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Composition, Structure, and Color of Fat Bloom Due to the Partial Liquefaction of Fat in Dark Chocolate

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Abstract A type of fat bloom, which had not previously been fully characterized, was investigated to identify the state of its existence and its formation mechanism. Samples of bloom on solid chocolate resulting from the partial liquefaction of fat during temperature variations were analyzed to determine the crystal characteristics, fat contents, and triacylglycerol (TAG) compositions. Also, observation and elemental analyses were performed by scanning electron microscope with an energy-dispersive X-ray spectrometer, color analyses of minute regions were made by using PARISS®, and Fourier-transform infrared (FT-IR) analyses were performed. The dark- and light-brown areas did not show any differences in fat content or TAG compositions that could lead to the observed color differences. Although differences in component distributions were noted in micrometer-sized regions, no relation to the colors was confirmed. The bloom samples in this study and bloom developed without a tempering process resembled each other in the tone of color at their discolored regions, but the states they adopted differed from one another. It is suggested that the color in this type of bloom was affected by the roughness and/or porosity of the microstructure and could also be a result of the coarsened fat crystal network and of the liquid fat migration.

Keywords Chocolate · Cocoa butter · Color · Fat bloom · FT-IR · Liquid fat · PARISS® · SEM–EDS · TAG composition · XRD

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Introduction

The formation of a fat bloom on chocolate adversely affects the appearance and texture of the chocolate, and is a major problem in the confectionery industry. Although a number of detailed studies on fat bloom have been conducted over a long period, much remains to be discovered regarding why blooms exist and the mechanism for their formation. A variety of bloom types can occur [1–4], depending on the type of finished chocolate product and the storage conditions, and this variation in causes and consequences makes it difficult to obtain a better understanding of bloom phenomena. The generally accepted perception is that visual fat bloom is a consequence of separation of cocoa butter to the surface of the chocolate [5–8]. The typical crystal sizes of about 4–5 μm which scatter light, gives the chocolate surface a whitish appearance [5]. This separation is accompanied by the transformation of cocoa butter crystals [8]. This change takes place not only when cocoa butter of form βV transforms to form βVI [9, 10], but also when unstable crystals transform to βV. Thus, there is no dependency on a specific polymorphic form [8]. Recently, however, another form of visual fat bloom has been reported [4, 11, 12]. This type develops when the fat content is unevenly distributed, and the

portions with a lower fat content become light colored. This type of bloom occurs when no seed crystals are formed; for example, when solidification occurs without a tempering process.

In this study, we focused on a new type of visual fat bloom that is different to the conventional types of bloom caused by cocoa butter separation or bloom caused by uneven fat distribution, as recently reported. The new type of fat bloom is characterized by the appearance of streamline patterns or plain light-brown tones. By using solid dark chocolate containing cocoa butter, the composition of the bloom and other states attributable to the partial liquefaction of fats were studied, differences between dark- and light-brown sites were investigated, and the formation mechanism was inferred.

Experimental Procedures

Chocolate Preparation and Bloom Development

Chocolate containing 38% cocoa butter was prepared with refining by a roll refiner and conching from sugar, cocoa mass, cocoa butter, and lecithin. A solid chocolate sample was prepared with tempering. The tempering procedure was as follows: a bowl of chocolate melted at 60 °C was cooled to about 40 °C, and two-thirds of this chocolate was turned over on a marble slab with a scraper until the chocolate was cooled to about 28 °C. The chocolate was added back into the bowl and mixed thoroughly using a silicone spatula. The final temperature was then about 30 °C.

The tempered chocolate was poured into a polycarbonate mold and cooled to 15 °C for 30 min. The sample was removed from the mold and exposed to temperature cycling between 20 and 32 °C at 12 h intervals to induce the development of bloom as a result of the partial liquefaction of fat. After 1 week, a chocolate sample with bloom that was surrounded by a light-brown color was obtained (Fig. 1), and this was subjected to further series of analyses.

Analysis by X-ray Diffraction

The bloomed chocolate sample was carefully separated into dark- and light-brown parts using a scalpel and a stereomicroscope. The dark-brown part means the dark inner part of the chocolate piece exhibited in Fig. 1B and the light-brown part is the outer layer. Each part was set in an X-ray diffraction (XRD) sample holder, and its diffraction pattern was obtained between 2 and 30° (2θ, Cu Kα) by using a RAD-X (Rigaku, Tokyo, Japan). The sample holder holding each colored part was then heated at 50 °C to melt the fat component completely and the XRD diffraction pattern of each colored part was measured again. The measurement conditions were as follows:

voltage 40 kV, current 25 mA, scan speed 2 deg/min, Step scan 0.02°, Slit 1°(DS), 1°(SS), 0.3 mm (RS), 0.6 mm (Rsm).

All the experiments, unless otherwise indicated, were carried out at 20–24 °C. The diffraction pattern of the fat component in each colored part, which was obtained by subtracting the diffraction pattern measured at 50 °C from that measured at room temperature, was used to evaluate the fat in the bloomed chocolate [11].

Fat Content and Triacylglycerol Composition Analysis

The bloomed chocolate sample was separated into dark- and light-brown parts as described above. The fat content of chocolate before developing the bloom and of each colored portion of the bloomed chocolate sample were determined by Soxhlet extraction with diethyl ether. The triacylglycerol (TAG) composition

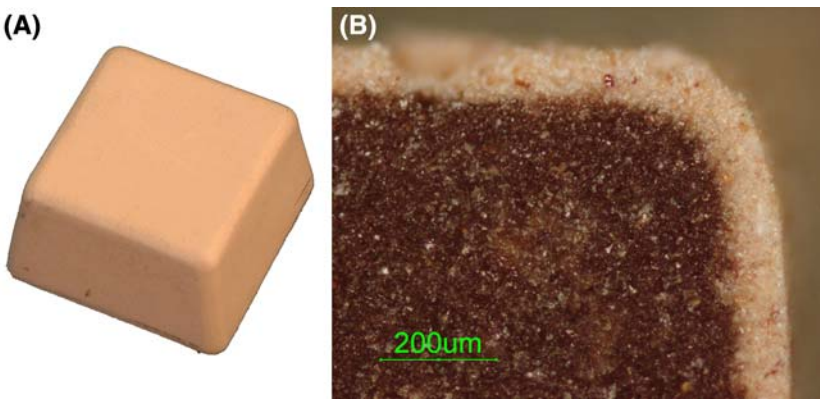

Fig. 1 **A** Chocolate sample bloomed due to the partial liquefaction of fat and **B** a micrograph of a cross section of the sample by optical microscopy

of the chocolate before developing a bloom and of each of the colored portion of the bloomed chocolate sample was determined by high-performance liquid chromatography (HPLC). Analysis for TAG was carried out according to previously described methods [11].

Observation and Analysis Using a Scanning Electron Microscope with an Energy Dispersive X-Ray Spectrometer

A cross section of the bloomed chocolate sample, prepared by cutting with a microtome, was studied by scanning electron microscopy with an energy-dispersive X-ray spectrometer (SEM–EDS; JSM-5600LV + JED2200, JEOL, Tokyo, Japan). The sample used for SEM–EDS was coated with an approximately 8-nm-thick layer of gold by using an ion coater. After coating, the dark- and light-brown parts of the bloomed chocolate sample were examined by SEM. EDS was used for element-mapping analysis in the same view.

Analysis with PARISS®

The prism and reflector imaging spectroscopy system (PARISS®) is an imaging spectrometer that can be used to determine microscopic spectral characteristics. This system acquires 240 spectra simultaneously in a slice of an image at wavelengths between 395 and 800 nm. Each slice is defined by the width of the entrance slit. A slice of the image projected by a microscope is focused onto the entrance slit of the spectrometer, strikes the first curved surface of a prism, is refracted, strikes the second surface, and exits to strike a spherical mirror. The light, with its wavelength dispersed, then returns through the prism to be focused onto a charge-coupled device array. An image of the entire targeted field is acquired by translating the sample across the slit aperture of the spectrometer. The number of acquisition points depends on the size of the entire targeted field and the magnification of the microscope's objective. All the acquired spectra can be classified by similarity, and spectrally classified mapping images can be produced.

The cross section of the bloomed chocolate sample used for PARISS® was prepared in a similar manner to that described above. In this study, the PARISS® system (LightForm Inc., NJ, USA) and an optical microscope (ECLIPSE ME600L; NIKON, Tokyo, Japan) with a 50× objective was used to obtain imaging spectra for the dark- and light-brown parts in the bloomed chocolate sample. The entrance slit used was 50 μm × 5 mm and the entire targeted field was 100 × 100 μm^2. The size of a single acquisition point was 1.0 × 0.4 μm^2. A tungsten halogen lamp was used as the source of incident light. The spectral reflectance was obtained by dividing the spectral intensity by that of a standard white board (Japan Color Research Institute). All the experiments were carried out at 20–24 °C.

Analysis by Fourier-Transform Infrared Spectroscopy

The cross section of the bloomed chocolate sample used for Fourier-transform infrared (FT-IR) spectroscopy was prepared in a similar manner to that described above. Reflectance spectra were acquired on an FT/IR-660Plus + IRT-30 (JASCO, Tokyo, Japan) spectrometer attached to a microscope, using the reflectance mode. The measurement area was 250 μm square for the dark- and light-brown parts of the bloomed chocolate sample. Scans were accumulated between 650 and 4,000 cm^{-1} with a 4 cm^{-1} resolution using a mercury cadmium telluride (MCT) detector. All the experiments were carried out at 20–24 °C.

Analysis of the Elimination of Bloom as a Result of Temperature Changes

A bloom sample with a cross section was prepared as described above, and small slices were shaved separately from the light-brown site at the surface of the sample by using a razor. These were separately heated to 32 °C and color changes of the specimens were observed. The bloom sample, whose appearance turned from light brown to dark brown as a result of heating, was held at 32 °C to retain the dark color and allow sampling from the surface. Then, 50-μm-thick specimens were sampled from the surface with a microtome, and their TAG compositions were analyzed as described above. The color at the 50-μm-thick site was light brown before heat was applied.

Results and Discussion

To elucidate differences between the dark- and light-brown parts in the bloom formed through partial liquefaction of fat, the compositions and states of each part were studied. We will discuss the results in comparison with those for bloom formed without tempering [11], which shows a close similarity in color tone to the light brown site. The states of fat crystals at each

site were examined by means of XRD. Figure 2 shows XRD patterns at (a) room temperature, and (b) 50 °C in (A) a dark part and (B) a light brown part, as well as XRD patterns accounting for the fat components based on the obtained data. The XRD patterns accounting for the fats were obtained by calculating the difference between the (a) and (b) intensities to eliminate the interfering effect of crystalline sugar on the XRD pattern of cocoa butter. Both the light-brown and dark-brown parts showed the diffraction patterns of cocoa butter βVI, and no clear differences were observed in the XRD patterns.

Table 1 shows the fat contents and TAG composition analysis results for dark- and light-brown parts of the bloom sample and for the chocolate before bloom formation. As shown in Table 1, the fat content is lower in the light-brown part than in the dark-brown part, and levels of *sn*-1,3-saturated acyl *sn*-2-oleoyl glycerols (Sat-O-Sat), which are the main components of cocoa butter, are higher. Although the fat content was lower in the light-brown part (31.0%), chocolate with this fat content is normally dark brown in color, and so this figure cannot explain why the color is light brown. Actually, if the light-brown part is removed, heated with stirring, and re-solidified, the color turns to dark brown. In contrast, in the bloom obtained without tempering [11], the light-brown color is retained. Similarly, the higher Sat-O-Sat level itself in the light-brown part than in the dark-brown part does not explain the reason why this part is light brown. From these results, it is concluded that although the light-brown part in the bloom caused by partial liquefaction of fat appears similar in color tone to that of bloom on untempered chocolate, the state of the bloom is

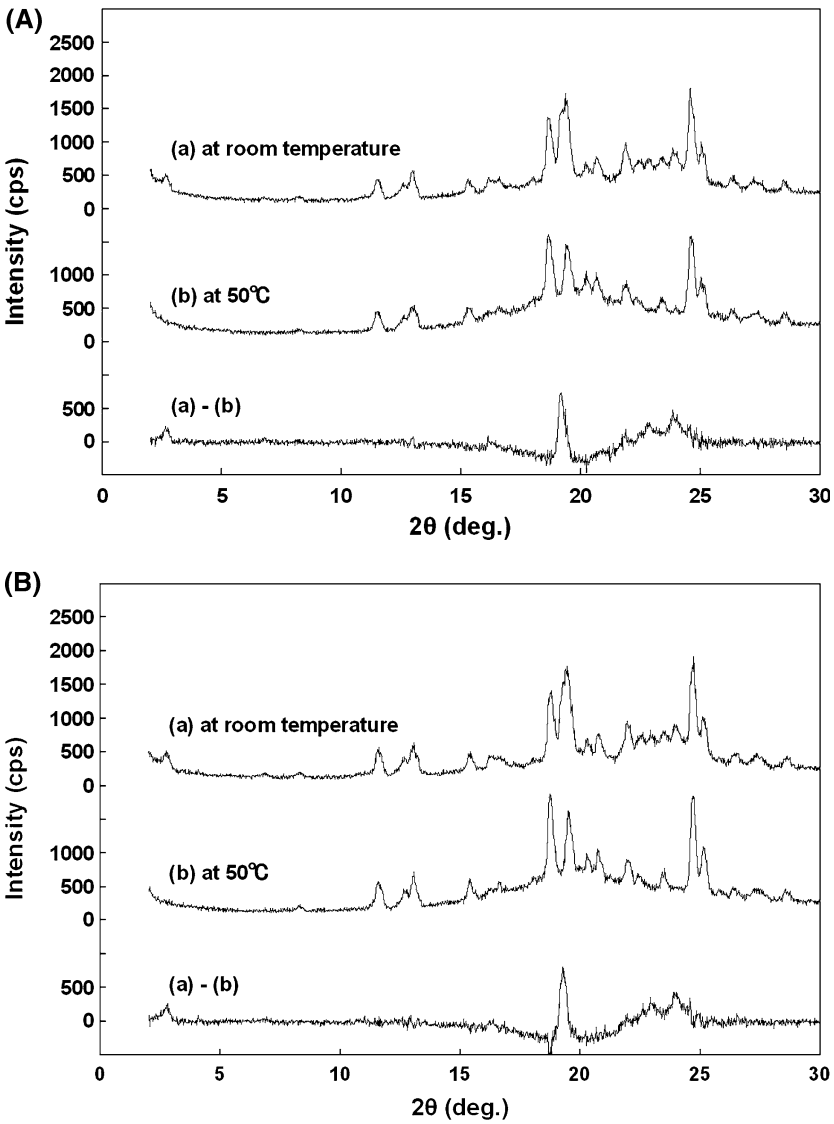

Fig. 2 X-ray diffractograms of **A** dark-brown and **B** light-brown sites in a chocolate sample bloomed through the partial liquefaction of fat, measured at (*a*) room temperature and (*b*) 50 °C; (*a* – *b*) is the difference spectrum when (*a*) is subtracted from (*b*)

Table 1 Fat and TAG concentrations (wt%) in chocolate before developing bloom as a result of the partial liquefaction of fat; dark and light brown parts of bloomed chocolate; and light-brown parts of bloomed chocolate heated to 32 °C in contact with the dark-brown parts that have turned dark brown

	Chocolate before bloom	Dark-brown part	Light-brown part	Light-brown part at 32 °C
Fat content	38.0	38.1	31.0	38.8
PLO	0.8	0.8	0.3	0.8
PLP	1.7	1.7	1.2	1.7
OOO	0.5	0.6	0.6	0.9
SLO	0.6	0.6	0.4	0.6
POO	3.0	3.1	1.9	3.3
PLS	3.1	3.0	2.2	3.1
POP	15.2	15.1	13.0	14.6
PPP	0.4	0.5	0.3	0.3
SOO	4.2	4.5	2.5	4.3
SLS	1.9	1.8	1.4	1.8
POS	35.9	35.5	37.1	34.6
PPS	0.4	0.4	0.5	0.4
AOO + ALS	0.4	0.3	0.5	0.4
SOS	25.4	24.8	30.7	25.6
SPS	0.5	0.5	0.6	0.5
SOA	1.4	1.4	1.6	1.4
other	4.6	5.4	5.2	5.7
Total	100.0	100.0	100.0	100.0
Sat-O-Sat	77.9	76.8	82.4	76.2

A arachidic acid, *L* linoleic acid, *O* oleic acid, *P* palmitic acid, *S* stearic acid

Sat-O-Sat POP, POS, SOS, SOA

different, and a lower fat content itself is not the reason it looks lighter color in the former case.

Distributions of fat and other chocolate components were analyzed on a micrometer scale. Figures 3 and 4 show a secondary electron image, backscattered electron image (compo), and carbon-atom-mapping images in the same field obtained by SEM–EDS of the cross sections of the dark- and the light-brown parts of the bloomed chocolate sample. The higher the average value of atomic numbers in a particular region of the specimen, the more backscattering of electrons occurs, and thus the image appears brighter. Therefore, in the backscattered electron image, fats, which have a lower average value of the atomic number, appear darker than other components. Also, the positions of fats can be confirmed by carbon–atom mapping. This is because fats contain higher proportions of carbon compared with other components. In Figs. 3 and 4, the dark brown regions of the bloomed chocolate sample have fats evenly distributed in relatively larger chunks, whereas in the light-brown regions, fats exist sparsely in thinner portions.

Next, PARISS® spectroscopy was performed to elucidate the relationships between the distribution of components of the chocolate on a micrometer scale and the color of the sample. Figures 5 and 6 show spectral reflectance and mapping images of the dark- and light-brown cross sections, classified by in terms of spectral similarity within the visible-light wavelength range. The colors in regions in the mapping image correspond to the respective colors in the spectral reflectance. According to Figs. 5 and 6, within the respective measuring range (100×100 μm^2), although some variations are observed in visible-light reflectance spectra depending on the measuring points, light-brown parts show a higher reflectance in spectral imaging than the dark-brown parts at all the measuring points. The measuring range at each point was 1.0×0.4 μm^2 and, therefore, at least at this resolution level or larger, color tendencies at dark- and light-brown parts are made apparent. This means that at the 1.0×0.4 μm^2 scale or larger, colors are not formed by an additive color mixing effect, where multiple colored lights are distributed in minute mosaic formations of a size below the spatial discrimination limit of the human eye [13], but that both the dark- and the light-brown regions are composed of elements of similar colors, which are minutely distributed, and consequently these elements make up the chocolate and the bloom colors, respectively. SEM–EDS analysis (Figs. 3, 4) showed distributions of fat and some other components in similar enlarged observation ranges of a micrometer order, whereas PARISS® (Figs. 5, 6) showed color tendencies in dark- and light-brown areas, where no relationships were found with the SEM–EDS result. Thus, no relationship exists between the components contained in the areas and colors that appeared, which suggested that other factors might determine how the colors arise.

Figure 7 shows FT-IR reflectance spectra of the cross sections of dark- and light-brown parts of a bloomed chocolate sample. The forms of the spectra differ markedly between the dark- and the light-brown parts, with the reflectance spectrum dominant in the dark-brown parts and the absorption spectrum dominant in the light-brown parts. In this study, such spectral differences in reflectance measurements may be attributable to differences in the surface textures of the samples. If the sample surfaces are smooth in terms of infrared wavelength, irradiated light tends to undergo regular reflection and therefore yields a reflection spectrum. On the other hand, for samples with rough surfaces, such as powdery substances, part of the irradiated light diffuses in the sample, then passes inside and is radiated outward, and the spectrum becomes more similar to a transmission spectrum [14]. On this basis, it is estimated that the dark-brown parts, which

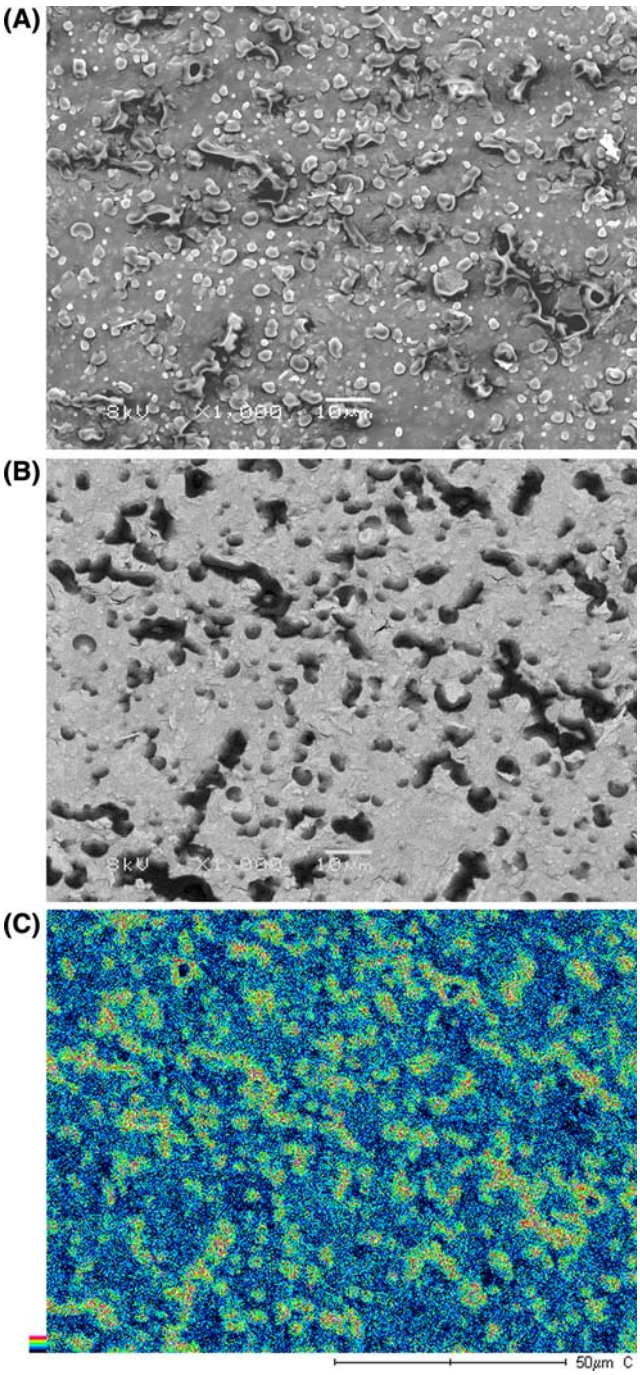

Fig. 3 **A** Secondary-electron image, **B** backscattered electron image (compo) and **C** carbon-mapping image by SEM–EDS of the cross section in the same view of the dark-brown part of a chocolate sample bloomed due to the partial liquefaction of fat

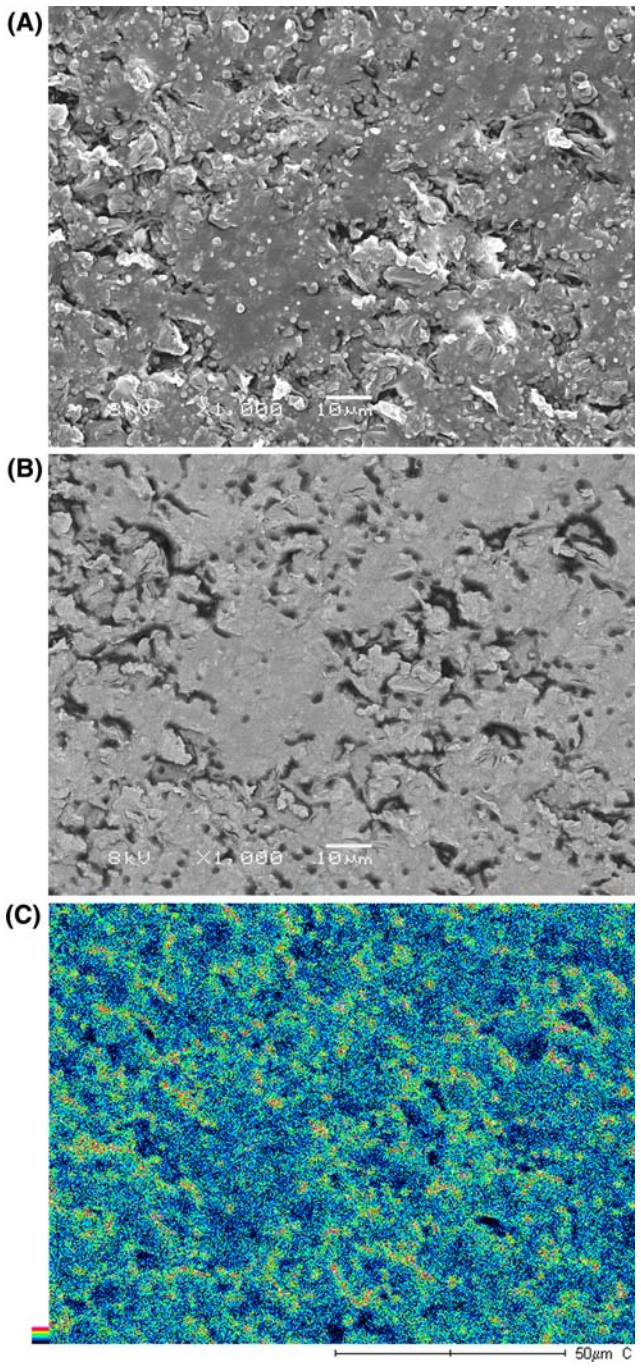

Fig. 4 **A** Secondary electron image, **B** back-scattered electron image (compo) and **C** carbon-mapping image by SEM–EDS of the cross section in the same view of the light-brown part of a chocolate sample bloomed due to the partial liquefaction of fat

yielded reflection spectra, should have smooth surfaces at infrared wavelengths, whereas the light-brown parts, which yielded absorption spectra, should have rough surfaces where infrared diffusion and absorption tend to occur. It is a generally observed phenomenon that particles and surface textures affect behavior towards visible light and colors. The FT-IR results suggest that on the light-brown part of the bloomed chocolate sample, convexities and concavities exists at a level that can diffuse or absorb infrared light, and that those convexities and concavities and/or more-minute convexities and concavities cause the discoloration.

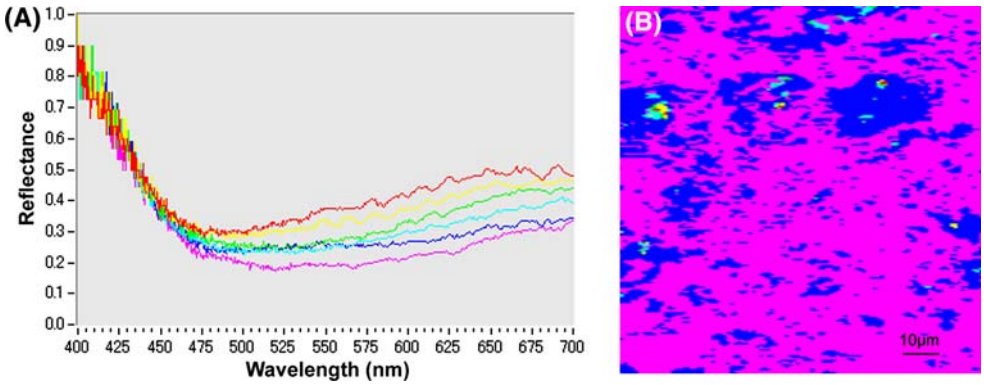

Fig. 5 **A** Spectral reflectance classified by similarity and **B** a spectrally classified image represented by the corresponding color in **A** by PARISS® on a cross section of the dark-brown part of a chocolate sample bloomed due to the partial liquefaction of fat

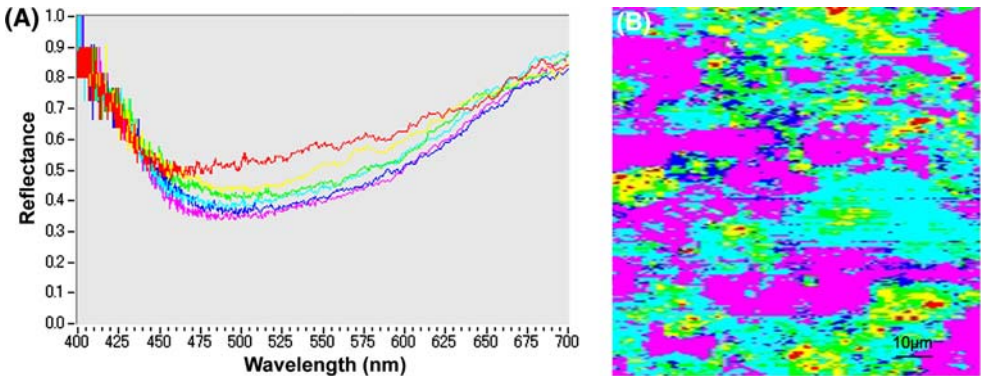

Fig. 6 **A** Spectral reflectance classified by similarity and **B** a spectrally classified image represented by the corresponding color in (**A**) by PARISS® on a cross section of the light-brown part of a chocolate sample bloomed due to the partial liquefaction of fat

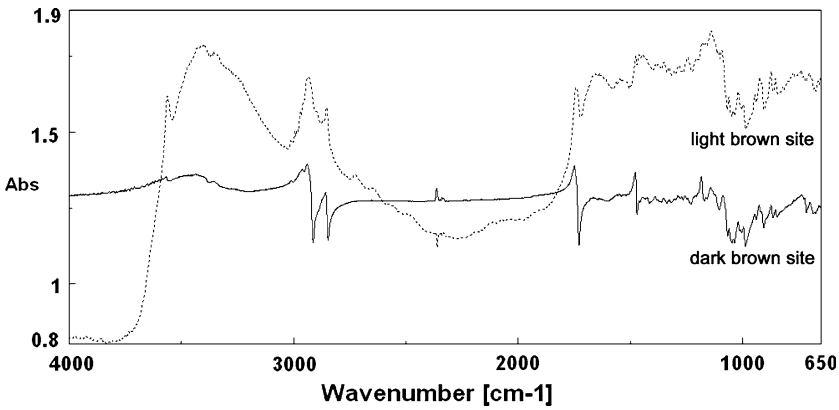

Fig. 7 FT-IR reflectance spectra of the cross section of a dark- and a light-brown site in a chocolate sample bloomed due to the partial liquefaction of fat

On the basis of these discussions, the following mechanism for the formation of bloom as a result of the partial liquefaction of fat, which occurs during temperature variations, is proposed, referring to the claims of Hodge et al. [15]:

(1) When the temperature of cocoa butter is raised to immediately below the melting point, partial melting of cocoa butter crystals occurs and the βV to βVI transition, in which oil-mediated transformation is dominant, occurs gradually [8].
(2) On cooling, recrystallization to βV or βVI occurs. This is the result of the presence of seed crystals. During this cooling process, contraction also takes place, pulling liquid fats into the body of the chocolate.
(3) As this process is repeated, a high-melting-point structure is formed and more constituent separations of liquid fats occur.
(4) When contraction due to solidification occurs, pulling the liquid fats into the body of chocolate, the high-melting point structures are left at the surface, giving a light-brown color at the site. These form the visible bloom.

During the temperature-change cycles, partial liquefaction and recrystallization of fats are repeated. This process leads to TAG composition separation, in

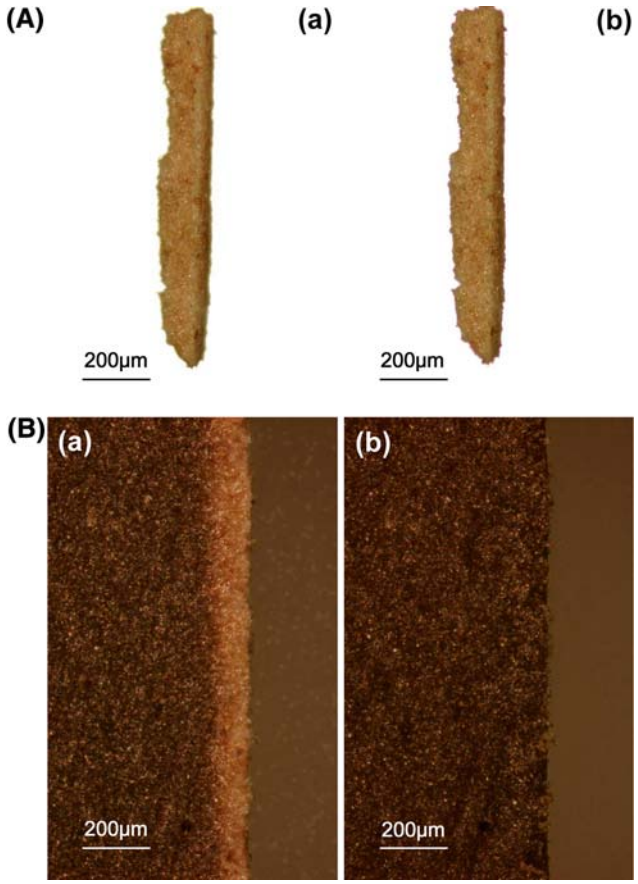

Fig. 8 Optical micrographs of (**A**) the light-brown part separated from a chocolate sample bloomed due to the partial liquefaction of fat and (**B**) the cross section of the bloomed sample, (*a*) at room temperature and (*b*) at 32 °C

other words, separation of high-melting- and low-melting-point components [16], and to βV to βVI crystal transitions of cocoa butter; both of these processes cause coarsening of the fat crystal network. As a result, it is hypothesized that the quantity of separated liquid fats increases and migration to the matrix is enhanced on cooling. There is also a resultant increase in roughness and/or porosity that affects light scattering by the high-melting-point structure left by the migration of liquid fat. Together, these effects cause a change to a light-brown color. Loisel et al. [17] says that a porous matrix is partly filled with liquid cocoa butter fractions existing in chocolate, and the gaps range from 1–4% in a chocolate sample with 31.9% cocoa butter content. Thus, it is believed that, as a result of the coarsened fat crystal network and the migration of the liquid fat component, the gaps are relocated and/or increased, affecting the color tones. Evidence for the TAG composition separations and migration of liquid fat components to the bulk is supported by the fat content and TAG composition analysis results (Table 1). Further evidence that the color change to light brown is produced by the residual structures includes the phenomenon that if a light-brown portion extracted from the bloom produced by the partial liquefaction of fat is held static and heated, no significant color change occurs, but if it is left in contact with the dark-brown part, the color turns dark brown on heating (Fig. 8). The TAG composition of a portion whose color was changed from light brown to dark brown as a result of this heating process is shown in Table 1. The darkened part resulting from heating contains more liquid fat compared with the light-brown part before darkening. Thus, the darkening is considered to be caused by migration of liquid fats from the dark-brown part as a result of heating, suggesting that when the liquid fats leave a site, the color turns light brown.

In general, the color change to light brown, as discussed in this article, does not occur if the temperatures in the rising phase of the temperature cycles are lower. In this case, it is believed that the liquefaction ratios of fats become lower in the rising temperature phases, and TAG composition separation and coarsening of the fat network do not readily occur. In this study, cases of bloom formation of solid chocolate in temperature cycling between 20 and 32 °C environment were investigated. However, if the conditions for coarsening of fat crystals and the withdrawal of the liquid fat component are present, blooms of this final state could be also formed in conditions other than those in this study.

This study revealed that the light-brown parts of chocolate that bloomed as a result of the partial liquefaction of fat are not a result of fat separation to the surface, as conventionally believed, or look lighter color for low fat contents compared with the dark-brown parts, as observed with bloomed chocolate that is not subjected to tempering. Although details are yet be made clear, at least three states of visual fat bloom are presented. Moreover, because this type of bloom is quite commonly seen in the marketplace as a result of remelting of chocolate through the effect of heat during storage, we are pleased to be able to enhance the understanding of the state of the bloom so that possible preventative measures can be adopted. Before this study, different types of bloom were sometimes confused during discussion, but, as a result of this study, it is now possible to draw a clear distinction between the various types of bloom when conducting discussions of bloom on chocolate. We expect to continue our studies with a view to achieving a complete understanding of causes bloom formation and its consequences.

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