Scale-sensitive Fractal Analysis of the Surface Roughness of Bloomed Chocolate

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ABSTRACT: The surface roughness of stored chocolate bars was studied by scanning laser microscopy and area-scale fractal analysis. Topographic data were expressed by the statistical average roughness (Sa), by two parameters from area-scale analyses—the fractal complexity (Asfc) and the scale of the rough-to-smooth transition (SRC)—and by the relative area as a function of scale. The roughness measured with Asfc showed extremely low correlation with the SRC, indicating that these two parameters can be considered to be independent. Asfc appeared to have some correlation (R^2 = 0.82) with the Sa, indicating that for these data Asfc and Sa are somewhat related. As surface roughness (Asfc) increased during storage, gloss decreased in a linear fashion (R^2 = 0.96), which is consistent with the proposal that surface roughness is intimately related to gloss. The scales of observation from about 0.5 to 100 μm^2 were characteristic of the fat bloom interaction with chocolate surface and with the gloss. Fractal analysis provides parameters (Asfc and relative area) that are better than conventional arithmetic mean roughness for describing the surface changes during storage of chocolate. Both the complexity (Asfc) and the relative areas showed strong correlations with gloss (0.96 and 0.94, respectively), which is consistent with a facet-based scattering model.

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KEY WORDS: Chocolate, fat bloom, gloss, microscopy, relative area, roughness, surface topography.

Chocolate bloom. Fat migration to the surface during storage of chocolate products is a major problem that affects the chocolate industry because it compromises visual appearance and texture quality. Fat bloom is a surface defect that is recognized as a whitish film on the surface of chocolate, causing it to appear dull, old, and stale. There are many hypotheses for its formation, such as inadequate tempering procedures used in the cooling process of chocolate (1) and/or the presence of foreign fats (2). The object of tempering is therefore to develop a sufficient number of seed crystals to encourage the total fat phase to crystallize in the more stable polymorphic form V (3). Fat bloom in block chocolate tablets, which occurs after several weeks of storage, is believed to be caused by the recrystallization of cocoa butter from the polymorphic state V into the most stable form VI (4). The specific mechanism by which fat bloom occurs is incompletely known, although several theories have been proposed. Diffusion has been a preferred hypothesis to explain fat migration within the chocolate matrix (5). Recently, Aguilera *et al*. (6) argued that diffusion may not be a dominant mechanism, since the liquid fraction of cocoa butter most likely moves through interparticle pores by capillary forces.

Methods to quantify bloom vary from the subjective visual perception of "fat bloom" to quantitative techniques such as colorimetry (7), computer vision (8), and magnetic resonance imaging (5). Although color seems to be a good proxy for bloom, it does not provide information about changes in the topography or microstructure of the surface and their possible relation to bloom. It is documented that during storage of chocolate, small cocoa butter crystals begin to appear at the surface, initiating primarily at the cracks and crevices of the surface. Over time, these crystals of fat increase in number and size as bloom progresses (9). Preliminary work showed that changes in the surface microstructure of bloomed chocolate correlate well with increased surface roughness (10). The surface microstructure of milk chocolate that had been subjected to temperature cycles (from 20 to 32, 33, or 34°C) was examined directly by atomic force microscopy (11). In 24 h, the average surface roughness increased from 278 nm to 736 nm for the 20–34°C cycle, and it was described as "consisting of jutting crystals and large raised yet smooth areas randomly located within the chocolate matrix." Hence, it can be hypothesized that surface roughness is directly related to the extent of fat crystallization at the surface and that this phenomenon, in turn, is the main cause of bloom in chocolate.

Area-scale fractal analysis. Determining structural changes at the surface of chocolate requires precise methods to quantify surface roughness at the relevant scale (i.e., some microns). Area-scale fractal analysis, developed by Brown *et al*. (12), and standardized (13), combined with laser microscopy, seems an appropriate tool to quantify the surface of chocolate and other foods.

Area-scale analysis by the patchwork method is based on the principle from fractal geometry that the area of a rough surface is not unique but depends on the scale of observation (14). This method determines apparent areas over a range of scales by repeated virtual tiling exercises with triangular patches on the measured surface. Within each repetition the triangular tiles all have the same area in a 3-D space. The area of the triangle represents the scale of observation. For each scale analyzed, a relative area is calculated as the area calculated by the tiling

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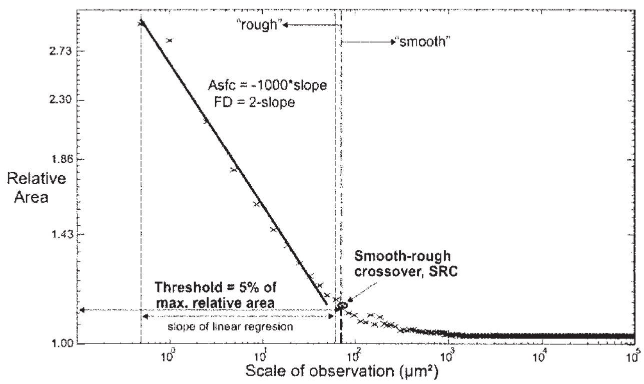

FIG. 1. Area-scale plot, showing the logarithm of the relative area as a function of the logarithm of the scale of observation or triangular patch area for a series of tiling exercises from the surface of a chocolate sample measured on day zero. The threshold in relative area is used to find the smooth-rough crossover (SRC), and the slope is used to calculate the area-scale fractal complexity (Asfc) and fractal dimension (FD).

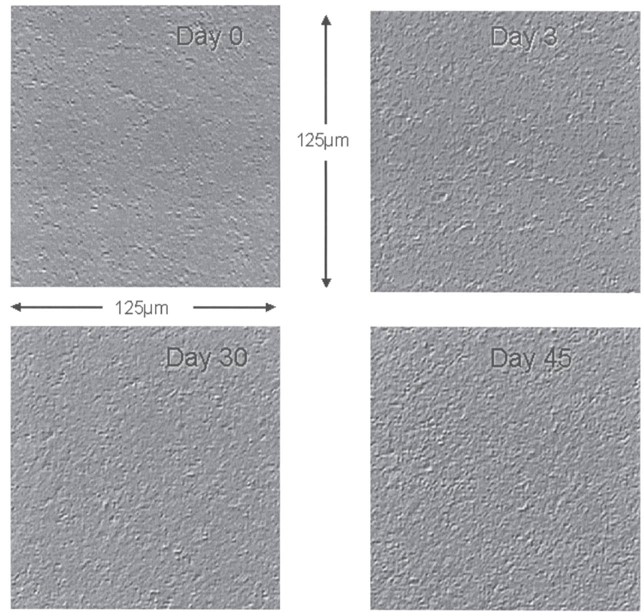

FIG. 2. Height maps constructed from four surfaces measured after 0, 3, 30, and 45 d of storage.

exercise (the number of triangle patches times the area of the patches) divided by the nominal or projected area of the surface that has been covered in the tiling. The minimum relative area is 1. Graphs of the logarithm of the relative area vs. the logarithm of the triangular patch area used in tiling are called area-scale plots. Area-scale relations can be determined using an algorithm such as that found in programs such as Sfrax or Kfrax® (www.surfract.com), developed by Brown *et al.* (12,15). The algorithm calculates relative area as a function of scale using virtual, triangular tiling exercises repeated over a range of scales as represented by the triangle size. The measured area is calculated from the number of triangles times the triangle size. Relative areas are the measured area, divided by the projected, or nominal, area of the tiled region. Area-scale plots (Fig. 1) show the relative area vs. the scale (tile size) on log scales. In addition to the relative areas, two kinds of characterization parameters can be calculated from the area-scale analysis: crossovers and complexities.

The smooth-rough crossover (SRC) is the scale of observation, or triangular patch area, above which the surface is essentially smooth and can be easily described with Euclidian geometry, and below which it is essentially rough and is more easily described with fractal geometry. Figure 1 shows that, at large scales, the relative area tends to 1, and any interactions with the surface at these scales will perceive the surface as smooth (e.g., licking a piece of chocolate with the tongue). At finer scales the relative areas increase so that interactions at these scales will see the surface as being rough. A threshold in relative area must be selected so that the scale of the SRC can be defined.

The slope of the log-log plot is an indication of the geometric complexity. The slope is used to define the area-scale fractal complexity (Asfc), which is −1000 times the slope, and to characterize the complexity of the texture in this range of maximal complexity (see Fig. 1). The larger the negative slope is, the greater the complexity. At scales below the SRC, the logarithm of the relative area of many surfaces increases linearly for two or more orders of magnitude. The fractal dimension of the measured surface, which is also a measure of complexity, is equal to two minus the slope of the area-scale plot.

In conjunction with area-scale analysis, a scale-based functional correlation can be derived when another property, such as gloss, is measured. Scale-based functional correlations relate that property to scale so as to determine the characteristic scale interaction. It has been shown that scale-based functional correlations between surface characteristics and a particular property can be determined by plotting series of linear regressions between the desired property and the relative areas, then displaying the regression coefficients as a function of scale. Ideally, in the clearest cases of scale-based functional correlations, at large scales there would not be a correlation to the property being examined, then as the scale decreases the value of the correlation coefficient, R^2, begins to increase to some maximum before either decreasing or leveling off. This scale of the maximum in R^2 is considered to be the characteristic scale of interaction for a particular property (16).

This paper applies area-scale fractal analysis to characterize the surface of bloomed chocolate so as to develop a better understanding of roughness-sensitive phenomena on chocolate surfaces, to find correlations between behavior of the fat bloom on gloss and roughness, and to identify the important scales of interaction with a rough surface.

MATERIALS AND METHODS

Materials. Rectangular bars (4.2 × 15.7 cm) of commercial chocolate (milk chocolate; Hershey's, Hersey, PA) were purchased from a local market in Worcester, Massachusetts. Three chocolate bars were immediately stored in a chamber and ex-

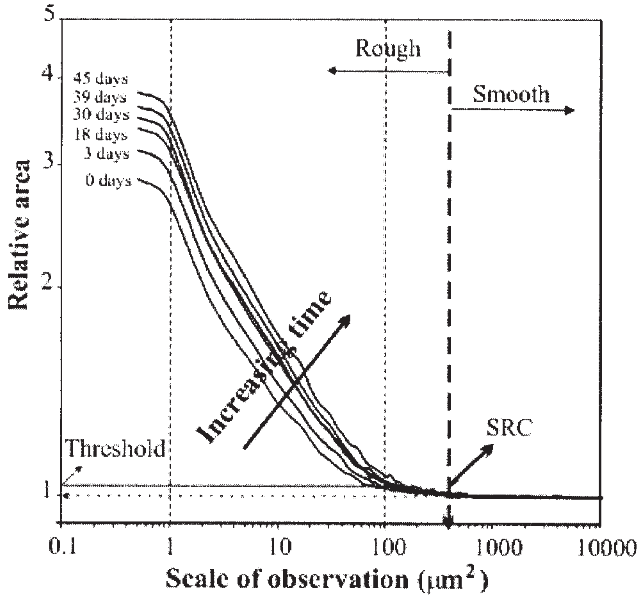

FIG. 3. Relative surface area as a function of scale of observation (μm^2) for chocolate bar during selected storage times. For abbreviation see Figure 1.

posed to 12-h cooling–heating cycles between 16 and 28°C for 45 d, as described in Briones and Aguilera (8). RH never exceeded 50%.

Surface measurement. Topographic data sets were acquired from the surface of chocolate bars by measuring heights, *z*, as a function of position (*x, y*) with a scanning laser microscope (SLM), i.e., scanning laser profiler, every 2 d (in triplicate) during storage. The SLM was a UBM Microfocus (provided by Solarius Development Corp., Sunnyvale, CA) with a dynamic focus sensor and positioning tables. Regions (500 × 500 μm) were scanned in three positions along the chocolate bar surface using the SLM and a sampling interval of 1 μm. Data were stored digitally for subsequent analysis.

Surface data analysis. The topographic data sets were analyzed to determine the roughness parameters such as the arithmetic mean surface roughness (Sa) and the fractal parameters (area-scale fractal complexity, Asfc, and smooth-rough crossover, SRC). Sa is the arithmetic mean value of the (absolute) deviation of the protrusions and depressions of the roughness profile from an averaged center line along the measuring distance. This parameter was determined using MountainsMap® analysis software by Digital Surf (www.digitalsurf.fr/).

Area-scale analysis by the patchwork method was used to determine the Asfc and the SRC. The relative areas were determined over a range of scales of observation from half the square of the sampling interval 0.5 to over 10,000 μm^2. Area-scale relations were obtained directly from Kfrax® software.

The SRC was determined by setting a threshold in relative area at 5% of the greatest relative area calculated, which is consistently found at the finest scale of the calculation, one-half the sampling interval squared, or 0.5 μm^2. The first scale that

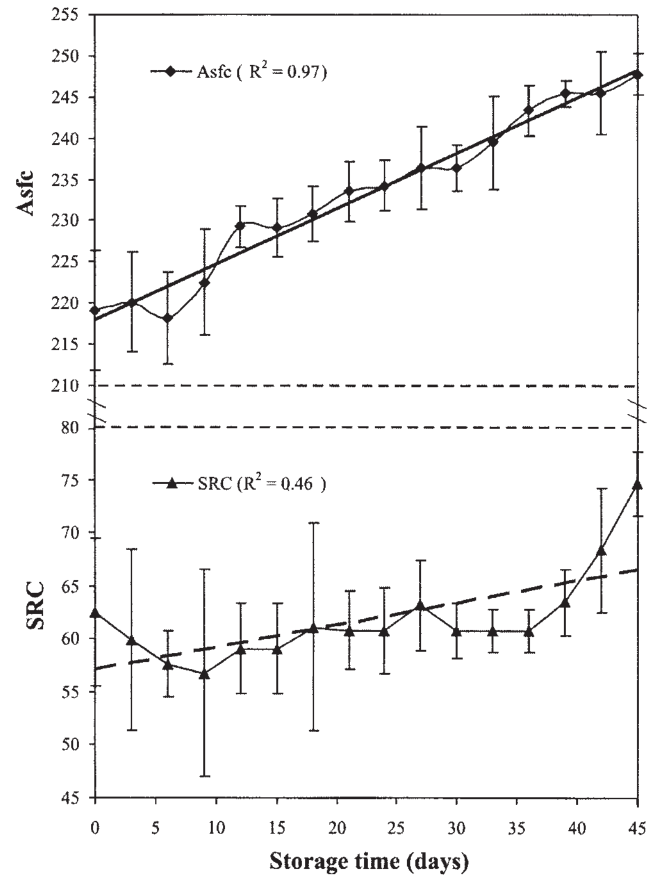

FIG. 4. Fractal parameters Asfc and SRC plotted with their means and SD as a function of storage times with corresponding regression coefficients. For abbreviations see Figure 1.

corresponds to the calculated relative area that exceeds the threshold, going from the coarser scales toward the finest, is the SRC. The Afsc was calculated from the section of the plot corresponding to the two orders of magnitude in scale where the negative slope was the steepest, i.e., that section of the plot, with a width of two orders of magnitude, that has the greatest complexity. This region of greatest complexity was consistently at the finest scales analyzed, from 0.5 to 500 μm^2.

The SRC was used to assist with the experimental design. The SRC was calculated from some initial trial measurements and used to select the size of the measurement region used in the bulk of the experiment. Since there is no information about the complexity at scales greater than the SRC (15), one to two orders of magnitude in areal scale above the SRC should be largely sufficient for capturing any potentially interesting changes in relative area with respect to scale that could influence the conclusions, even allowing for some margin of safety in case the trial surfaces happened to be atypically smooth. As the SRC were found to be between 50 and 75 mm^2, there is no need to acquire topographic data over regions significantly larger than a few hundred square micrometers in order to provide information on area-scale relations.

It is more difficult to determine the SRC precisely than the Asfc. A threshold in relative area is used to determine the SRC

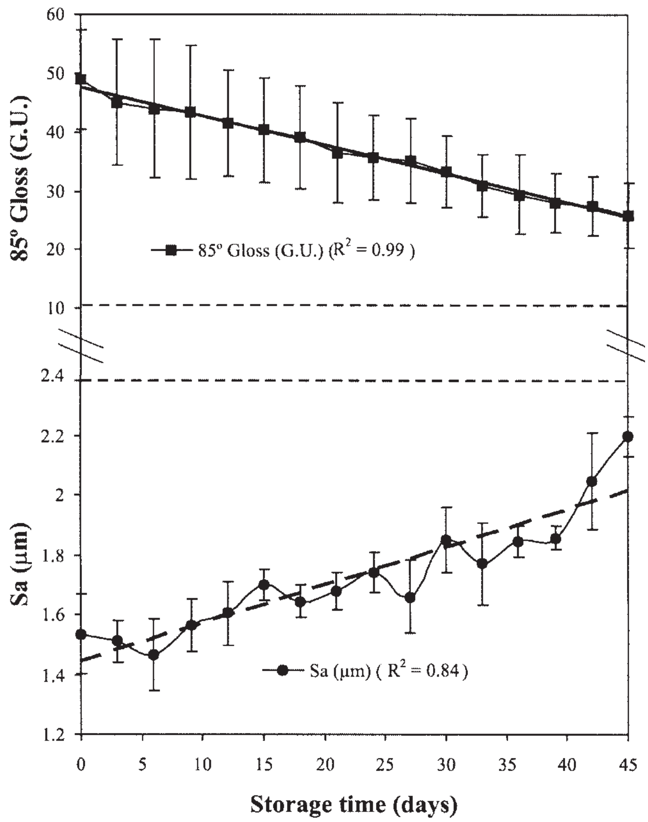

FIG. 5. Average roughness (Sa) and gloss plotted with their means and SD as a function of storage times with corresponding regression coefficients.

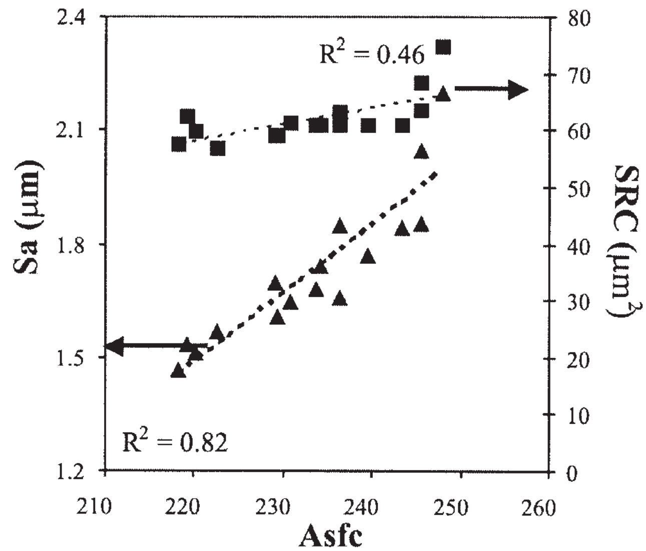

FIG. 6. Asfc vs. Sa (µm; ▲) and SRC (µm^2; ■) (triangles and squares indicate average values per day). For abbreviations see Figures 1 and 5.

in a region where the slope of the plot is low. A small difference in the threshold in relative area makes a relatively large difference in the resulting SRC. Therefore, the SRC will in general be less sensitive to trends than the Asfc.

Measurement of gloss using the Micro-Tri-Gloss meter. Gloss of the chocolate surface was measured using the multiple-angle Micro-Tri-Gloss meter (BYK Gardner, Silver Spring, MD). Reflectance was measured at an incidence light angle of 85° from the normal to the chocolate surface, in accordance with ASTM method D523. A polished black glass plate with a refractive index of 1.567 was used as standard surface (17) and given arbitrarily a gloss value of 100. Gloss was reported as gloss units (G.U.; percentage of standard) based on determinations (in triplicate) at six positions along a chocolate bar.

Determination of the fundamental scale. For chocolate bars, the relative areas at one scale of observation were plotted vs. gloss during storage time. The least squares regression coefficients, R^2, were determined. The process was repeated at other scales, so R^2 values were determined over a significant range of scales. Then the regression coefficients were plotted vs. scale of observation to see whether there was a tendency toward a maximum. A maximum of R^2 with respect to scale was expected, and the scale with the highest regression coefficient, i.e., where R^2 was a maximum, should be the characteristic scale for this fat bloom interaction with chocolate surface (16).

RESULTS AND DISCUSSION

Examples of measurements made from different surfaces after 0, 3, 30, and 45 d of storage are rendered as shaded heights in Figure 2. The change in the topography as a function of storage time is evident but subtle. Evidently, the number of fine scale features increases with time. Increase in size of the features is not as evident.

Examples of area-scale plots are shown in Figure 3. At large scales (>300 µm^2) the relative area tends toward 1 and hence the surface appears microscopically smooth. At finer scales the relative area deviates significantly from 1. Above the SRC, the surface is smooth and could be well characterized with Euclidean geometry as essentially planar. At scales below the SRC the surface is characterized using fractal geometry. Over the scale range from 1 to about 100 µm^2, the relative area increases monotonously with a decreasing logarithm of the scale of observation. In the same region, the linearity of the plot suggests that the geometry of the surface was statistically self-similar over the scales of this linear region for all chocolate surfaces during storage time. Hence, a single parameter indicative of the complexity, such as Asfc, can be used to characterize the surface topography. Over this region the area-scale generated from the measured surfaces shows that the slope and therefore the complexity increase with time (Fig. 3).

The values of the Afsc and SRC as a function of storage time are shown in Figure 4. The Asfc increases with storage time and is strongly correlated with storage time as shown by the R^2 of 0.97. The SRC has a comparatively low correlation coefficient with storage time. The gloss and the average roughness (Sa) are plotted vs. the storage time in Figure 5. Both of these parameters tend to increase as a function of storage time. The decrease in gloss is strongly correlated with the increase in storage time, with an R^2 of 0.99. The average roughness (Sa) is correlated with the storage time with an R^2 of 0.84.

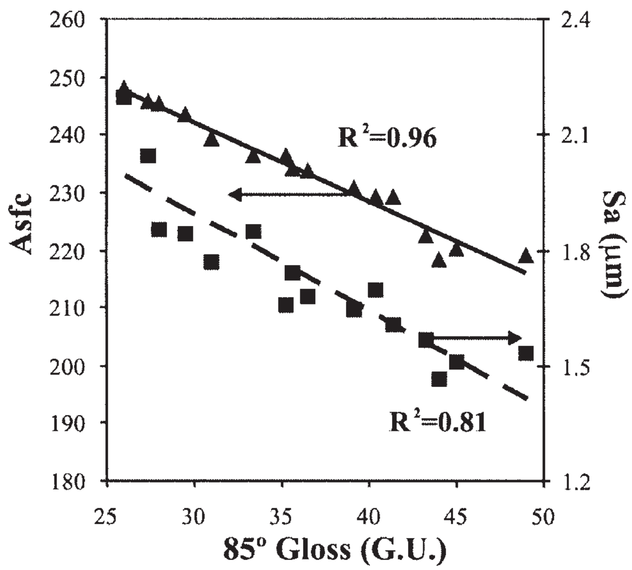

FIG. 7. Asfc (▲)and Sa (■) vs. 85° gloss (G.U.) showing the means and SD for each day and the regression coefficients. For abbreviations see Figures 1 and 5.

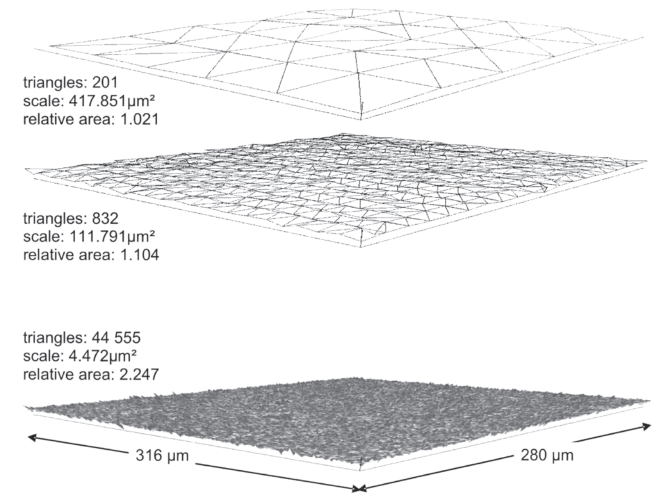

FIG. 8. A sequence of three tiling exercises on a section of a surface measured after 45 d of storage. The triangular tile sizes, 417.851, 111.791, and 4.472 μm^2 represent the scales. The relative areas are 1.021, 1.104, and 2.247, respectively. A height image of the measured surface is shown on the bottom.

The SRC as shown in Figure 4 appears to be poorly correlated with storage time, and hence with gloss. The SRC is thought to be more sensitive to the size of features on the surface, whereas the Asfc is more sensitive to the number. The fact that the SRC does not correlate with storage time, but that Asfc does, is an indication that the growth of fat bloom features with time may be less important than the increase in the number of bloom locations. This suggests that decrease in gloss during the observed storage time is due more to nucleation of new bloom features than to the growth of existing features.

A potentially intriguing trend that starts at about day 35 and continues through the end of the study at 45 d suggests that the SRC might steadily increase at sufficiently large storage times. If this were found to continue, it could be indicative of more pronounced growth of blooming features on the surface than was observed prior to day 35.

The roughness measured with Asfc shows extremely low correlation with the SRC (Fig. 6). This indicates that these two parameters can be considered to be independent, since they are orthogonal parameters and contain distinctly different information about the surface topography.

Since the Sa is commonly used to describe the average geometric impact of protrusions and depressions of the surface, it was compared with Asfc. The complexity and therefore fractal dimension, as indicated by the Asfc, appear to have some correlation ($R^2 = 0.82$) with the Sa, as shown in Figure 6. This correlation is highly data dependent, rather than systematic and universal, and is not generally found in other systems. The direct correlation between arithmetic mean roughness and complexity shown here indicates that the surface features that are causing the complexity occur in the scale range over which the Asfc was calculated, 0.5 to 50 μm^2. Although the Sa is commonly used to describe the roughness of a finished surface, it lacks specificity, as there are many distinctly different surface geometries that can have the same average roughness. The value of Sa does not depend on the order of the points, thus, this average will give no information about the shape of irregularities or the surface of the particle or solid (18). Furthermore, it is difficult to determine the specific range of scales that has influenced the Sa values.

Blooming of chocolate gives the surface a greyish and dull appearance, which is caused by the dispersion of light by small crystals of fat (19). The relations between surface roughness, as characterized by Asfc and by Sa, and gloss are shown in Figure 7. Figure 7 indicates that as irregularities in the chocolate surface, as indicated by the Asfc roughness, increased, gloss decreased in a linear fashion ($R^2 = 0.96$). The Sa also seems to be indicative of the gloss in this case, although the correlation is not as strong ($R^2 = 0.81$) as with the Asfc. These trends of an inverse correlation between specularly reflected light and surface roughness are well known by materials scientists; however, the correlation coefficient with Asfc in this case is remarkably high. The reason for the decreasing glossiness with storage probably comes from the larger light scattering of increasingly irregular surfaces. For example, it has been reported that polished surfaces of denture base resins have larger gloss values than those of unpolished samples (20). Since increased roughness may be due to a new phase of chocolate fat accumulating on the surface (21), the idea that this change in composition has an effect on measured gloss may not be discarded.

Unlike the Asfc, the Sa does not have a specific scale range associated with it. In addition, the tiling algorithm on which the Asfc is based can be interpreted physically in the context of gloss as light scattering off facets approximated over a range of scales by the triangular tiles (Fig. 8) on a surface. These tiles provide a logical relation between the Asfc through the relative

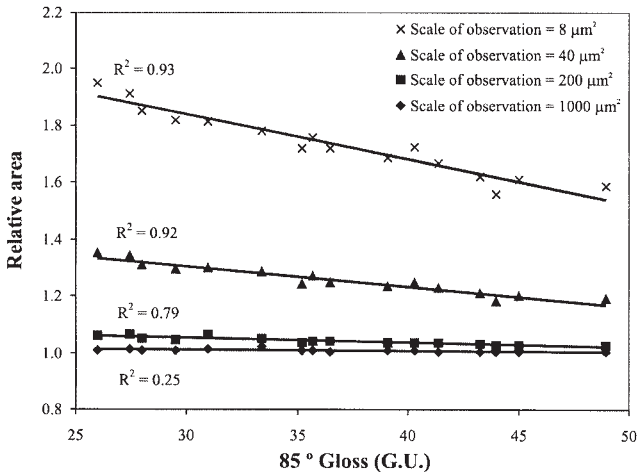

FIG. 9. Gloss vs. relative areas at scales of 1000, 200, 40, and 8 μm^2, with regression coefficients.

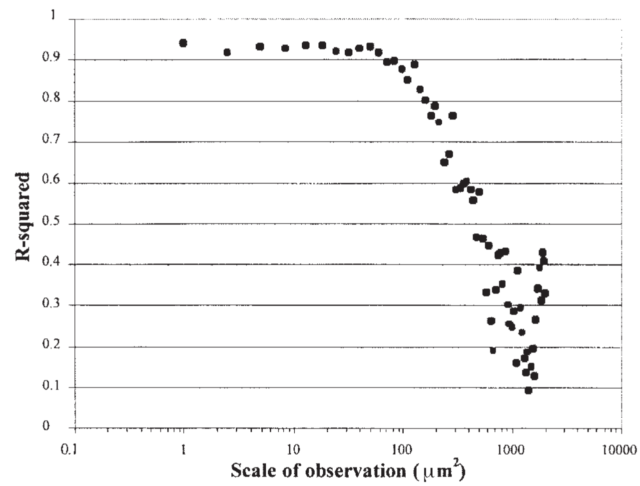

FIG. 10. Based on plots similar to those shown in Figure 8, regression coefficients, R^2, for relative area as a function of gloss during storage time, are plotted vs. the scale of observation.

areas to light scattering and hence gloss (22,23). This kind of clear physical interpretation for understanding the link between the surface parameter and gloss is lacking in the case of Sa, which, unlike Asfc and relative areas, contains little information on feature shape, size, or orientation that can be interpreted as leading to scattering. The range of scales over which the relative areas best correlate with gloss can be determined and are indicative of the scales of interaction with the bloom features as shown for an adhesive system by Brown and Siegmann (16).

The triangular tiling algorithm used for determining relative areas can be interpreted in the context of gloss by considering that the individual triangles act as reflectors, scattering the light by some amount related to their inclination (22,23). As the inclination of the triangles increases, which tends to happen at finer scales, the amount of scattering increases and hence the gloss would be expected to decrease. A typical tiling sequence over progressively finer scales is shown in Figure 8. The relation between the relative area and inclination of the tiling triangles can be shown to be equal to a weighted average of 1 divided by the cosine of the angle of inclination of the normal to the triangular tile with the normal to the nominal surface,

$$\text{Rel Area} = \sum_i \frac{1}{\cos\theta_i}\frac{p_i}{L} \qquad [1]$$

In Equation 1, θ_i *is* the inclination angle of the *i*th tiling triangle, p_i is the area of the *i*th tiling triangle projected onto the nominal horizontal plane, and L is the total projected area of all the tiling triangles on the nominal horizontal plane.

From the foregoing discussion, it could be expected that the relative areas themselves would correlate well with the gloss over some scale-specific range. The scale range over which they correlate could be considered to be a kind of characteristic or fundamental scale relating the gloss to the features on the surface responsible for interacting with the light. For discovering the fundamental scale or scales of interactions with the surface that could be used for determining gloss, the relative areas were plotted vs. 85° gloss values over scales of observation between 1 and 2040 μm^2. Examples of the plots at four of the scales are shown in Figure 9. The least square regression coefficients (R^2) were determined at each scale considering relative area at a specific scale to be a topographic characterization parameter. The relative area describes the geometric opportunity for interactions with the surface (16). For example, Brown and Siegmann (16) show that the relative areas below a characteristic scale are better predictors of adhesive strength than a conventional parameter of roughness (Sa). Furthermore, the area-scale fractal approach was able to show that adhesion related to an available surface area at or below a particular scale. Then, in the current work, the regression coefficients for gloss vs. relative areas were plotted vs. scales of observation between 1 and 2040 μm^2 to see if there would be a tendency for a maximum (see Fig. 10). The scale where R^2 is a maximum should be a characteristic scale for this phenomenon (fat bloom). Figure 10 shows a marked increase in the regression coefficient, R^2, as the scale is reduced from about 2040 down to 100 μm^2. In the fine-scale range of the scale of observation, the regression coefficient remained high and constant down to 0.5 μm^2.

It is not clear in Figure 10 whether R^2 remains high to a lower scale of 0.5 μm^2 because of the physics of the scattering or whether this is indicative of some limit on the resolution of the measured surface. The instrument and the sampling interval of 1 mm allegedly should support an areal resolution down to 0.5 mm^2; however, it is not clear what the spatial resolution of the sensor is and how the other factors such as noise could diminish the resolution of the measurement. There is currently no standard means of experimentally determining the resolution of surface texture measuring devices. The limitation in this observation due to the uncertainty in the resolution of the instrument is in knowing how far below 100 μm^2 the correlations extend. However, possible limits on the resolution of the measurements do not affect the observation that the correlations between gloss and relative area and with Asfc are significantly above 0.9 through some range of scales below 100 μm^2.

Clearly, surface roughness is intimately related to gloss, but some uncertainty remains with regard to the scale ranges. It might be reasonable to suppose that, if the measurement could be made with a sufficiently fine resolution and if the area-scale relations were calculated to sufficiently fine scales, eventually the R^2 would begin to diminish at some sufficiently fine scale. At this very fine scale, the tiling would eventually be sensitive to features that are finer than those responsible for the scattering. The facet-scattering model would then be overestimating the scattering, and underestimating the gloss, and the R^2 values would therefore diminish. Nonetheless, the results of the current work apparently indicate that the scales of observation from 0.5 to 100 μm^2 are characteristic of the fat bloom interaction with chocolate surface.

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