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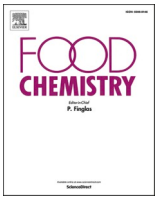

Effect of alternative sweetener and carbohydrate polymer mixtures on the physical properties, melting and crystallization behaviour of dark compound chocolate

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ABSTRACT

This study aims to evaluate the effect of sucrose replacer mixtures (erythritol, mannitol, or tagatose in combination with inulin or polydextrose) on the crystal morphology, particle size distribution, rheology, melting properties, and fat polymorphism of dark compound chocolate. The result showed that the replacer mixture's hygroscopicity, particle size, and sugar crystal shape might significantly impact dark compound chocolate's rheological and textural properties but had no substantial impact on the melting properties and fat crystallization. Mannitol-containing samples exhibited the highest rheological value, likely related to their high moisture content, small particle size, and elongated crystal shape. Due to the similar specific surface area and comparable D90 value, the sample containing erythritol-polydextrose mixture resulted in a similar ($P \geq 0.05$) Casson yield value (46.184 ± 2.45 Pa) compared to the sample containing sucrose (38.348 ± 1.68 Pa). It could be a potential sucrose replacer in the dark compound chocolate.

1. Introduction

Chocolate, a fat-based confection with high sugar content, has processability, quality, and sensory properties of the final product that are closely associated with the fat crystal morphology, polymorphic behavior, and interactions of nonfat ingredients (West & Rousseau, 2018). Sucrose, a disaccharide with an α-(1 → 2) glycosidic linkage, is the major non-fat ingredient in chocolate that gives caloric energy of 4 kcal/g. It contributes to chocolate's sensory, textural, and rheological properties (Afoakwa, Paterson, Fowler & Vieira, 2008). The high sugar content of chocolate makes it a less healthy food choice, driving the development of sucrose-free chocolate.

A sucrose replacer is an ingredient that can replace the functionality of sucrose in food applications. Alternative sweeteners and low digestible carbohydrate (LDC) polymers are the common sucrose replacers in chocolate. Alternative sweeteners are classified into two general types: high-potency sweeteners (HPS) and bulk sweeteners. HPS has intense sweetening power, but they do not have bulking properties; on the other hand, bulk sweeteners that have bulking properties but possess lower sweetening power than HPS. The bulk sweeteners can be sugar alcohol (polyol), monosaccharides, and disaccharides. Polyols are bulk sweeteners containing one hydroxyl group (–OH) attached to each carbon atom of their sugar molecule. Erythritol is a non-caloric polyol (0 kcal) with 60 % sweetness relative to sucrose, while mannitol is a low-calorie polyol (2.4 kcal/g) with 50% as sweet as sucrose (O'Brien-Nabor, 2012). Both polyols are non-hygroscopic as they do not absorb water until the relative humidity exceeds 90% (Wong, Thoo, Tan & Siow, 2022). Tagatose ($C_6H_{12}O_6$) is a low-calorie monosaccharide (1.5 kcal/g), that is 92% as sweet as sucrose. It is a reducing sugar that takes part in the Maillard reaction and caramelizes more readily than sucrose at high temperatures (O'Brien-Nabor, 2012). LDC polymers are composed of sugar such as glucose, mannose, and fructose, linking together to become a high molecular weight polysaccharide. Inulin and polydextrose are low-calorie LDC polymers (1—1.5 kcal/g) that provide body and texture to the chocolate. They are a non-digestible carbohydrate considered a dietary fiber by the United States Food & Drug Administration (USDA) (USDA, 2021). Inulin is a polysaccharide with 3 to 60 fructose units connected by β-2 → 1 links and terminated by a glucose unit. The melting temperature, glass transition temperature, capability of gel formation and the subsequent gel strength, interaction with other food components, taste, and solubility of inulin depends on its molecular structure (Mensink, Frijlink, Van Der Voort Maarschalk, &

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Hinrichs 2015). Polydextrose is a randomly linked amorphous glucose polymer by all possible glycosidic linkages, including α- and β-1–2, 1–3, 1–4, and 1–6, with some branching. (Beckett 2009).

Chocolate is a shear-thinning, non-Newtonian pseudoplastic material. The rheological properties are important because they affect the processing efficiency, e.g., extrusion, enrobing, etc., and organoleptic properties. In the context of non-fat ingredients, chocolate rheology highly depends on the solid packing intensity and specific ingredient interactions (e.g., emulsifier) (West & Rousseau, 2018). The reduction in particle size of the sucrose replacer increases the solid packing intensity, surface area, and interaction with the continuous phase, attributing to the rise of the energy barrier needed to overcome the yield stress and resulting in much higher viscosity (Beckett, 2009). Despite particle size, moisture often has a direct effect on chocolate. It could cause agglomeration by clustering the hydrophilic particulates through attractive polar forces and capillary action, resulting in poor mixing and adversely impacting viscosity (Novakova et al., 2002). Saputro et al. (2017) also showed that incorporating hygroscopic palm sugar increased moisture content and the generation of amorphous sugar, causing the sugar particles to crumble together and form agglomerates, increasing the viscosity during chocolate production. Hence, it is critical to understand the particle size and hygroscopicity of the sucrose replacer mixture to maintain the rheology and prevent undesirable texture.

The term “hygroscopic” refers to materials that absorb more than 5% by mass between 40 and 90% RH at 25 °C (Ford & Willson, 1999). A preliminary hygroscopic study revealed that sucrose is a low hygroscopic disaccharide because it does not absorb water until relative humidity is 85%. Sucrose replacer mixtures containing erythritol, mannitol, or tagatose in combination with inulin or polydextrose were found to be potential sucrose replacer mixtures because of their low hygroscopicity at a relative humidity of 65%, which is the average humidity of tropical countries like Malaysia (Wong, Thoo, Tan & Siow, 2022). Thus, this study aimed to evaluate the effect of the sucrose replacer mixtures on the physical (crystal morphology, moisture content, particle size distribution), rheological, fat polymorphism, and thermal properties of dark compound chocolate.

2. Material and method

Sucrose was supplied by MSM Prai Sdn Bhd., Malaysia; erythritol, mannitol, and tagatose were provided by Salus Nutra, China; inulin (average DP: 10) and polydextrose (average DP: 12) were purchased from Xi’an Saiyang Bio-Technology, China. Hydrogenated palm kernel oil (HPKO) was supplied by Welltop Food Ingredients Sdn Bhd, Malaysia; Cocoa powder (10–12 g fat/100 g, pH 6.8–7.2, moisture 5 g/100 g) and cocoa butter were supplied by Guan Chong Cocoa, Malaysia; soy lecithin (moisture 0.19 g/100 g) was purchased from Cargill, Shanghai, China. Isopropanol, chloroform, and methanol were supplied by Sigma-Aldrich (Missouri, USA). All the chemicals used were of analytical grades.

2.1. Dark compound chocolate production

The alternative sweetener and LDC polymer ratio were obtained through a preliminary acceptance sensory test. This ratio and the formulation of the dark compound chocolate are listed in Table 1. Dark compound chocolates were produced using the method described by Kadivar, De Clercq, Van de Walle & Dewettinck (2014) with slight modifications. Firstly, the sugar (sucrose, alternative sweetener, or LDC polymer) was finely ground and passed through a 100 μm sieve. In a separate step, the cocoa butter (CB) and hydrogenated palm kernel oil (HPKO) were weighed and melted for 30 mins at a temperature of 40 °C. The melted CB and HPKO were combined in a bowl and well-mixed using a spatula. Subsequently, the sugar, cocoa powder, and 2/3 of the melted fat (CB/HPKO) mixture were refined and conched in a stone melanger (Spectra 11, UK) for 4 h. Once the viscosity was reduced, a paste-like mixture was obtained. The remaining 1/3 of the melted fat mixture was added and thoroughly mixed for 2 h to achieve the desired flow characteristics. The liquid chocolate was then tempered manually according to the method described by Talbot (1994). Tempering was performed to produce the desired β crystals in the chocolate products. Tempering was carried out as follows: i) chocolate was melted at 48 °C to remove crystal history, ii) approximately 2/3 of the chocolate mixture was poured onto a marble slab and mixed with a flexible spatula until the product reached 27 °C to produce seed crystals and iii) the thickened chocolate was then mixed with the remaining 1/3 warm chocolate (40 °C) to melt the unstable crystal polymorphs and to reach the chocolate working temperature of 31–32 °C. The tempered chocolate was poured into a plastic mold (98 mm × 30 mm × 11 mm) and cooled at 10 ± 1 °C for 30 min to solidify the chocolate (De Clercq et al., 2012). The dark compound chocolate bars were de-molded and stored in a sealed plastic container. The samples were conditioned in a cold room at 7 °C for 2 weeks to stabilize the fat crystal before the analysis. Moisture content was determined after the 2-week storage period using the Karl Fischer method (ICA 1998).

2.2. Texture analysis

Hardness (N, the maximum force required to penetrate the sample) of the dark compound chocolates was measured with a Texture Analyser (TA-XT plus, Stable Microsystems Ltd., Surrey, UK) equipped with a 2 kg load cell and probe (P/2N needle stainless) using the following parameters: product height 10 mm, penetration depth 5 mm, speed 1 mm/s, test speed 2 mm/s, post speed 10 mm/s and the duration of the test at ~ 1–2 min (Kadivar, De Clercq, Van de Walle & Dewettinck 2014). Maximum compression force in penetration was denoted as sample hardness.

2.3. Particle size distribution

Particle size distribution analysis was performed using a MasterSizer 3000 (Laser Diffraction Particle Size Analyser, Malvern Instruments Ltd.,

Table 1
Formulation of the dark compound chocolate.

Sample code	Ingredient (% w/w)								
	Sucrose	Erythritol	Mannitol	Tagatose	Inulin	Polydextrose	Cocoa butter	HPKO	Cocoa powder
Suc	46.0						31.4	1.6	21.0
EryI		35.2			10.8		31.4	1.6	21.0
EryP		35.2				10.8	31.4	1.6	21.0
ManI			35.2		10.8		31.4	1.6	21.0
ManP			35.2			10.8	31.4	1.6	21.0
TagI				35.2	10.8		31.4	1.6	21.0
TagP				35.2		10.8	31.4	1.6	21.0

Sample codes were used to identify the dark compound chocolate that was made from sucrose (Suc) or different sucrose replacer mixtures: EryI for erythritol and inulin mixture; EryP for erythritol and polydextrose mixture; ManI for mannitol and inulin mixture; ManP for mannitol and polydextrose mixture; TagI for tagatose and inulin mixture; and TagP for tagatose and polydextrose mixture. HPKO for hydrogenated palm kernal oil.

Malvern, Worcestershire, UK) equipped with a Hydro EV. Approximately 0.5 g of chocolate slivers was mixed with 10 ml of isopropanol. The sample (~0.2 ml) was dispersed in isopropanol until an obscuration of 10.5%, as recommended by the instrument software. The sample was sonicated for 2 min to ensure particles were independently dispersed and maintained by stirring during the measurement. PSD was determined based on the Mie-Theory using the refractive index of 1.59 for dark chocolate (Afoakwa, Paterson, Fowler & Vieira, 2008). Results were provided as a relative volume (%) of particles in size compared with particle size curves (Malvern MasterSizer Micro Software). PSD parameters obtained included specific surface area and largest particle size (D90).

2.4. Rheological properties

The rheological properties of the molten chocolates were studied using a rheometer (Anton Paar (MCR51 model, Austria) with a concentric cylinder system (cup and bob with model # CC40-0-60 42626). Chocolate samples were melted at 45 °C before analysis. The stage temperature was set at 40 °C to prevent the solidification of samples during analysis. Approximately 20 ml of the sample was transferred to the cup, and measurement was taken with a bob and a gap of 1 mm. Samples were equilibrated at 40 °C for 15 min prior to taking measurements, followed by pre-shearing at 5/s for 5 min. Shear stress was measured by increasing the shear rate from 2/s to 50/s, holding the shear rate at 50/s for 5 min, then decreasing from 50/s to 2/s, following the parameters set by the International Office of Cocoa, Chocolate and Confectionery (IOCCC) as suggested by Rheocompass software (Rheocompass 1.20, Anton-Paar Ltd., Graz, Austria). Data was fitted in a Casson model to obtain yield stress and viscosity measurements using the Rheocompass software. The data of the upward flow curve were then fitted to the Casson model to derive the Casson yield value (σ_{CA}) and Casson plastic viscosity (η_{CA}). Thixotropy was obtained by determining the difference between the ramp-up and ramp-down shear stress at 5/s. The Casson model is denoted by $\sqrt{\sigma} = \sqrt{\sigma_{CA}} + \sqrt{\eta_{CA}} \bullet \sqrt{\gamma}$, where σ: yield stress; σ_{CA}: Casson yield value; η_{CA} Casson viscosity; and γ shear rate.

2.5. Melting properties

The melting properties of the dark compound chocolate samples were determined using a differential scanning calorimeter (DSC-60, Shimadzu, Japan). About 5 mg of each sample was loaded into an aluminum pan. The hermetically sealed pan was then heated from 0 to 70 °C at a scan rate of 5 °C/min. An empty aluminum pan was used as a reference. The onset temperature (*T*-onset), peak temperature (*T*-peak), endset temperature (*T*-endset), and enthalpy of melting (ΔH_{melt}) were automatically computed and were calculated from the melting thermogram using DSC software (Aidoo, Afoakwa & Dewettinck, 2014).

2.6. Polymorphism

To identify the polymorphic transformations of dark compound chocolate, a D8 Discover X-ray Diffraction (Bruker, Karlsruhe, Germany) fitted with Cu-Ka radiation (k = 1.5418 Å, voltage 40 kV and current 40 mA) was used at room temperature. Approximately 5 g of the sucrose, sucrose replacer (sucrose, erythritol, mannitol, tagatose, inulin, or polydextrose), and the chocolate slivers were used as samples in this study. The chocolate silver was prepared by chopping the surface of the chocolates with a scalpel. All the samples were analyzed at 2θ angles of 10° – 30° with a scan rate of 1.5°/min. Short (d) spacing (Å) was determined using the EVA-diffraction software (Bruker, Karlsruhe, Germany). Assignments of polymorphs were based on the following short spacing (SS) characteristics of CB: γ form (d = 3.7/4.19 Å); α form (d = 4.24 Å); β' forms (d = 3.8–4.3 Å) and β forms (d = 4.5/4.6 Å) (Wille & Lutton, 1966).

2.7. Microstructure analysis

Microstructures were observed at room temperature using a high-resolution polarised light microscope (PLM, Olympus BX51, Tokyo, Japan) fitted with a digital camera (Nikon, DS-Filc, Tokyo, Japan). The dark compound chocolate sample was melted at 55 °C to destroy crystal memory. Approximately 10 mg of the molten chocolate was placed on a preheated (55 °C) glass microscope slide. A coverslip was placed over the sample and centered on the melted sample to ensure uniform thickness. PLM images were captured using a 100x objective lens after the slides were kept at room temperature for 4 h to solidify the particles.

2.8. Statistical analysis

One-way ANOVA tests were used for data analysis since only one independent variable was taken into account in each analysis. Using SPSS software, version 23 (IBM Corp., Chicago, USA). Tukey post hoc tests were employed to determine the significant difference in the analyses at a 95% confidence level. All analyses were conducted in triplicate, and the results were expressed in mean ± standard deviation.

3. Results and discussion

3.1. Moisture content

The moisture content of the dark compound chocolate could affect its textural and storage stability by changing the microscopic structure of chocolate (West & Rousseau, 2018). Beckett (2009) emphasized the importance of maintaining the chocolate moisture content below 1% to avoid the agglomeration of the hygroscopic component that can affect the chocolate viscosity.

Although the moisture content of all the samples remained below the 1% tolerance (Fig. 1), the sucrose-free sample (EryI, EryP, ManI, ManP, TagI and TagP) had significantly ($P < 0.05$) higher moisture content than the sucrose-containing sample (Suc), indicating that sucrose replacer mixture could have higher hygroscopicity than sucrose and absorb more moisture during the 2-week storage period. The preliminary hygroscopic test result that was previously shown in Wong, Thoo, Tan & Siow (2022) showed that sucrose was non-hygroscopic at a_w 0.65 because it did not absorb any moisture, while the sucrose replacer mixtures with minimal hygroscopicity absorbed moisture<5% in weight at a_w 0.65. This might indicate that the hygroscopicity of the sucrose replacer mixture has a direct impact on the moisture content of the dark compound chocolate. Such a result was similar to Shah, Jones & Vasiljevic (2010), which also showed that the incorporation of whey protein with high water holding capacity could significantly ($P < 0.05$) increase the moisture content in milk chocolate.

Among the sucrose-free samples, the polydextrose-containing sample had significantly ($P < 0.05$) higher moisture content than the corresponding inulin-containing samples with the same alternative sweetener (polyol or monosaccharide). This difference can be attributed to the high hygroscopic nature of polydextrose, which could absorb more moisture during the 2-week storage period. This finding aligns with a preliminary hygroscopic study by Wong, Thoo, Tan & Siow (2022), which demonstrated that polydextrose exhibits a greater water affinity than inulin in achieving equilibrium at an a_w of 0.65. This might be attributed to the higher degree of branching and polymerization (average DP:12) of polydextrose compared to the linear structure of inulin (average DP:10). According to Singh, Indoria, Jisha, and Gardas (2021), the presence of increased branching in polysaccharides enhances their solubility by introducing steric hindrance, which reduces intramolecular interactions between the polysaccharide molecules. As a result, highly branched polysaccharides have more available hydroxyl groups (OH) for hydrogen bonding with water molecules, contrasting with linear polysaccharides. Similar results can be found in previous studies. Mensink, Frijlink, Van Der Voort Maarschalk, K. & Hinrichs (2015) demonstrated

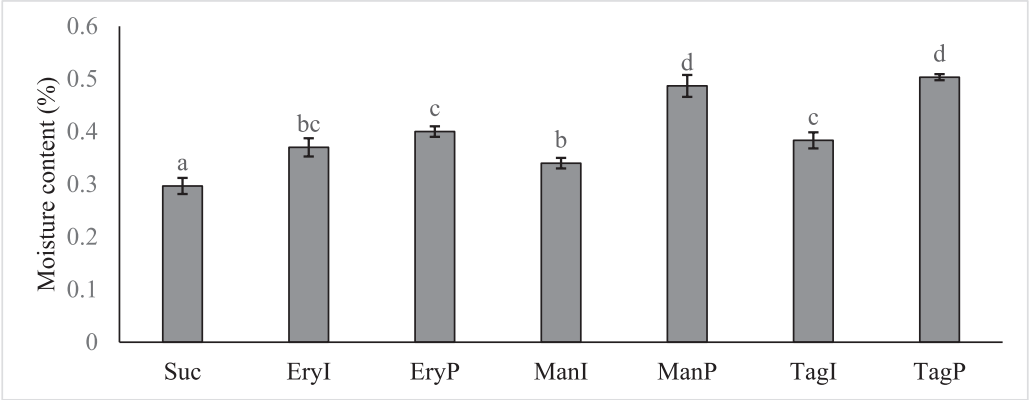

Fig. 1. Moisture content of the dark compound chocolate samples from the triplicate analysis (n = 3). [a, b, c] Different superscript letters indicate a significant difference ($P < 0.05$) detected between the samples in the moisture content test.

that the solubility of the polydextrose is 80% w/w at 25 °C, which is higher than the solubility of inulin with a DP of 12, as reported by Fallourd & Viscione (2009) at 12% w/v at the same temperature. Moreover, Ünal & Arslan (2022) discovered that the jelly made with polydextrose exhibited a moisture content of 45.07 ± 5.04%, significantly exceeding the moisture content of the jelly made with inulin (22.73 ± 0.60%) after 30 days of storage at 4 °C.

Despite that, the moisture content difference between the sucrose-containing and sucrose-free samples was minimal, measuring<0.1%. As a result, all the sucrose replacer mixtures demonstrated the potential to replace sucrose in dark compound chocolate, with the exceptions of ManP and TagP.

3.2. Particle size distribution

The particle size distribution (PSD) is crucial in understanding the chocolate properties because it gives information about the surface area and solid packing density. Chocolate particle size is usually controlled below 30 μm; exceeding this size results in a coarse mouthfeel (Schumacher et al. 2009). To characterize the particle size, the D90 value is usually used as it indicates that 90% of the particle is finer than this size; while the specific surface area (SSA) gives information about the surface area available for the particle to interact in the chocolate matrix (Sokmen & Gunes 2006).

Table 2 shows that the D90 value of all samples was below the 30 μm tolerance limit. The Suc sample had the largest D90 value (29.467 ± 0.65064 μm) and the lowest SSA (8455.667 ± 636.76 m^2/kg) among all the samples. This indicated that the sucrose-free sample had a significantly ($P < 0.05$) smaller particle size than Suc, increasing solid packing density and the surface area available for particle–particle interaction. Moreover, the volume histogram (Fig. 2) also showed that all the sucrose-free samples displayed a narrower unimodal particle size distribution with a higher distribution maximum than the Suc sample. As no specific trend was found between the sucrose replacer mixture, the difference in the particle size could be generated from the milling process and less related to their molecular structure. This was similar to Aidoo, Afoakwa & Dewettinck (2014), which showed no specific trend in D90 of dark chocolate with different inulin and polydextrose content. Rohm, Böhme & Skorka (2018) also stated that the particle size of the sucrose replacer was affected by the rotational speed and intensity of grinding during production.

3.3. Rheological properties

Casson plastic viscosity is the chocolate flow that relates to the internal friction of the chocolate. It characterizes the dark compound chocolates pumping and filling characteristics, coating properties, and sensory perceptions (Servais, Jones & Roberts 2004). The plastic viscosity of samples in this study was between 2 and 4.5 Pas, within the value reported in Saputro et al. (2017). The Casson yield value, which relates to the energy needed for the chocolate to start moving from a resting state, characterizes its shape retention, molding, or enrobing ability (Servais, Jones & Roberts 2004). The Casson yield values of samples in this study were between 38 and 103 Pa, which was not in the

Table 2
Effect of sucrose replacement on particle size and melting properties of dark compound chocolate.

Sample	D90* (μm)	Specific surface area (m^2/kg)	Low-temperature melting endotherm peak				High-temperature melting endotherm peak			
			T_{onset} (°C)	T_{peak} (°C)	T_{end} (°C)	ΔH_{melt} (J/g)	T_{onset} (°C)	T_{peak} (°C)	T_{end} (°C)	ΔH_{melt} (J/g)
Suc	29.467 ± 0.65^{c}	8455.667 ± 636.76^{a}	11.0 ± 0.6^{bc}	16.3 ± 0.83^{a}	19.2 ± 0.9^{a}	20.1 ± 0.7^{d}	30.5 ± 0.0^{a}	34.3 ± 0.3^{a}	36.3 ± 0.2^{a}	32.3 ± 0.7^{a}
EryI	19.033 ± 0.87^{a}	13570.000 ± 788.86^{b}	11.5 ± 0.1^{c}	16.4 ± 0.1^{a}	18.9 ± 0.3^{a}	15.7 ± 0.2^{c}	30.1 ± 0.2^{a}	34.5 ± 0.3^{a}	36.2 ± 0.4^{a}	36.5 ± 2.0^{a}
EryP	21.900 ± 1.21^{b}	9767.333 ± 874.62^{a}	8.9 ± 0.3^{a}	13.8 ± 1.2^{a}	17.8 ± 0.5^{a}	1.4 ± 0.1^{ab}	30.0 ± 0.3^{a}	33.7 ± 0.0^{a}	35.5 ± 0.1^{a}	39.2 ± 2.3^{a}
ManI	19.133 ± 1.64^{a}	11841.000 ± 883.52^{b}	10.0 ± 0.9^{abc}	15.2 ± 1.4^{a}	18.6 ± 0.7^{a}	1.6 ± 0.1^{ab}	30.2 ± 0.4^{a}	34.4 ± 0.4^{a}	36.1 ± 0.6^{a}	37.0 ± 1.8^{a}
ManP	20.367 ± 0.60^{ab}	16200.000 ± 70.00^{c}	9.9 ± 0.8^{abc}	14.8 ± 1.9^{a}	18.1 ± 0.9^{a}	0.8 ± 0.0^{a}	29.9 ± 0.6^{a}	33.9 ± 0.4^{a}	35.7 ± 0.6^{a}	35.4 ± 3.4^{a}
TagI	18.633 ± 0.64^{a}	12440.000 ± 630.95^{b}	9.9 ± 0.6^{abc}	14.2 ± 1.3^{a}	17.8 ± 0.9^{a}	0.9 ± 0.0^{a}	29.9 ± 0.4^{a}	33.9 ± 0.4^{a}	35.9 ± 0.4^{a}	37.0 ± 3.0^{a}
TagP	18.700 ± 0.82^{a}	13613.333 ± 309.90^{b}	9.5 ± 0.6^{ab}	13.9 ± 0.9^{a}	17.8 ± 0.6^{a}	1.8 ± 0.1^{b}	30.2 ± 0.2^{a}	34.0 ± 0.2^{a}	35.9 ± 0.3^{a}	39.7 ± 0.4^{a}

Mean values ± standard deviations from the triplicate analysis (n = 3). [a, b, c] Different superscript letters in the same column indicate a significant difference ($P < 0.05$) detected between the samples.

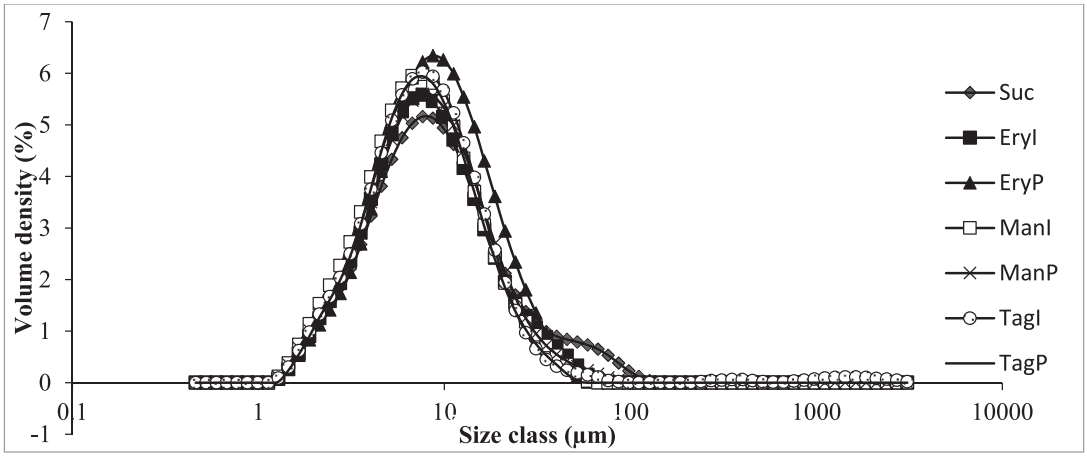

Fig. 2. Particle size distributions of dark compound chocolate sweetened with Suc (-◆-line); EryI (-■-line); EryP (▲line); ManI (□line); ManP (✕ line); TagI (○line) and TagP (—line).

range that had been previously reported (4–35 Pa) (Aeschlimann & Beckett, 2000). The difference might be due to the absence of an emulsifier in the formulation of this study because the emulsifier is important in reducing the interaction of dispersed solids and lowering the sample viscosity (Beckett, 2009).

Fig. 3 and supplementary material 1 showed that all the sucrose-free samples had significantly higher plastic viscosity ($P < 0.05$) than the sucrose-containing sample, indicating that more force was needed to maintain the chocolate flow in the sucrose-free sample than the sucrose-containing sample. This was probably due to their difference in PSD. With a narrow PSD (Fig. 2) and smaller D90 (Table 2), sucrose-free samples had higher solid packing ability and larger specific surface area for particle–particle interaction. Increasing the points of contact between the particles might increase internal friction and cause a rise in plastic viscosities. This is in agreement with Servais, Jones & Roberts (2004), who reported that a few percent increases in solid content could double the viscosity of high solid content suspensions. The same phenomena can be seen in the Casson yield value. The mannitol-containing samples (ManI & ManP) and tagatose-containing samples (TagI & TagP) that had smaller D90 values and higher SSA values than sucrose-containing samples (Table 2), exhibited significantly ($P < 0.05$) higher Casson yield value than the later. This indicated that higher stress was needed to initiate a flow or destruct the shape of mannitol-containing and tagatose-containing samples compared to the sucrose-containing sample. The result conformed with Mongia & Ziegler (2000), who stated that the flow behavior was mainly dominated by the particle–particle interactions of chocolate components at low shear stresses. The stronger the particle–particle interaction, the higher the stress needed to move the chocolate from the resting state.

Despite plastic viscosity and yield value, thixotropy is important in characterizing the rheology of dark compound chocolate because it indicates the degree of agglomeration in the sample that could be affected by the chocolate components. The thixotropy result (Fig. 3) showed that incorporating sucrose replacer mixtures could affect the agglomeration of dark compound chocolate. This can be seen in the erythritol-containing (EryI and EryP) and tagatose-containing samples (TagI and TagP) that exhibited significantly ($P < 0.05$) higher thixotropic behavior than the sucrose-containing sample. This might be due to the

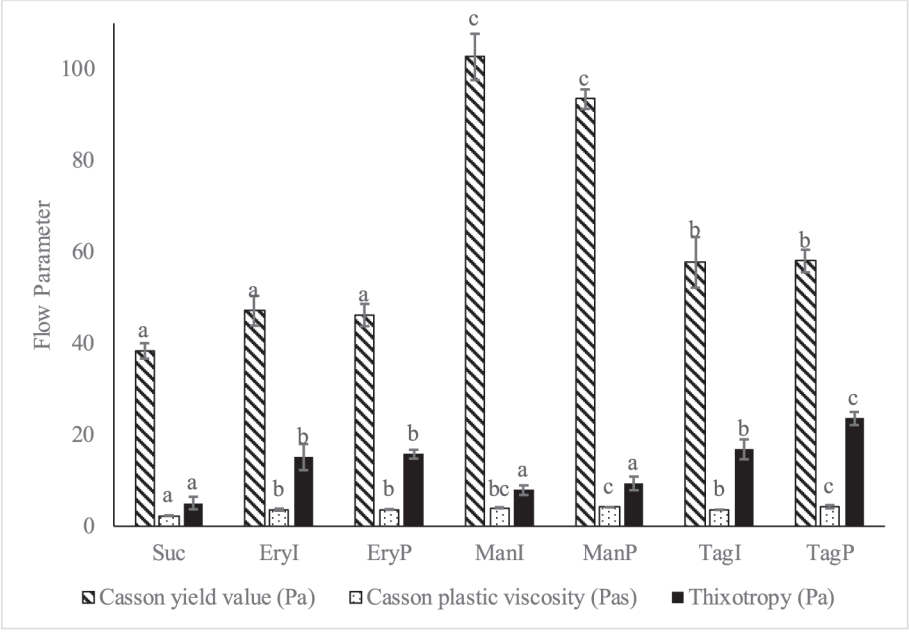

Fig. 3. Effect of sucrose replacement on Casson yield value, Casson plastic viscosity, and thixotropy of dark chocolates from the triplicate analysis (n = 3). [a, b, c] Different superscript letters indicate a significant difference ($P < 0.05$) detected between the samples.

incorporation of more moisture (Fig. 1) by the erythritol-containing and tagatose-containing samples, leading to the rise in agglomeration of the particles in these samples. This was in accordance with Saputro et al. (2017), which stated that high moisture content in dark chocolate could create agglomeration of the particles, thus affecting the chocolate flow.

Other than moisture content, the thixotropy of dark compound chocolate might be affected by the crystal shape of the sucrose replacer mixture. Mannitol with an elongated crystal shape (Supplementary material 2), displayed an exceptional ($P < 0.05$) low thixotropy compared to the other sucrose-free samples. This indicates that the elongated shape of the mannitol crystal might reduce the agglomeration of the particulate in the sample. This was in accordance with Ochsenbein, Vetter, Morari, & Mazzotti (2015) which had shown that the shape of the crystal could affect the agglomeration of the particles. However, the underlying mechanism was unclear.

3.4. Melting properties

The melting properties of dark compound chocolate were mainly dominated by the crystallization and polymorphism of triacylglycerols (TAGs) in the chocolate. Cocoa butter (CB) TAG can crystallize into polymorphic forms γ, α, β', and β with their melting point at 18.0, 23.5, 28, and 34.5 °C, respectively (Lonchampt & Hartel, 2004). The stable βV and βVI polymorphs are typically preferred in chocolates because of their melting points and stable shelf-life. 5% of CB was substituted by hydrogenated palm kernel oil (HPKO) in producing dark compound chocolate samples in this study. HPKO is a lauric cocoa butter substitute (CBS) with different TAG compositions but possesses physical properties resembling CB's (Wang et al., 2010).

Table 2 and supplementary material 3 showed that the chocolate samples melted at around 13 to 35 °C, within the expected range of the chocolate melting profile reported previously (Afoakwa, Paterson, Fowler, Vieira, 2009). According to Aidoo, Afoakwa & Dewettinck (2014), T_{onset} corresponded to the temperature at that a crystal polymorph started to melt; T_{peak} corresponded to the temperature at which the melting rate was the greatest; and T_{end} represented the completion of liquefaction of the crystal. ΔH_{melt} showed the heat required for the complete melting of the product.

There were two melting endotherm peaks detected: a small endotherm at low temperature (T_{peak}, 13–16 °C with ΔH_{melt} of 0.8–20 J/g) and a large endotherm at high temperature (T_{peak}, 33–34 °C with ΔH_{melt} of 32–39 J/g), indicating the presence of more than one type of fat polymorph in the samples. Such observation was in accordance with Wang et al. (2010), who had also identified a double endothermic peak in HPKO-made dark compound chocolate at 26.9 and 34.7 °C with melting enthalpies of 5.51 and 14.76 J/g, respectively. The author also speculated that the long-chain trisaturated TAG in the samples corresponded to the high-temperature melting peak while the other TAGs corresponded to the low-temperature melting peak.

There was no significant difference ($P \geq 0.05$) in T_{onset}, T_{peak}, and T_{end} for the high-temperature melting peak of all the samples (Table 2), indicating that the stability of the crystals was similar in all the samples. The samples had a T_{onset} at 30 ± 1 °C, showing the presence of βV fat crystal; while T_{peak} and T_{end} were detected at 34 ± 1 °C and 35 ± 1 °C, respectively, indicating the presence of βVI fat crystal in all the samples (Beckett 2009). There was no significant difference ($P \geq 0.05$) in the melting enthalpies (ΔH_{melt}) of all the samples, indicating that all the samples had a similar fat crystallinity. Such observation suggested that the molecular structure and the difference in PSD of these sucrose replacer mixtures did not interfere with the fat polymorphism of βV and βVI form in dark compound chocolate. This corresponded to Fernandes, Mɛuller & Sandoval (2013), who showed that changes in the PSD of sucrose did not affect the formation of βV and βVI fat crystals in CB-made dark chocolate.

At the low melting peak, T_{peak} at around 13–16 °C indicated the presence of low melting fat crystals in all the samples. Suc had significantly higher ($P < 0.05$) T_{onset} and ΔH_{melt} compared to the other sucrose-free samples. This indicated its higher amount of low-melting fat crystal in the Suc sample, causing a delay in the start of the melt, and more energy was needed for the sample to meltdown. The larger particle size of the Suc sample (Table 2) might allow more fatty acids to move freely in the chocolate matrix. This might change the fatty acid distribution, and increase the low-melting fat crystallinity in the sample. Sarfarazi & Mohebbi (2020) stated that the weak inter-particle interactions of the chocolate sample could increase the free-moving fat in the chocolate matrix. The higher mobility of TAG could affect the crystallization in the dark compound chocolate (Rigolle et al., 2015). Nevertheless, the impact might be marginal because the high melting fat crystal dominated the melting profile of the samples. This was indicated by the higher ΔH_{melt} of the high-temperature melting peak than the low-temperature melting peak.

3.5. Texture analysis

The hardness of the dark compound chocolate samples is shown in Fig. 4. Except for EryI, all the sucrose-free samples ranged between 12 N and 14 N, which was in accordance with the previous studies (Saputro et al. 2017; Philip, Ohene & Dewettinck 2015). Sucrose replacement significantly ($P < 0.05$) increased the hardness value in dark compound chocolate compared to the Suc sample (8.285 ± 0.423 N). This was likely attributed to the smaller particle size (Table 2) and narrower particle size distribution (Fig. 2) of the sucrose-free sample leading to the higher solid packing intensity and particle–particle interaction. This result was in accordance with Belscak-Cvitanovic et al. (2015), which stated that the hardness of chocolate was directly correlated with the particle size. In addition to particle size, the higher moisture content in the sucrose-free sample (Fig. 1) might also create strong sugar networks resulting in a higher force needed to penetrate the sample, as reported in the previous studies (Shourideh et al. 2012; Saputro et al. 2017).

However, EryI, showed a slightly ($P \geq 0.05$) lower hardness value than Suc, even though it had a significantly ($P < 0.05$) smaller particle size and higher moisture content compared to Suc. This could be related to Table 2, which showed that the EryI sample had a significantly ($P \geq 0.05$) higher ΔH_{melt} of 15.7 ± 0.2 J/g than the other sucrose-free sample at 16.4 ± 0.1 °C, indicating more fat crystal melted at room temperature. This might be due to the poor tempering of EryI leading to the increased amount of low melting fat crystal in the sample. As manual tempering was used in preparing chocolate samples, the rate of increasing or decreasing the samples' temperature was difficult to control manually. The EryI sample might not have had sufficient time to form the fat crystal at 27 °C attributed to its under-tempered condition. Such phenomena were similar to Shah, Jones & Vasiljevic (2010), which discovered that a poor degree of tempering might result in a low hardness value in sucrose-free chocolate despite its high solid packing density.

Therefore, in the case of well-tempered dark compound chocolate, the sucrose replacement could affect the hardness due to their difference in particle size and moisture content with sucrose. Although all the sucrose-free samples had similar hardness values ($P \geq 0.05$), TagI and TagP had higher potential for sucrose replacement in dark compound chocolate due to their closer hardness value to Suc.

3.6. Polymorphism

XRD analysis was used to identify fat polymorph in the samples and confirm the DSC analysis results. Peaks indicated by the black arrows in Supplementary material 4 were analyzed in this study as these peaks were related to the fat and were not observed in the control samples with only the sweeteners.

Sucrose-made chocolate samples displayed multiple peaks (Supplementary material 4a), indicating the presence of different types of crystals in the samples. A strong peak was detected at d = 4.58 & 4.52 Å,

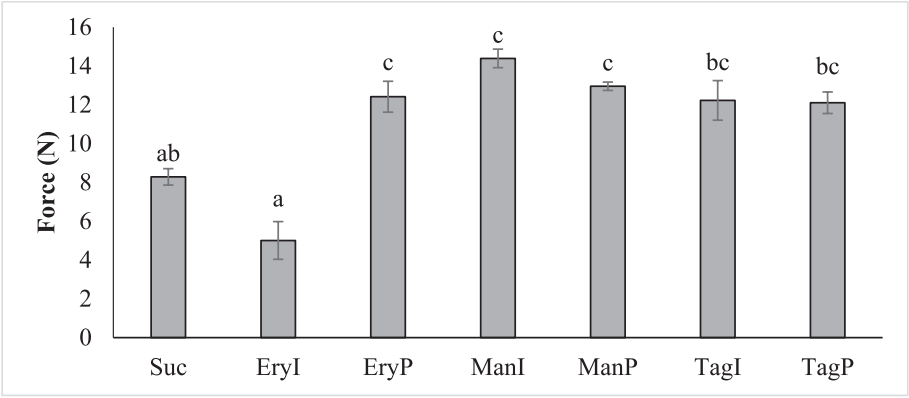

Fig. 4. The hardness of dark compound chocolates formulated with different sucrose replacer mixtures from the triplicate analysis (n = 3). [a, b, c] Different superscript letters indicate a significant difference ($P < 0.05$) detected between the samples.

indicating the presence of β polymorph (d = 4.5 to 4.6 Å) as the major fat crystal; weak peaks were detected at d = 4.26 and 4.35 Å, corresponding to the presence of α polymorph (d = 4.24 Å) and β'polymorph (d = 3.8 to 4.3 Å) in the sample. β polymorph was the desired fat crystal in dark compound chocolate to obtain chocolate with a melting temperature of around 34 °C, with a good snap, and a stable shelf-life. These peaks can also be detected in all the sucrose-free dark compound chocolate samples (Supplementary material 4b-d), indicating the presence of similar fat polymorphs in all the samples.

Despite the peaks indicated earlier, a weak peak was detected in the sucrose-made dark compound chocolate sample at d = 3.77 Å, corresponding to γ polymorph (d = 3.7 or 4.19 Å); however, this peak was not detected in the sucrose-free sample. The presence of γ polymorph might be attributed to the poor compatibility between HPKO and CB due to the difference in TAG. CB mainly consists of symmetrical TAG: glycerol-1,3-dipalmitate-2-oleate (POP; P = palmitic, O = oleic; 13.6–15.5%), glycerol-1-palmitate-2-oleate-3-stearate (POS; S = stearic; 33.7–40.5%) and glycerol-1,3-distearate-2-oleate (SOS, 23.8–31.2%) (Bootello et al. 2012); while the HPKO contains tri-laurin (LLL) as the predominant TAG. The two incompatible fats might tend to separate leading to the occurrence of eutectic which is the dilution of solid fat content in chocolate, causing the delayed onset of TAG crystallization. The absence of γ polymorph in the sucrose-free sample might be attributed to their high solid packing intensity. The smaller particles might reduce the movement of TAGs and act as nucleating agents, promoting their heterogeneous nucleation and crystallization from the γ polymorph to a more stable fat crystal. This result was in accordance with Kalic et al. (2018) who found that the onset of crystallization was accelerated by reducing the particle size in cocoa powder/sugar/palm oil blend, forming a more dense and homogenous structure of fat crystal network.

3.7. Microstructure analysis

The micrographs (Supplementary material 2) showed that the sucrose-containing sample had a larger particle size than the sucrose-free sample under the polarized light microscope. This result confirmed the earlier particle size distribution analysis (Table 2), which showed that the sucrose-containing sample had the largest particle size. Minimal variations were observed among all the sucrose-free samples. The small particle size in the sucrose-free sample might cause an extensive particle–particle interaction, resulting in a higher Casson yield value (Fig. 3) and hardness value (Fig. 4) than the sucrose sample. Servais, Ranc & Roberts (2004) stated that the number of small particles and their mechanical interactions could affect the chocolate yield value. This mechanical interaction is the inter-particle contact that depends on the chocolate's mean particle size and specific surface area. Afoakwa, Paterson, Fowler & Vieira (2009) also stated that the samples with less open structures reduced the fat to fill in the void spaces between the crystal network and thus increased the resistance to flow in the sample.

Despite particle size, the crystal shape of the sucrose replacer might also affect the solid packing intensity of the sample. ManI and ManP (Supplementary material 2d-e) had elongated shape crystals, while the other sucrose replacer mixtures displayed crystalline particles but were less uniform in shape. The crystal shape of mannitol crystal could result in a higher solid packing intensity and thus increase the rheological and hardness values of the samples. This was similar to Kaialy & Nokhodchi (2013), which showed that the elongated mannitol crystal samples had smaller convexity and higher compactness values than the irregular-shaped crystal. The variations in sugar crystal shape could be attributed to the intrinsic crystal properties such as intramolecular space and attachment energies between the crystal layers (Docherty, Clydesdale, Roberts & Bennema, 2000). Thus, the microstructure of the sucrose-free dark compound chocolate could be affected by both the size and crystal shape of the sucrose replacer mixtures, consequently affecting the chocolate properties. The ManI and ManP might not be suitable for sucrose replacement due to their small particle size and elongated crystal shape, increasing the viscosity and hardness of dark compound chocolate.

4. Conclusion

Sucrose replacement could cause a significant impact on the rheological and texture properties and a minimal impact on the crystallization and melting properties of dark compound chocolate. The changes brought by the sucrose replacement in dark compound chocolate might be due to the replacer's hygroscopicity, particle size, and shape. A sucrose replacer with a smaller particle size (18.6—21.9 μm) and elongated crystal shape resulted in higher solid packing intensity and, thus, higher rheological and hardness values. The small particle size might also act as a site for the crystallization of the TAG from an unstable γ polymorph to a more stable polymorph. This impact might be marginal because the melting profile of all samples was dominated by the stable high melting crystal polymorph, indicated by the higher ΔH_{melt} of the high-temperature melting peak than the low-temperature melting peak in the DSC thermogram.

Despite particle size, the hygroscopic sucrose replacer mixture could increase the moisture content during production and storage, thus elevating the chocolate's hardness and viscosity due to particle agglomeration. Nevertheless, moisture content and hardness were not part of the selection criteria of the sucrose replacer mixture in this study because all the sucrose-free samples were below the 1% moisture content tolerance limit and had similar hardness values. Among all sucrose replacers, EryP is suitable as a sucrose replacer mixture in dark compound chocolate as it exhibited a similar Casson yield value as sucrose-

containing samples; it attributed to their similar specific surface area and comparable D90 value as well as the non-elongated crystal shape in both samples.

CRediT authorship contribution statement

Keat Yi Wong: Conceptualization, Methodology, Investigation. **Yin Yin Thoo:** Validation. **Chin Ping Tan:** Validation. **Lee Fong Siow:** Supervision.

Declaration of Competing 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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2023.137118.

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