuvette, and the cuvette was connected to the butyrometer. After that, ± 12 mL of Neusal solution was added into the cuvette. Then it was heated in a water bath type WBU 45 (Mettler) at 60 - 65 °C, then shaken vigorously so that the sample dissolves completely. Warm distilled water was added into the butyrometer until the fat content can be read, after which it was homogenized again using a centrifuge type SuperVario-N (Funke - Dr. N. Gerber Labortechnik GmbH) with a speed of 1100 ± 100 rpm for 3 minutes. Then the fat content was read on the butyrometer scale as a percentage.

Phase separation

Ice milk samples of ± 500 mL in beaker glass were frozen in the freezer for a maximum of 24 hours with a temperature range of -18 to -24 °C. Then ice milk samples were thawed at room temperature (25 ± 2 °C) for a maximum of 3 hours. Ice milk samples were placed in a chiller at 4 ± 1 °C for three days. Daily observations of the watery layer in the sample were conducted, then the height of the serum layer phase that was formed at the bottom of the beaker glass was measured. This test was carried out by observing samples in the middle, left and right of the beaker glass [7].

Fat globule size analysis

This analysis referred to Goff *et al.* [7] using light microscopy method. The sample was poured as much as 1 mL into a 25 mL beaker, and then 20 mL of distilled water was added, stirred until evenly distributed. The mixture was dropped as little as possible using a stirring rod on the base preparation glass and covered with a glass cover. Then, immersion oil was dropped on it. Visible fat globules were observed and measured using a BA210 Motic microscope with a 100x objective lens and a 10x ocular lens. The fat globule size distribution of ice milk was measured at 5 °C after ageing during a certain storage period and the mixing speed.

Sensory evaluation

Six trained panelists were asked to do a two-way test between the test sample and the control (blind control), but not between the test samples [10]. The questions of the questionnaire were whether the test sample with the control (blind control) was the same or different and, if different, how far the difference using the ordinal scale 1: No difference, 2: Very slight difference, 3: Slight/moderate difference, 4: Moderate difference, 5: Moderate/large difference, 6: Large difference, 7: Very large difference. The master sheet of difference from control (DFC) test of mix ice milk can be seen in Table 1.

Table 1. Master sheet of different from control test with replication 1, 2, and 3

Sample code	K	35 rpm (B1)	45 rpm (B2)
I/booth 3 (A1B1)	A1B1	A1B1	A1B2
II/booth 2 (A1B2)	A1B2	A1B1	A1B2
III/booth 1 (A2B1)	A2B1	A2B1	A2B2
IV/booth 5 (A2B2)	A2B2	A2B1	A2B2
V/booth 4 (A3B1)	A3B1	A3B1	A3B2
VI/booth 6 (A3B2)	A3B2	A3B1	A3B2

Information: K was the blind control and also the test sample held by each panelist. Example: panelist I (A1B1) then the blind control was the same sample (A1B1)

35 rpm (B1): Sample combination treatment using an agitation speed of 35 rpm.

45 rpm (B2): Sample treatment combination using an agitation speed of 45 rpm.

A1B1 = ageing of 4 h, speed of 35 rpm

A1B2 = ageing of 4 h, speed of 45 rpm

A2B1 = ageing of 24 h, speed of 35 rpm

A2B2 = ageing of 24 h, speed of 45 rpm

A3B1 = ageing of 48 h, speed of 35 rpm

A3B2 = ageing of 48 h, speed of 45 rpm

This test was worked in pairs between panelists I and II; panelists III and IV; panelists V and VI.

Quantitative descriptive analysis (QDA®) of ice milk

This sensory evaluation of QDA [11] was carried out if, in the sensory evaluation, different test from the control found significant differences at the 5 % level. There were two combinations of presentations, namely AB and BA. Each presentation booth was presented with a different code. The assessed sensory attributes were a vanilla flavor, sweet taste, cooked milk taste, creamy taste, coarse texture, gummy texture, smooth texture, melt rate in the mouth, mouth-coating, and vanilla aftertaste. These attributes were measured on unstructured horizontal lines of 10 cm on their intensities, where 0 - 5 was poor/less, 6 - 7 was fair, 8 - 9 was good, and 10 was excellent.

Data analysis

Data of physicochemical properties were processed statistically using analysis of variance (ANOVA) and Duncan's post hoc test at 5 % level. Sensory evaluation of the DFC test method was processed using ANOVA and Dunnett's post hoc test. QDA® was processed by paired t-student comparison. Fat globule diameter was measured using Motic Image Plus 2.0 ML software.

RESULTS AND DISCUSSION

Physicochemical stabilities of vanilla ice milk

The effect of the interaction of ageing time and agitator speed on fat globule, viscosity, phase separation, total solids, and fat content of ice milk can be seen in Table 2.

Table 2 shows that interaction between the ageing process and agitation speed has a tendency not significant affects in the properties of viscosity, total solid, fat content, phase separation, while it slightly affects the phase separation in the ageing of 48 h and a speed of 45 rpm at the level of 5 %. The interaction of them also affects the diameter of the fat globule in ice milk, where the shorter the ageing time and the faster the agitation speed tend to the lower the fat globule diameter.

Table 2. Interaction of ageing time and agitator speed on physicochemical stabilities of ice milk

Physicochemical properties	Treatment parameters						
	4 h		24 h		48 h		
	35 rpm	45 rpm	35 rpm	45 rpm	35 rpm	45 rpm	
Fat globule [μm]	8.29 ^c	6.40 ^c	10.93 ^a	6.30 ^c	9.24 ^b	7.26 ^d	
Viscosity [cP]	50.80 ^a	57.67 ^a	52.47 ^a	53.97 ^a	52.53 ^a	54.27 ^a	
Total solid [%]	27.13 ^a	27.07 ^a	27.00 ^a	27.13 ^a	26.82 ^a	27.23 ^a	
Fat content [%]	3.97 ^{a,b}	4.00 ^{a,b}	3.90 ^b	3.93 ^{a,b}	4.03 ^a	3.93 ^{a,b}	
Separation phase [cm]	Right side	1.96 ^a	1.71 ^a	1.68 ^a	1.60 ^a	0.33 ^b	1.28 ^a
	Middle side	1.91 ^a	1.74 ^a	1.73 ^a	1.57 ^a	1.28 ^a	0.48 ^b
	Left side	1.84 ^a	1.71 ^a	1.58 ^a	1.54 ^a	1.18 ^a	0.37 ^b

Different superscript letters on each parameter box show significantly different at $\alpha = 0.05$.

The effects of a single factor of ageing time and agitator speed on fat globule diameter, viscosity, phase separation, total solid, and fat content of ice milk are depicted in Table 3. The viscosity of ice milk in this study is much lower compared to the viscosity of whole ice cream in Alkalin *et al.* (2008) and Dogan and Kayacier [12] due to ice milk having lower fat content than regular ice cream. Adapa *et al.* [13] explained that the elastic properties of the ice cream mixture decreased with lower fat content. In addition, the ageing time and agitation speed have no significant effect at the 5 % level on the viscosity of ice milk. This is in accordance with Bazmi and Relkin [14] who reported no change in the viscosity of ice cream at ageing times of 4 and 24 hours at 4 °C. The stability of viscosity during agitation during the ageing process indicates that the viscosity of vanilla ice milk does not change significantly.

The total solid of ice milk in this study is about 27 %. Deosarkar *et al.* [15] reported that low-fat ice cream had a lower total solid content than regular ice cream, which was in the range of 28 - 32 %. Bajad *et al.* [16] stated that low-fat ice cream in India required a total solid that must not be less than 26 %. Goff and Hartel [7] explained that low total solids could be increased by sugar solids, liquid sweetener solids, and stabilizers. The total solid was related to the viscosity of ice cream products where the higher the total solids, the higher the viscosity formed. The statistical result shows that total solid is not significantly different, where it supports the viscosity data, which does not change much in the ageing process. Hartatie [17] explained that stirring during the ageing process will damage the protein membrane that surrounds the fat globule and, if the protein membrane was damaged, the fat globule can be close together and then the cream will rise to the surface. However, the stabilizer and emulsifier play a role in preventing this mechanism.

The fat contents of vanilla ice milk in this study are 3.9 - 4.0 %. These fat levels are in line with those reported by Deosarkar *et al.* [15] that the range of low-fat ice cream fat contents were 2 - 5 %. The ageing process has no significant effect on the fat content of treated ice milk. Clarke [3] described two essential processes that occur during ageing, namely the adsorption of emulsifiers to the surface of fat droplets and the replacement of some milk proteins as well as the crystallization of fats in the droplets. Slow fat crystallization causes the fat content of ice milk to be stable during storage in the ageing tank. Stable fat, together with total solid, also supports the viscosity of ice milk during ageing and agitation.

The stable phase separation was caused by the stabilizer used in the mix [18 – 20]. The statistical result in Table 3 shows that the ageing process tends to reduce the phase separation rate where at the ageing of 48 hours there is a decrease in the phase separation that occurs in observations on the right, middle, and left side of the beaker. Controlling the phase separation was strictly related to the texture, body, and stability of the produced ice milk [18]. In addition, the effect of agitation also tends not to affect phase separation. However, there is a slight decrease in phase separation in the middle side of the observed beaker glass. According to Di Scipio *et al.* [21], stirring will stabilize the viscosity of the emulsions formed. Stirring can reduce the interface tension and expand the surface of the globule.

The fat globule diameter of the ice milk in this study are in the range of 6-10 µm. Large fat globule diameter, which can be seen in Figure 2, is the result of aggregation that occurs between fat globules both granule and floccule type aggregation. Walstra *et al.*

[22] reported that the physical structure of fat globules in ice cream mixes has a diameter of fewer than 2 μm and a maximum fat globule cluster of 10 μm .

Table 3. Single factor of ageing time and agitator speed on physicochemical stabilities of ice milk

Treatment	Level	Fat globule [μm]	Viscosity [cP]	Total solid [%]	Fat content [%]	Phase separation [cm]		
						Right side	Middle side	Left side
Ageing time	4 h	7.34 ^y	54.23 ^x	27.10 ^x	3.98 ^x	1.83 ^x	1.83 ^x	1.78 ^x
	24 h	8.62 ^x	53.22 ^x	27.07 ^x	3.91 ^x	1.64 ^x	1.65 ^x	1.56 ^x
	48 h	8.25 ^x	53.40 ^x	27.03 ^x	3.98 ^x	0.76 ^y	0.88 ^y	0.78 ^y
Agitator speed	35 rpm	9.49 ^p	51.93 ^p	26.98 ^p	3.97 ^p	1.61 ^p	1.64 ^p	1.53 ^p
	45 rpm	6.65 ^q	55.30 ^p	27.14 ^p	3.95 ^p	1.21 ^p	1.26 ^q	1.21 ^p

Different superscript letters on the same column show significantly different at $\alpha = 0.05$.

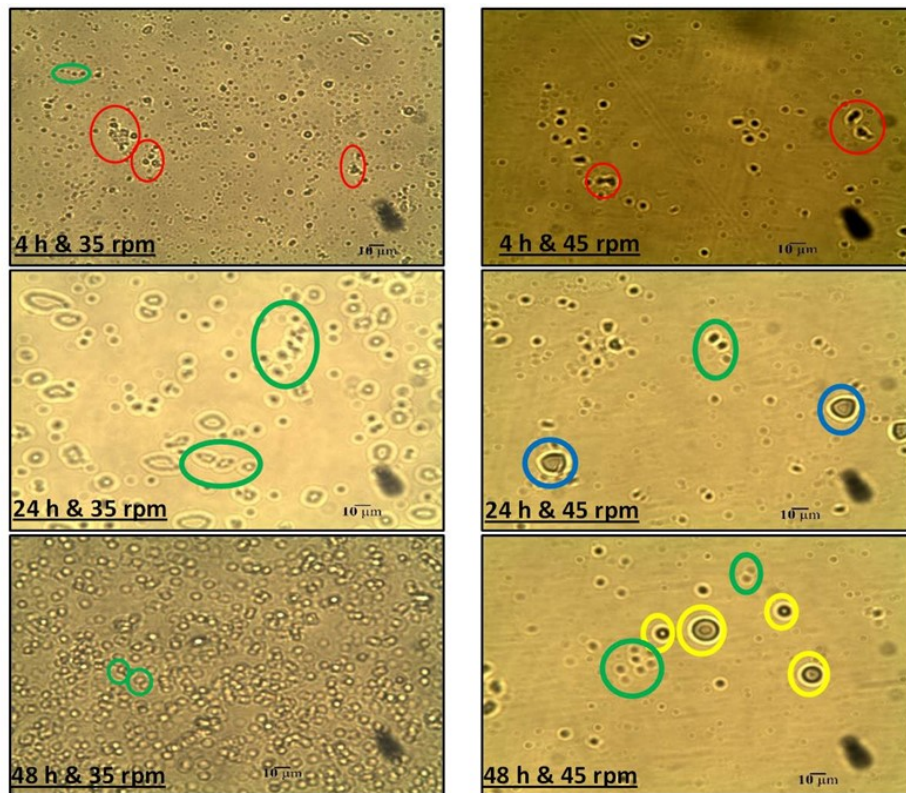


Figure 2. Optics of ice milk micrographs performed at different ageing time and agitation speeds show (the original width of the images for each treatment combination is $54.19 \times 104 \mu\text{m}$): O (red circle) = granule type aggregation; O (green circle) = flocculation type aggregation; O (blue circle) = partial coalescence; O (yellow circle) = coalescence

The clustering of fat globules in ice milk is thought to be caused by several primary factors, namely homogenizer type, homogenization pressure, two-stage homogenization process, fat content, and protein to fat ratio, surfactant type (emulsifier), temperature, and the proper operation of the homogenizer. Ye *et al.* [23] also stated that fat globules could be disturbed during homogenization by turbulence, cavitation, and the formation of air bubbles.

The statistical result in Table 3 shows that the longer the ageing process tends to increase the fat globule diameter. Rizzo [24] explained that the ageing stage could cause droplet aggregation, which impacted the more significant the diameter. The faster agitation produces high shear rates lead to the smaller the fat crystals and the narrower the particle size distribution. The agitation has little effect on the time of fat crystallization induction; however, heat transfer and diffusion were increased [25].

Sensory profiles of vanilla ice milk

Sensory evaluation of ice milk is conducted using DFC test between treatments whose results can be seen in Table 4.

Table 4. Different from control test of vanilla ice milk on overall attribute

Panelist Code	Sample		
	K	35 rpm (B1)	45 rpm (B2)
(I/ A1B1)	1.67 ^c	1.33 ^c	2.33 ^d
(II/ A1B2)	1.67 ^a	1.33 ^b	3.00 ^a
(III/ A2B1)	1.00 ^e	1.00 ^e	1.00 ^e
(IV/ A2B2)	1.00 ^e	1.00 ^e	1.00 ^e
(V/ A3B1)	1.00 ^e	1.00 ^e	1.00 ^e
(VI/ A3B2)	1.00 ^e	1.00 ^e	1.00 ^e

Information:

Different superscript letters on the same line show significantly different at $\alpha = 0.05$.

A1B1 = ageing of 4 h, speed of 35 rpm

A1B2 = ageing of 4 h, speed of 45 rpm

A2B1 = ageing of 24 h, speed of 35 rpm

A2B2 = ageing of 24 h, speed of 45 rpm

A3B1 = ageing of 48 h, speed of 35 rpm

A3B2 = ageing of 48 h, speed of 45 rpm

K = blind control

35 rpm (B1) = sample combination treatment using an agitation speed of 35 rpm.

45 rpm (B2) = sample treatment combination using an agitation speed of 45 rpm.

Dunnet test results show differences in the sensory attributes of vanilla milk ice in 4-hour ageing sample with a speed of 35 rpm and 45 rpm and no significant difference in other treatments. Goff and Hartel [7] explained that mixes were not ready entirely if they were directly processed for hardening, where mixes were generally aged for more than 4 to 24 hours. In addition, Clarke [3] reported that the ageing process was carried out overnight for production needs, but not more than three days. Ice milk with the ageing of 4 hours with the speed of 35 and 45 rpm then performed a quantitative descriptive analysis to see the specific attributes difference. The attributes are vanilla flavour, sweetness, cooked milk taste, creamy taste, coarse texture, gummy texture, smooth texture, melt rate in the mouth, mouth-coating, and vanilla aftertaste which can be seen in Table 5.

Table 5. Quantitative descriptive analysis of milk ice vanilla

Sensory Attribute	Sample	
	A1B1	A1B2
Vanilla Flavors	6.01 ^a	5.23 ^a
Sweetness	7.08 ^a	7.00 ^a
Cooked Milk Taste	2.71 ^a	1.73 ^a
Creamy taste	5.18 ^a	4.85 ^a
Coarse Texture	6.35 ^a	2.94 ^b
Gummy Texture	4.22 ^a	3.23 ^a
Smooth Texture	5.28 ^a	5.48 ^a
Melt Rate in Mouth	4.43 ^a	5.43 ^a
Mouth Coating	5.26 ^a	5.59 ^a
Vanilla Aftertaste	2.29 ^a	3.08 ^a

Information:

The different letter notations on the line show significantly different at the level of 0.05

A1B1: Ageing time of 4 hours; agitation speed of 35 rpm

A1B2: Ageing time of 4 hours; agitation speed of 45 rpm.

Table 5 shows that the ageing time of 4 hours with speeds of 35 rpm and 45 rpm is not significantly different in the sensory attributes of vanilla flavor, sweetness, cooked milk taste, creamy taste, gummy texture, smooth texture, melt rate in the mouth, mouth-coating, and vanilla aftertaste ($p > 0.05$). A significant difference is detected in the coarse texture attribute, where the ageing time of 4 hours with the agitation speed of 35 rpm has a higher coarse texture value than the speed of 45 rpm. It can be caused by the faster the agitation leads to the easier the mixture can be mixed. However, the agitation speed cannot be too fast because it can increase the temperature and accelerate the phase separation of the mix during the ageing time. Coarse texture can also be formed due to non-fat solid solids (milk solid non-fat), where the main components consisted of lactose, protein, and minerals [7].

The results in this study serve as the basis for the justification that the ageing process of 4 to 48 hours and the use of different agitation speeds at 35 and 45 rpm produce uniform and consistent physicochemical stabilities and sensory profile of vanilla ice milk. So that the industry can develop its products using these two different ageing tanks with the same excellent quality of ice milk.

CONCLUSION

It can be concluded that in general, the ageing time and the speed of agitation did not have a significant effect on viscosity, total solids, and fat content. However, it has little effect on the parameters of fat globule diameter and phase separation. Specifically for the phase separation, ageing at 48 hours and agitation speed of 45 rpm decreased the phase separation in ice milk. The sensory profile of ice milk showed that 4 hours aged ice milk with an agitation speed of 35 rpm was detected significantly different from that of 45 rpm speed based on overall sensory attributes. Based on QDA®, this difference occurred only in the coarse texture attribute, while the other nine parameters were not significantly different. This minor difference can be improved in the next process so that the product quality of ice milk in the two ageing tanks with agitation speeds resulted in uniform and consistent product quality.

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