Relation of Fat Bloom in Chocolate to Polymorphic Transition of Cocoa Butter

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ABSTRACT: A special chocolate with spray-dried sugar (50:50 w/w sucrose/20 Dextrose Equivalent corn syrup solids) was made to study the polymorphic changes in cocoa butter crystals using X-ray diffraction. Anhydrous milk fat (AMF) and high-, middle-, and low-melting milk fat fractions were used to replace 2% (w/w) of cocoa butter. Chocolates were tempered, and the consistency of temper among chocolate samples was verified by a temper meter. Chocolates were cycled between 19 and 29°C at 6-h intervals to induce fat bloom. The special chocolates were analyzed by X-ray spectroscopy and colormeter.

X-ray analysis on the special chocolates showed polymorphic transition from the βV to the βVI form of cocoa butter. After a lag phase, the percentage of the βVI form rapidly increased. However, the sample made with the high-melting milk fat fraction transformed slowly to βVI. Visual bloom appeared rapidly on the special chocolates made with AMF, middle- and low-melting fractions, whereas visual bloom was very slow to appear on the special chocolates made with high-melting milk fat fraction and on the cocoa butter control. The commercial chocolate responded consistently; the control bloomed rapidly, the AMF exhibited some bloom resistance, and the high-melting fraction inhibited bloom. Despite the βV to βVI transition, the control chocolates with amorphous sugar did not bloom. Since the only difference in the chocolates was sugar microstructure, differences in bloom formation were caused by the microstructure, not the polymorphic transition.
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KEY WORDS: Bloom, chocolate, milk fat, milk fat fractions, polymorphic transition.

Fat bloom is a major concern to the chocolate industry because it compromises both visual and textural quality. Fat bloom manifests itself as a white film and general dulling of the characteristic surface gloss. Several situations can lead to the development of fat bloom. If chocolate is not properly tempered it will bloom because stable βV crystals will not be present (1). Cooling too quickly after the tempering process will cause bloom because the formation of unstable polymorphs will be promoted (2). Storage temperatures that are too high or fluctuate between hot and cold will cause bloom owing to the melting and recrystallization of the cocoa butter (3,4). Abusive storage temperatures can cause the solid state transformation of cocoa butter from the βV to βVI form (3–5). Foreign fats with lower solid fat content than cocoa butter can interfere with the formation of the stable βV form during tempering, and may accelerate the development of bloom during storage. Adenier *et al.* (6) argue that the liquid triacylglycerol (TAG) phase, because of its relative importance and quantitative variation as a function of temperature, is the essential mechanism of bloom formation. The liquid TAG phase allows the migration of fat to the surface where it recrystallizes and causes fat bloom.

Theories of bloom development during storage fall into two main groups: phase separation and polymorphic transformation. The phase separation theory is based on separation of the high- and low-melting TAG in cocoa butter, with the high-melting TAG group causing bloom (5–7). Becker (7) proposed that under metastable conditions, high-melting fractions of the cocoa butter are able to separate out of the matrix. This theory was used to explain the difference in composition and melting profiles between the fat bloom and the chocolate. Kleinert (2) refuted this hypothesis, stating that pseudometastable regions are formed in chocolate that is cooled too quickly.

Schlichter-Aronhime and Garti (5) revived the phase separation theory, stating that the βVI form of cocoa butter is not a true polymorphic form but a separation of the solid β crystal into two distinct phases. The separation is caused by the limited incorporation of liquid TAG groups as the density of the crystal lattice increases. Therefore, the transformation from a lower- to a higher-melting point form is accompanied by phase separation (5).

The polymorphic transition theory is based on fat bloom forming as cocoa butter crystals change from an unstable to a more stable polymorphic form. The six polymorphic forms were described by Wille and Lutton (8) using X-ray diffraction spectroscopy. They suspected that the transformation of cocoa butter from the βV to βVI form caused fat bloom because they found the βVI form whenever they found bloom. The transformation from βV to βVI is generally found in well-tempered chocolate during storage. During storage, elevated and fluctuating temperatures increase the rate of transformation (3,9,10). Cebula and Ziegleder (3) found that storage of well-tempered chocolate at 5°C effectively inhibited the transformation from βV to βVI and stopped bloom devel-

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opment. However, Adenier *et al.* (6) did not always find bloom with the transformation from βV to βVI in well-tempered chocolate. Thus, there is still a question as to the exact mechanism for bloom formation in chocolate.

Milk fat and milk fat fractions successfully inhibit bloom, are an inexpensive cocoa butter substitute, and can be legally added to pure chocolate products (both dark and milk chocolate). Lohman and Hartel (11) and Wood (12) have shown in recent work that high-melting milk fat fractions show the greatest inhibition of bloom in chocolate and avoid the detrimental softening of chocolate often caused by addition of milk fat. However, the mechanism by which milk fat and its fractions inhibit bloom is still unknown.

Although there have been many observations to support each theory, the exact mechanism of bloom formation has not been confirmed. Through various investigations into the bloom mechanism, many methods for the prevention of bloom have been suggested. The mechanisms by which bloom develops and how milk fat and its fractions inhibit bloom were evaluated in this study through both visible and polymorphic analysis of chocolate.

EXPERIMENTAL PROCEDURES

Materials. Anhydrous milk fat (AMF) and three milk fat fractions were obtained from the Center for Dairy Research at the University of Wisconsin, Madison, using a pilot plant dry fractionation unit. The three milk fat fractions were differentiated by melting profile as high (labeled 25S)-, middle (25L)-, and low (16.4L)-melting. The nomenclature of S or L after the fraction refers to the solid or liquid state of the fraction at the crystallization temperature (°C).

Two cocoa butters were used: a middle-melting Ivory Coast cocoa butter (dropping point of 26.9°C) and a higher-melting cocoa butter (dropping point of 29.0°C). The Ivory Coast cocoa butter was obtained from the Guittard Chocolate Co. (Burlingame, CA) and the higher-melting cocoa butter (of undetermined origin) was obtained from the Ambrosia Chocolate Co. (Milwaukee, WI). Commercial chocolate could not be used for the X-ray analysis because of the interference of crystalline sucrose. Therefore, chocolates were made with a spray-dried amorphous sugar in place of crystalline sucrose. Chocolates were made with two cocoa butters, with addition of AMF and three milk fat fractions to replace 2% of the cocoa butter in the chocolate. Control chocolates were made with the two cocoa butters and no milk fat addition. The amorphous sucrose powder was prepared by dissolving a 50:50 (w/w) mixture of sucrose and corn syrup solids (20 Dextrose Equivalent) in water to make a 40% (w/w) solution. This mixture was then spray-dried and sieved between 44 and 80 μm sieves (E.H. Sargent & Co., Chicago, IL) to give a reasonably representative particle size for chocolate. To confirm the effect of the microstructure of the sugar, three chocolate samples were made using crystalline sucrose which was ground and sieved to a representative particle size. These chocolates were prepared with the Ivory Coast cocoa butter and could not be analyzed using X-ray diffraction. Crystalline sucrose was prepared using a Janke & Kunkel (Staufen im Breisgau, Germany) grinding mill and sieving to a representative particle size.

Chocolate preparation. Chocolate samples (500 g) were prepared by combining the defatted cocoa powder, sieved sugar (either amorphous or crystalline), melted (60°C) cocoa butter, and added fat. These ingredients were mixed together at 1000 rpm at 60°C for 20 min in a 1-L jacketed tempering beaker (Cole-Palmer, Chicago, IL). Lecithin was added and the chocolate mixed for another 10 min at 60°C. A coated paddle stirrer (Cole-Palmer) was used to mix the samples and a Master Servodyne motor head and controller (Cole-Palmer) controlled the rpm and monitored viscosity. A method modified from Kleinert (13) was used to temper each chocolate sample, using a copper-constantan thermocouple (Omega Engineering, Stanford, CT) to monitor temperature. Melted chocolate at 60°C was cooled initially to 26°C and held there until viscosity approached a constant value (about 1 to 1.5 h). Temperature was raised to 32.8°C to melt unstable crystals and promote formation of the stable polymorph. After viscosity had reached a constant value, temperature was again lowered to 29°C to allow further crystallization and allow the stable seeds to grow. After constant viscosity was attained, temperature was raised to 32.3°C for 30 min to obtain a chocolate with suitable viscosity for molding. Tempered chocolate was poured into a 12-cavity (disc-shape) plastic mold. About 12.5 g of chocolate was poured into each mold. The filled molds were stored at 13.5°C for 24 h to obtain properly tempered chocolate discs for further analysis.

Once prepared, all chocolates were stored in a custom-built bloom chamber with temperature cycling between 19 and 29°C at 6-h intervals to induce development of fat bloom. Samples were taken every 48 h. The chocolates made with amorphous sugar were analyzed for X-ray spectra and whiteness index by colormeter. The commercial chocolates were analyzed using the colormeter.

Chocolate analysis. A Hunterlab Color Quest (model Q45/0; Hunter Association, Inc., Reston, VA) was used to quantify the changes at the surface of the chocolate samples (11). The colormeter measured the *L*, *a*, and *b* values for each chocolate, which were converted to whiteness index (WI) values according to Equation 1:

$$WI = 100 - (100 - L)^2 + a^2 + b^2 \quad [1]$$

The colormeter was calibrated with a black-and-white standard plate prior to any sample measurement. Each disc of chocolate was then placed nonmolded side down on the sample portal, and *L*, *a*, and *b* values were measured. The chocolate disc was rotated approximately 90° and read again; this was repeated eight times to ensure that the entire surface of the sample was measured. Final *L*, *a*, and *b* values were automatically calculated with Hunterlab Color Quest software.

A Nicolet I_2v Polycrystalline X-ray Diffractometer (Nicolet Instrument Corp., Madison, WI) was used for the X-ray

analysis. The radiation source was a CuKα radiation source with a wavelength of 1.542 nm. Siemens Polycrystalline Software Package (Micro-vax release 2.41, 1989) was used to analyze all data. Diffraction intensity was measured between the angles (2θ) of 5 and 30°, with a count time of 2 s and a step width of 0.05° per count. Once a sample was analyzed, the diffraction pattern was contrasted with the curves from a standard plot. The *d* values for the short spacing region (15–25° 2θ) were also calculated by the software according to the Braggs equation. The *d* values were used to confirm the different polymorphic forms using the published values of D'Souza *et al.* (14).

Teflon sample holders (Siemens Industrial Automation, Inc., Madison, WI), which held approximately 0.75 g of material, were used for the X-ray analysis. The large chocolate discs were ground to fit into the sample holder. Samples were prepared by cutting each disc in half and placing it in a mortar and pestle (Coors, Golden, CO). Liquid nitrogen was then poured into the mortar and pestle, and the sample was ground to a fine powder. Liquid nitrogen was used so that there would be no change in the crystalline nature of the cocoa butter during sample preparation. Approximately 0.75 g of the ground chocolate was quickly pressed into the sample holder. All samples were analyzed at 18 ± 2°C.

To use X-ray spectroscopy to quantify changes in chocolate, the X-ray spectra of each polymorph and their blends were determined. Quantities of both the βV and βVI polymorphs were prepared by tempering cocoa butter using IUPAC method 2.150 (15) to obtain the βV polymorph. Temperature cycling of the tempered cocoa butter caused the βVI polymorph to form. The identity of the polymorphs was confirmed by comparison with published X-ray spectra (3,8,14). Samples with ratios of βV/βVI of 100:0, 80:20, 60:40, 40:60, 20:80, and 0:100 were prepared. The spectra were then arranged to show the effect of polymorphic change in cocoa butter. The determination of polymorphic form was conducted using visual comparison of the X-ray spectra for each chocolate sample with the X-ray spectra in Figure 1. Attempts to quantify the percentage of βVI present using ratios of peak heights was unsuccessful since the differences in these ratios were small at low levels of βVI. Thus, the sensitivity based on comparing ratios of peak heights was unsatisfactory and a more qualitative comparison was chosen. Schlichter *et al.* (16) published a plot similar to Figure 1 with percentages of βVI cocoa butter added to βV cocoa butter and used a similar visual approach. Schlichter *et al.* (16) determined error to be less than 10% for measurements made from visual evaluation of their figure. The spectra in Figure 1 correlate very well with those in the literature; therefore, the percentage error should be similar and no more than 20%.

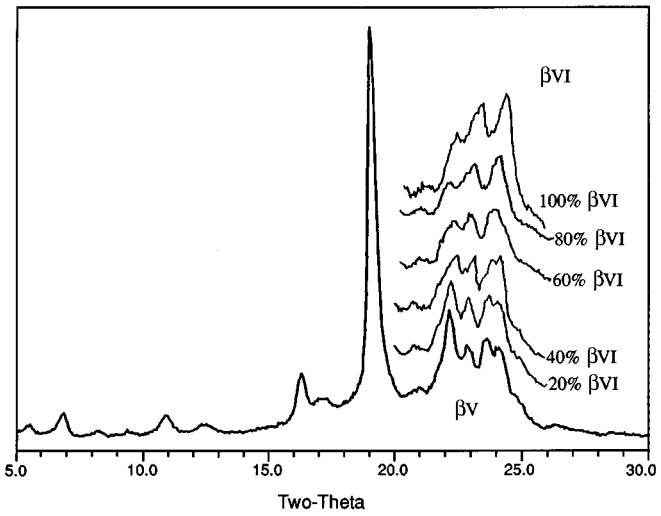

FIG. 1. Comparison of X-ray diffraction spectra for mixtures of the βV and βVI polymorphic forms of cocoa butter.

RESULTS AND DISCUSSION

To measure the polymorphic transition in the samples over time, X-ray diffraction analysis was used. X-ray analysis has been used to determine the polymorphic forms of cocoa butter in chocolate after storage or treatment (3,6,8), but it has not been used to directly characterize the ongoing changes in polymorphic form during storage of chocolate. Figures 2 and 3 show the increases in content of βVI during storage of chocolates made with high- and middle-melting (Ivory Coast) cocoa butters, respectively.

Chocolates made with Ivory Coast cocoa butter (Fig. 3) showed a lag phase of several days where there was no recognizable change in polymorphic form. This may be partly due to a lack of X-ray data for low addition levels (5, 10, or 15%) of βVI to βV. However, after the lag phase, all of the samples except chocolate made with the high-melting milk fat fraction exhibited a gradual and approximately linear increase in the percentage of βVI. As expected, the high-melting milk fat fraction was able to slow the polymorphic transformation in cocoa butter. However, AMF and the middle- and lower-melting fractions had virtually no effect. The chocolates made with high-melting cocoa butter (Fig. 2) were similar to the Ivory Coast chocolates, although there was less of a lag phase. In fact, the chocolate made with the low-melting milk fat fraction exhibited no lag phase and the chocolates made with AMF and middle-melting milk fat fraction had less pronounced lag phases than the Ivory Coast cocoa butter. The final level of βVI developed was about the same in chocolates made with both cocoa butters.

Colormeter readings were taken in conjunction with X-ray diffraction analyses for both sets of chocolates in order to compare polymorphic changes with onset of visual whiteness. Colormeter results are shown in Figures 4 and 5 for chocolates made with high-melting and Ivory Coast cocoa butters, respectively. The chocolate made with low-melting milk fat fraction showed the greatest development of bloom in chocolates made with high-melting cocoa butter (Fig. 4). This was followed by the chocolate made with AMF, whereas the middle-melting fraction caused moderate inhibition relative to the low-melting milk fat fraction and AMF. The chocolates made with Ivory Coast cocoa butter and low-melting milk fat frac-

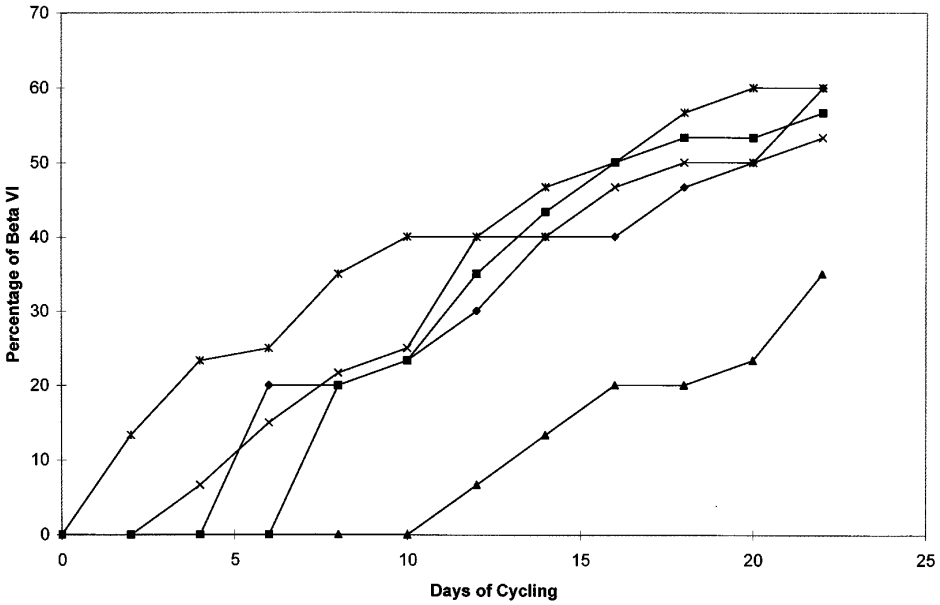

FIG. 2. Increase in ratio of βVI to βV polymorphic form during storage in chocolates made with amorphous sugars and a high-melting cocoa butter (◆), with addition of either 2% anhydrous milk fat (AMF) (■), a high-melting milk fat fraction (25S) (▲), a middle-melting milk fat fraction (25L) (×), or a low-melting milk fat fraction (16.4L) (✳). Storage temperature of chocolates cycled between 19 and 29°C every 6 h.

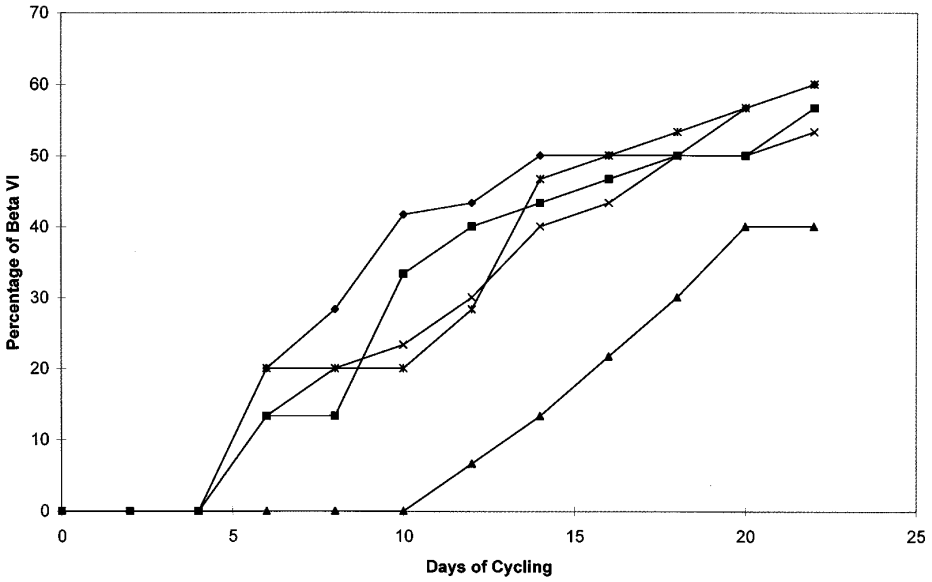

FIG. 3. Increase in ratio of βVI to βV polymorphic form during storage in chocolates made with amorphous sugars and a middle-melting cocoa butter (Ivory Coast) (◆), with addition of either 2% anhydrous milk fat (AMF) (■), a high-melting milk fat fraction (25S) (▲), a middle-melting milk fat fraction (25L) (×), or a low-melting milk fat fraction (16.4L) (✳). Storage temperature of chocolates cycled between 19 and 29°C every 6 h.

tion or AMF had the greatest level of bloom development. Addition of middle-melting milk fat fraction caused moderate bloom inhibition relative to the low-melting milk fat fraction and AMF. In both chocolates, addition of high-melting milk fat fraction resulted in the greatest resistance to bloom development. The control chocolates in both cases showed very little visual bloom development. The results of the control chocolate are not consistent with the results reported previously by Lohman and Hartel (11) or Wood (12). Typically, the control chocolate without added milk fat or milk fat fractions exhibits the greatest and most rapid development of bloom when compared to chocolates made with added milk fat.

The behavior of the control chocolate is difficult to explain, especially in light of the results obtained from the X-

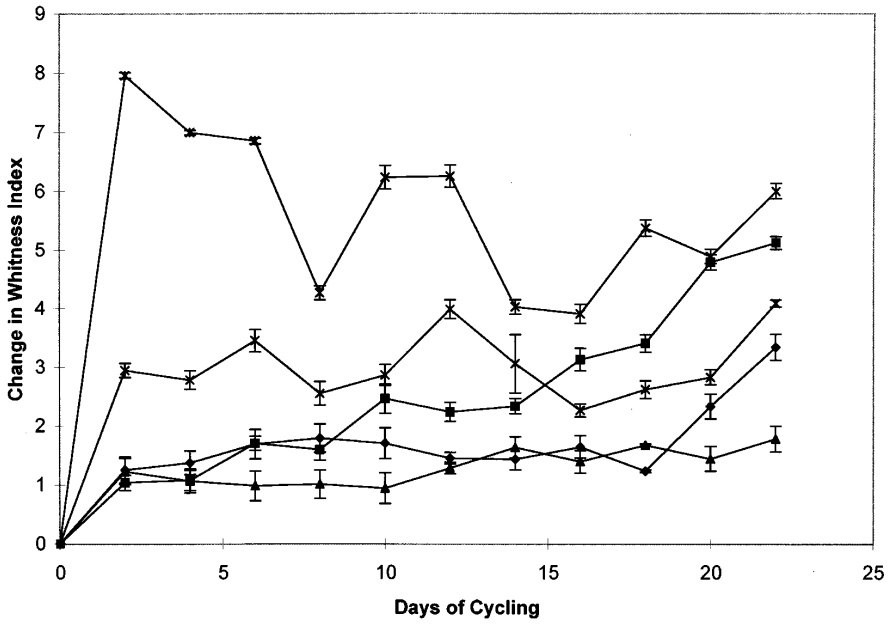

FIG. 4. Increase in whiteness, as change in whiteness index measured by colormeter, during storage of chocolates made with amorphous sugars, a high-melting cocoa butter (◆), and addition of either 2% anhydrous milk fat (AMF) (■), a high-melting milk fat fraction (25S) (▲), a middle-melting milk fat fraction (25L) (×), or a low-melting milk fat fraction (16.4L) (✳). Storage temperature of chocolates cycled between 19 and 29°C every 6 h. Vertical bars represent standard deviations.

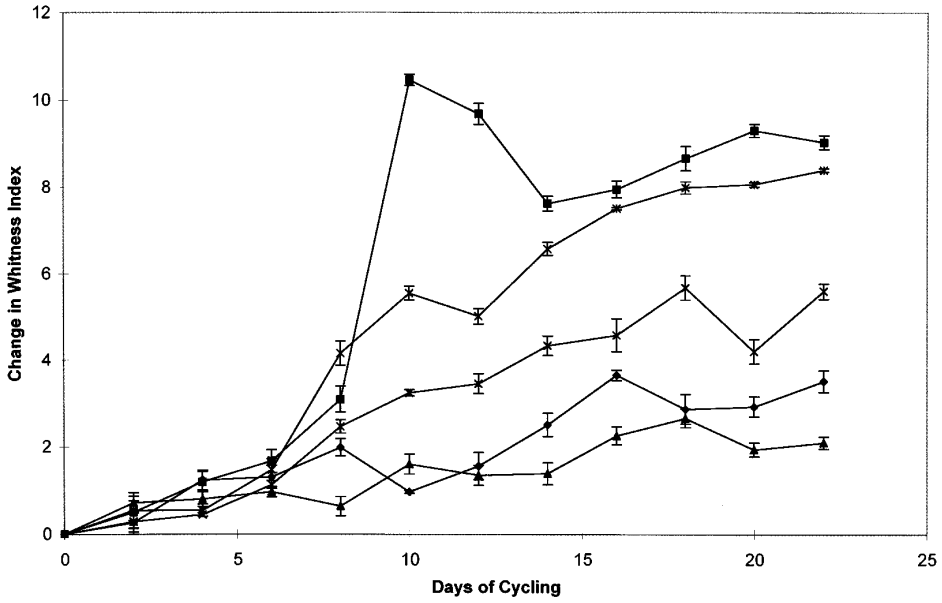

FIG. 5. Increase in whiteness, as change in whiteness index measured by colormeter, during storage of chocolates made with amorphous sugars, a middle-melting cocoa butter (Ivory Coast) (◆), and addition of either 2% anhydrous milk fat (AMF) (■), a high-melting milk fat fraction (25S) (▲), a middle-melting milk fat fraction (25L) (×), or a low-melting milk fat fraction (16.4L) (✳). Storage temperature of chocolates cycled between 19 and 29°C every 6 h. Vertical bars represent standard deviations.

ray analyses. A consistent increase in content of the βVI polymorph was seen for both of the control chocolates, whereas the colormeter analysis showed no visual bloom formation. The microstructure of sugars in the chocolate was believed to be the cause of the discrepancy in visual bloom development and polymorphic transitions. To test this theory, crystalline sucrose was ground to create a sugar consistent with commercial chocolate on the basis of microstructure. These samples were prepared identically to the samples made for the X-ray analysis but were analyzed only for changes in whiteness index during storage.

Figure 6 shows the colormeter results for these samples.

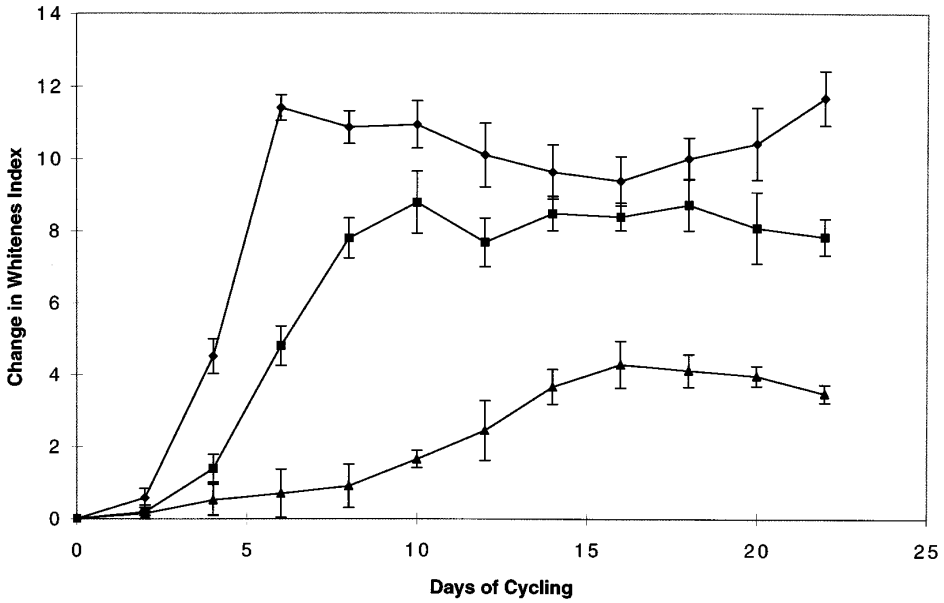

FIG. 6. Increase in whiteness, as change in whiteness index measured by colormeter, during storage of chocolates made with crystalline sucrose, a middle-melting cocoa butter (Ivory Coast) (◆), and addition of either 2% anhydrous milk fat (AMF) (■) or a high-melting milk fat fraction (25S) (▲). Storage temperature of chocolates cycled between 19 and 29°C every 6 h. Vertical bars represent standard deviations.

The results are consistent with those previously reported (11, 12) with the control sample having the greatest rate of bloom formation, AMF showing some inhibition and high-melting milk fat fraction dramatically inhibiting bloom formation. Since these samples were prepared and tempered identically to the modified chocolates, the microstructure of the sugar seems to be the only explanation for the observed discrepancies.

These results clearly show that the onset of polymorphic transformation from βV to βVI can not be the "cause" of visual bloom. We can hypothesize the following general series of events that must occur for visual fat bloom to develop during storage of well-tempered chocolates. First, there must be a mechanism to promote migration of liquid fat to the surface. Slight temperature fluctuations cause a resultant change in solid fat content as the system strives to maintain the equilibrium fat phase volume. As the fat crystals melt and grow with temperature fluctuations, the liquid fat may be "pumped" to the surface where the thermal fluctuations are the highest. Cracks in the chocolate due to rapid cooling are likely to promote this liquid migration, as suggested by Kleinert (2). The nature of this liquid fat phase is also important as there may be some solubility of high-melting components in the more liquid, low-melting components (6). At the surface, the high-melting components will change solubility as temperature fluctuates, resulting in recrystallization of the fat phase. However, this recrystallization, which may be due to a combination of solubility effects and fluctuating solid fat content of the main crystalline phase, is not by itself sufficient to cause visual bloom. There must be a type of surface available to promote recrystallization in the form of needle and spike formation of crystals extending out of the surface of the chocolate. These extensions cause diffuse reflection of light, leading first to dulling and eventually to onset of visual bloom. The rate of fat bloom formation may be influenced by any of these individual events.

Any explanation for the results shown in this study must relate to the microstructure of the sucrose in this chocolate. The amorphous sugar mixture consisted of smooth, mostly spherical sugar particles. The microstructural shape was very different from the sharp edges evident in the ground, crystalline sucrose. The rounded surface of the amorphous sugar mixture might affect the formation of bloom in a number of ways. The round shape of the sugar may allow for tighter packing of the sugar crystals in this chocolate. This may interfere with the migration of liquid TAG molecules to the surface and consequently reduce the development of bloom. Since the AMF and low- and middle-melting milk fat fractions melted near or below the temperature of heat cycling, it is reasonable that they would be more liquid and therefore more mobile. The increased mobility of the fat allowed the liquid TAG molecules to migrate to the surface. As the liquid TAG molecules migrate, high-melting TAG fractions of the cocoa butter are carried to the surface in the liquid phase. Once at the surface, the high-melting component recrystallizes and grows into sharp crystals which appear as bloom. The fact that the high-melting milk fat fraction inhibited polymorphic transformation is in itself an explanation for the lack of bloom. Another potential explanation is that the higher melting temperature of the fat makes it less susceptible to melting during any given heat cycle, resulting in less liquid TAG migration to the surface.

There is also a possibility that the interaction of lecithin at the surface of the sugar particle influences formation of bloom crystals. The lecithin, which aligns at the surface of

the sugar particles in chocolate, may have different surface energetics depending on the nature of the sugar surface. This may influence bloom formation by either affecting the migration of liquid cocoa butter TAG through the chocolate or by changing the recrystallization process at the surface.

In this study, we have clearly demonstrated that the onset of formation of the βVI polymorph of cocoa butter is not the "cause" of visual bloom formation, but rather is one of the consequences. Chocolates containing significant quantities of the βVI polymorph can still appear to be visually unbloomed. However, the mechanism(s) of bloom formation and the factors that influence the onset of bloom remain somewhat unclear. Further work is warranted to fully understand when and where each of the steps required for bloom formation is important.

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