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Microstructural and rheological influence of different strategies to mitigate oil migration in chocolate pralines during storage in limiting conditions

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ABSTRACT

Chocolate pralines are metastable systems in which oil migration, particularly at high storage temperatures, represents the main contributor to quality loss. In this study, two strategies were compared to retard this migration respect to a control sample (C); the use of a barrier consisting of cocoa butter high-melting (CB) and the use of an oleogel realized with beeswax and sunflower oil (CA). Samples were stored in thermal abuse conditions (28 °C) and subjected to: microstructural, rheological and ability to retain oil in the filling analysis and colorimetric evaluation. Microstructure highlighted the highest percentages of oil in CA samples filling during storage, respect to C and CB, as confirmed by oil release. According to fundamental rheological analysis, C and CB showed the highest filling yield stress values respect to CA, which showed a linear and homogenous trend. Dynamic rheological analysis underlined the presence of an increase in the elastic modulus during storage, suggesting matrix aggregation and gummy filling cream, which was lowest for CA, supporting the positive effect exhibited by this barrier. For empirical-imitative analysis, CA showed a more spreadable and creamy filling. Results revealed the ability of beeswax oleogels to improve quality attributes of pralines during storage, also at higher temperatures.

1. Introduction

From a chemico-physical point of view, chocolate can be defined as a concentrated suspension of sugar, cocoa, and eventually milk powder embedded in a fat matrix, mainly cocoa butter, which is partially solid at room temperature (Delbaere et al., 2016). Chocolate industry represents a significant manufacturing sector in Europe. Over 200 000 people in Europe are involved in the chocolate production in over 2000 companies of which around 90% are SMEs (Dahlenborg et al., 2015). In the last few decades, an increasing demand within the chocolate industry for filled products, such as pralines, filled chocolate bars, and chocolate-coated bakery products, was registered.

Chocolate pralines are complex food products that contain a soft filling surrounded by a chocolate shell (Slettengren, 2010). These kinds of products are in general metastable systems in which shelf life is often limited by component migration and phase transition, and one of the main reasons for their storage instability can be attributed to the migration of filling components, in particular oil, into the surrounding chocolate shell (Choi et al., 2005; Franke et al., 2022). This behaviour can be reasonably attributed to the triacylglycerol (TAG) concentration differences at the interface between chocolate shell and filling that induce a diffusive transport representing the driving force of this phenomenon (Ziegleder et al., 1996a; Ziegleder et al., 1996b; Ziegleder & Schwingshandl, 1998; Böhme et al., 2021).

The degree and intensity of oil migration in pralines is affected by both product characteristics and storage conditions, in specific by high temperatures (Choi et al., 2005; Dahlenborg et al., 2015). This phenomenon, which tends to increase during storage, results in a bloom formation, a softening of the shell and a rheological transformation of the filling, which tends to be more gummy, losing its fluidity and spreadability, becoming less pleasant to the consumer (Talbot, 1990).

In recent years, oleogels have been developed as an innovative method for oil structuring. Their purpose is to entrap bulk oil in a thermally reversible three-dimensional supramolecular network that is created by edible oleogelators (Cerqueira et al., 2017). In order to minimize oil migration during the manufacturing process, oleogels have been utilized in the production of cakes, meat products, cookies, confections, ice cream, and chocolate pastes or spreads. Oleogelators can be divided into three main categories: crystalline particles, such as monoglyceric stearate, fatty acids, fatty alcohols, and plant waxes;

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self-assembly fibers, like β-sitosterol plus oryzanol and lecithin plus sitosterol; and polymeric strands, like ethyl cellulose (Han et al., 2014; Pehlivanoglu et al., 2018). Among plant based waxes we can find, beeswax, obtained from the honeycombs of bees (*Apis mellifera* L, Fam. Apidae) after the honey is removed.

Beeswax is mainly made up of a mixture of fatty acid esters and fatty alcohols, paraffinic hydrocarbons and free fatty acids, minor amounts of free fatty alcohols are also present; it has a melting point between 60 °C and 75 °C. Food applications of beeswax include its use as a component of dietary food supplements (soft gelatin capsules and tablets), glazes and coatings, texturizer in chewing gum, flavored water-based beverages, and as a carrier of food additives (including flavors and dyes) (Kuznesof, 2005). Its current applications and low cost make it a promising candidate for use in fat-based food in the form of oleogel. According to Doan et al. (2018), the physicochemical properties as well as the storage stability of wax-structured oleogels are affected by the oil content and wax as well as by the processing parameters, such as the cooling rate and the stirring temperature.

The aim of this study was to compare different strategies to reduce oil migration in chocolate pralines, (from filling to shell), in order to improve their shelf life, avoiding the softening of the shell, delaying the bloom formation and overall maintaining optimally rheological properties of filling in terms of elasticity, mouthfeel and spreadability, also in thermal abuse conditions. Oil migration from filling to shell, is in fact the major responsible for pralines quality loss during storage especially at high temperatures. In specific, two different barriers were tested, each one individually, in comparison with a barrier-free control sample. The two adopted barriers were respectively: a high melting cocoa butter coating and an oleogel barrier made up with beeswax and sunflower oil, interposed between the shell and the filling. The effectiveness of the strategies was evaluated in accelerated storage by applying: microstructural, rheological and physico-chemical analysis.

2. Materials and methods

The research was conducted on model pralines produced on a lab scale, starting with milk chocolate used as shell and spreadable cream as filling; both raw materials were provided by an Italian local company. Milk chocolate was made up of: sugar (39.5%), whole milk (26 %), cocoa butter (20%), cocoa mass (13%), milk fat (1%), soy lecithin (0.30%) and vanilla extract (0.20%), while filling cream was realized by sugar (34.50%), nut paste (23%), sunflower oil (13%), cocoa powder (12%), milk powder (12%), cocoa butter (5%), soy lecithin (0.30%) and vanilla extract (0.20%).

2.1. Pralines control production

Milk chocolate was previously tempered by following a two-steps process made up of heating in a water bath reaching a temperature of around 50–52 °C and a subsequent cooling to 30 °C during constant stirring. Samples were then poured into the molds, which were previously heated to 30 °C to prevent temperature changes in the chocolate, paying attention to eliminate air bubbles and to obtain a thin and homogeneous shell of around 3.00 ± 0.03 mm, before being placed in a cell at a temperature of approximately 4 °C for 10 min. Filling cream was heated to 52 °C in a water bath and then cooled to 30 °C. Through a pastry bag, shells were filled with cream and placed in a refrigerated cell at 10 °C for 15–20 min. Another layer of thin temperated milk chocolate around 0.3 mm was used to close pralines by slightly heating the shell surface, in order to facilitate the adhesion of the lower part. Pralines were cooled in the cell at 10 °C for at least an hour and then taken out of the mold.

2.1.1. Pralines with cocoa butter barrier (CB) production

The shells were realized according to the previously described step (2.1), but before injecting the cream, the shells were covered with a layer of high-melting cocoa butter (Eulip, S.p.A), after which the mold was turned upside down to remove the excess. Subsequently filling was inserted and before pouring the bottom shell, an additional layer of fat was applied to the latter, to isolate the cream from the shell. The thickness of the barrier was approximately 1.00 ± 0.02 mm. In particular, the cocoa butter was heated to 60 °C, after which it was cooled (kept in agitation) to a temperature such that it did not cause damage to chocolate, therefore up to about 30–33 °C.

2.1.2. Pralines with beeswax oleogel (CA) production

The preliminary preparation of the oleogel consisted of a complete melting of 50 g of a commercial food-grade beeswax and 500 g of sunflower oil with a magnetic stirrer at 75 °C in order to achieve a homogenous mixture, based on preliminary trials. Subsequently, this oleogel 10% w/w, was applied into praline shells following the same procedure as the CB: it was cooled to a temperature compatible with the chocolate shell (about 30 °C), poured into the mold; the excess was removed by reversing the mold and an additional layer was applied between the cream and the bottom. The barrier had a thickness of about 1.00 ± 0.02 mm.

2.2. Samples storage

Samples were subjected to an accelerated shelf-life test, in order to simulate the main degradation phenomena and evaluate their stability in thermal abuse temperature conditions. In countries with high environmental temperatures, such as southern Europe and the southern United States, fat migration in confectionery is a significant issue; consequently, systems are frequently stored at 28 °C to simulate these conditions (Choi et al., 2005; Delbaere et al., 2016; Dicolla et al., 2019). Pralines were stored in a thermal chamber (Memmert, Buchenbach, Germany) at 28 °C and 50% relative humidity. The analyses were carried out at time 0 (T0), that is on freshly produced chocolates and then at 20-day intervals (T1, T2, and T3) on 30 samples of each type of product (C, CB and CA).

2.3. Analytical determinations

2.3.1. Microstructural analysis

The microstructure of filled cream was analyzed by using an optical microscope (BH-2 RFCA, Olympus, Hamburg, Germany) at 10x of magnification. The evaluation of the microstructural characteristics of the captured images was carried out using the image processing program Image Pro-plus 6.0 (Media Cybernetics Inc Bethesda, USA). In particular, the following parameters were determined: size of the oil droplets (diameter of Feret) according to Glicerina et al. (2013), by evaluating the distance between two tangent lines to the two opposite sides of droplets and the percentage area occupied by them.

2.3.2. Determination of retained oil in the filling

According to the method described by Han et al. (2022), 1 g of each filling cream was weighed with an Ohaus analytical balance (Nänikon, Switzerland; accuracy ±0.01 mg) into a 1.5 mL plastic centrifuge and centrifuged at a rate of 14.500 rpm for 7 min using a laboratory centrifuge MPW-55 (MPW Med. Instruments, Warsaw, Poland). After centrifugation, the centrifuge tube was placed upside down on the desk for 24 h to completely remove the released liquid oil.

Then, the following equation was used to calculate the amount of oil retained in the filling (RO):

$$RO\ (\%) = 100 - \left(\frac{(m_1 - m) - (m_2 - m)}{(m_1 - m)}\right) * 100 \quad (1)$$

where m_1 is the mass of the initial sample and centrifuge tube, m_2 is the mass of the sample and centrifuge tube after removing the excess oil, and m is the mass of the centrifuge tube.

2.3.3. Fundamental rheological analysis

Rheological tests were carried out at 25 °C, on the filling of pralines during storage, by using a strain-stress rheometer (Anton Paar, MCR 52, Ostfildern, Germany), equipped with a plate-plate geometry PP25.

2.3.3.1. Measurement in steady-state mode. After 1 min of waiting to attemperate samples, flow tests were carried out increasing the shear rate from 0.1 to 100 s^{-1}. In order to compare samples at different times of storage, yield stress values, expressed as the minimal amount of stress required to obtain material flow were collected. Yield stress parameters were calculated on the basis of the method recommended by the ICA (International Confectionery Association, 2000) and Glicerina et al. (2013), evaluating the stress corresponding to the value of 5 s^{-1} of shear rate.

2.3.3.2. Measurements in oscillatory mode. In dynamic conditions, oscillatory tests were performed to evaluate the viscoelastic properties of filling samples. Preliminary amplitude sweep tests were carried out to determine the linear viscoelastic region (LVR) where the properties of the materials are independent of the applied stress conditions, varying the amplitude of strain from 0.01 to 100 %, at a constant frequency of 20 rad/s. Consequently, in order to evaluate the samples elastic (G′) and viscous (G'') moduli, frequency sweep tests were performed at the strain value of 0.046 %, previously detected in the amplitude sweep test, in a frequency range from 6.28 to 628 rad/s.

2.3.4. Empirical-imitative rheological analysis

Empirical-Imitative analysis were performed by using a "TA-XT2i Texture Analyser" HDi500 (Stable Micro System, Vienna Court, England), equipped with a load cell of 5 kg, on the whole praline. The structural characteristics of the chocolate samples were evaluated by means of cutting tests in terms of breaking strength, to evaluate their mechanical response to the applied stress and hence their resistance to breakage at a constant speed of 1 mm/s. The obtained parameters were: the maximum force F (N), relative to the resistance generated by the product during the first phase of the blade descent, related to the resistance of shell for sample C and shell plus barrier for samples CB and CA and the linear distance which is a dimensionless index expressed by the following formula:

$$\mathrm{Ld} = \sum_{x=1}^{x=n} \sqrt{\left[F(X+1) - F(X)\right]^2 + \left[D(X+1) - DS(X)\right]^2} \quad (2)$$

This function is used to calculate automatically the length of the graph profile (Ld) by combining the lengths of the linear segments that connect the collected points between the prescribed intervals or distances. A more jagged profile, will be characterized by a higher linear distance. Although the estimated parameter is a length, it is dimensionless since the unit of measurement for an axis is force. This parameter has been calculated along the entire descent of the blade; therefore, it represents the distance traveled by the blade until the achievement of the break sample and therefore indirectly the creep resistance of the same through the whole product (shell + filling).

Generally, an increase in Ld corresponds to a more compact structure, which opposes greater resistance to blade sliding, covers a greater distance (Dar & Light, 2014).

2.3.5. Spectrophotocolorimetric analysis

The main colorimetric parameters of shell pralines were measured using a spectrophotocolorimeter CM-5 (Konica Minolta, Sensing Inc., Japan). The CIElab scale was used to determine the lightness (L*), red index (*a), and yellow index (b*). SCE mode (Specular Component Excluded), which excludes mirror-reflected light, was used for the analysis and is used to evaluate the color of an object related to visual perception. In SCE mode, reflective surfaces tend to measure darker than a matte surface of the same color, which is how our eyes see it. The SCE approach is effective in visually inspecting the production process to ensure that the color adheres to the color specifications (Kun et al., 2014). Then the numerical values of a* and b* were converted into the parameters of Hue angle (h°) and Chroma (C*) which represent the hue and the saturation index respectively, obtained by the following formulas (McGuire, 1992):

$$\text{Hue angle (h°)} = [\text{arctang (b*/a*)}/2\,\pi]^* 360 \quad (3)$$

$$\text{Chroma (C*)} = \sqrt{(a*)^2 + (b*)^2} \quad (4)$$

2.4. Statistical analyses

Analyses of variance (ANOVA) and the test of mean comparison according to Fisher Least Significant Difference (LSD) were conducted on all obtained data. Level of significance was $p < 0.05$. The statistical software used was STATISTICA, version 8.0. (StatSoft, Tulsa, Oklahom).

3. Results and discussion

3.1. Microstructure analysis

Fig. 1 shows the micrographs related to the microstructural characteristics of samples filling (C, CB and CA) analyzed at different storage times (T0, T1, T2 and T3), while in Table 1, the values related to the diameter of Feret, and their progressive occupied area during storage, are illustrated. From micrographs and the data shown in Table 1 it is possible to highlight that during storage a gradual reduction in oil droplet size was observed passing from 0 to 60 days of storage, which was less pronunciated in samples CA compared to others. This could be explained by the dispersion destabilization, that arises from the deformation of droplets, as a result of dimpling phenomena caused by differences in gradient concentration due to the compositional variation in terms of triglycerides between filling and shell (Chen et al., 2018). This phenomenon involves the elongation of droplets and their breakdown into smallest sizes (Duchemin et al., 2020; Liu et al., 2021). In C and CB samples there was also a reduction in the percentage of areas occupied by the oily lipid fraction over time, which decreased respectively from 27.1 to 20.8 for control samples, with a percentage reduction of 23.2%, and from 26.5 to 21.9 for CB with a percentage reduction over time of 17.3%. This reduction in oil percentage can be reasonably attributed to oil migration from filling to shell during time, which in both cases seems to be more intense during the first 20 days, reaching similar values at times T2 and T3 for both samples, probably because of the achievement of an equilibrium due to the concentration gradient of oil between filling and shell. In CA samples, unlike the behavior observed in C and CB ones, the amount of oil fraction was progressively increased during storage from T0 to T3 with a percentage increase of 11.2%. This development could reasonably be attributed to the barrier effect of the beeswax oleogel which prevented migration of the oily fraction from the filling to the shell, both creating a mechanical hurdle to this passage, thanks to its good heat stability, in relation to its melting temperature profile (around 60–75 °C) (Buchwald et al., 2008; Wang et al., 2024), but also probably by attracting filling oil by van der Waals interactions, hydrogen bonding and electrostatic interaction (Wijarnprecha et al., 2019; Sivakanthan et al., 2022). Moreover, the increase in the oil percentage highlighted in CA filling can be probably related to the high storage temperature used, which probably promoted a further fluidization of the solid fat matrix present in the filling formulation.

3.2. Determination of retained oil in the filling

The determination of oil retained by the cream in the filling, confirmed results previously obtained by microstructure analysis. As shown in Fig. 2, the amount of oil present in the filling tends to decrease for samples C and CB during storage, highlighting a reduction of 20% for

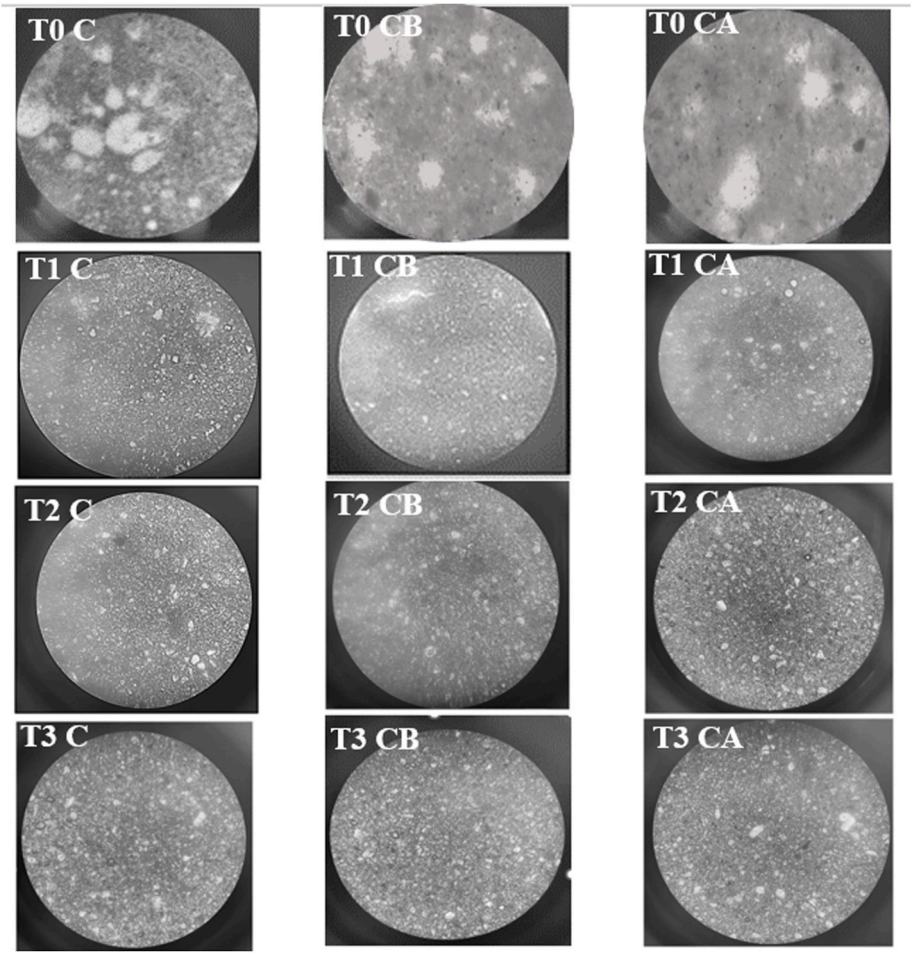

Fig. 1. Micrographs of the filling cream samples (C, CB, CA), obtained by using an optical microscope at the resolution of 10 x at the different storage times (T0, T1, T2 and T3).

Table 1
Percentage (%) of area occupied by the lipid fraction and average diameter of Feret evaluated on the oil droplets present in the filling of pralines at the different storage times (T0, T1, T2 and T3).

AREA OCCUPIED BY LIPID FRACTION (%)				
	T0	T1	T2	T3
C	27.1 ± 1.3^{aA}	20.5 ± 1.6^{bB}	21.9 ± 1.4^{bB}	20.8 ± 1.7^{bB}
CB	26.5 ± 0.9^{aA}	22.8 ± 1.9^{bB}	23.4 ± 2.1^{bB}	21.9 ± 1.8^{bB}
CA	26.1 ± 0.7^{aB}	29.2 ± 1.3^{aA}	28.8 ± 1.1^{aA}	29.4 ± 0.9^{aA}
AVERAGE FERET DIAMETER (μm)				
	T0	**T1**	**T2**	**T3**
C	769.9 ± 19.3^{aA}	587.1 ± 18.9^{bB}	414.17 ± 17.3^{bB}	443.1 ± 28.7^{bA}
CB	744.1 ± 17.3^{aA}	569.3 ± 32.3^{bB}	431.6 ± 29.2^{bB}	429.5 ± 38.6^{bA}
CA	760.1 ± 18.7^{aA}	638.1 ± 19.2^{aA}	514.1 ± 28.8^{aA}	478.3 ± 15.6^{aA}

$^{a-b}$ letters significantly differ at $p < 0.05$ for different samples at the same time of storage, within the same microstructural parameter.
$^{A-B}$ letters significantly differ at $p < 0.05$ for the same sample at different time of storage within the same microstructural parameter.

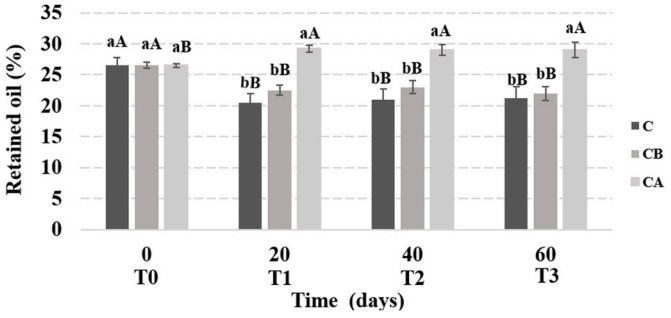

Fig. 2. Percentage of retained oil into the filling of C, CB and CA samples during the storage time.
$^{a-b}$ letters significantly differ at $p < 0.05$ for different samples at the same time of storage.
$^{A-B}$ letters significantly differ at $p < 0.05$ for the same sample at different time of storage.

the control and of a 17% for CB ones. Also for this parameter, a more drastic decrease was observed from T0 to T1, reaching similar values at the last storage times (from T1 to T3) corroborating the microstructural analysis results. This trend can be reasonably attributed to the phenomenon of oily fraction migration, from the filling to the shell, which tends to increase progressively over time. According to the literature, a high amount of oil loss from filling is related to poor quality pralines and so with their reduced shelf-life (Dalhemborg et al., 2015). On the contrary, in the CA pralines, there was an increase in the retained oily fraction at different times of storage, showing a percentage increase of 9.5 % from T0 to T3, which contributes to maintaining desirable structural and mouthfeel properties, also after 60 days at a higher storage temperature, more similar to the ones present in the sample at the beginning of storage. According to the literature, the fraction of oil remaining in the structure after applying external forces, mostly

centrifuges represents an index of the matrix's ability to retain its initial characteristics (Flöter et al., 2021; Sivakanthan et al., 202; Li et al., 2022).The CA behavior for this parameter, can also be reasonably explained by the high barrier effect displayed by the beeswax oleogel, as previously explained for microstructure results, which trapped the oily fraction inside the filling and prevented its migration, as well as by the high storage temperatures, which likely promoted fluidization of the solid lipid component while also promoting the release of some of the oil trapped inside the matrix.

3.3. Fundamental rheological analysis

3.3.1. Measurements in steady state

According to literature chocolate filling exhibited a typical plastic behavior, which was determined by the presence of a threshold (yield stress) that must be exceeded to let the samples flow (Juszczak et al., 2004). In Fig. 3, the extrapolated yield stress values, evaluated at 5 s^{-1} of shear rate for different samples during storage are showed. The graph underlines that in samples C and CB, an increase in yield stress was observed during storage probably due to particle aggregation, which increased the effort needed to flow samples. In specific, in control filling a linear increase was observed from T0 to T2 with values ranging from 70.9 to 911.9, reaching a plateau at T3. In pralines realized with the presence of a cocoa butter layer, a drastic increase in yield stress was noted from T1 to T2, followed by a decrease until T3, reaching values similar to the control ones. In both samples storage at higher temperatures affects in a negative way spreadability and the smoothness of these samples, underlined as cocoa butter layer was not able to preserve these quality characteristics during storage.

In CA samples, the trend was essentially straight and homogeneous during time, maintaining values around 100 Pa at high temperatures, consistent with previous findings (Glicerina et al., 2013; Lončarević et al., 2016), probably correlated with a less oil migration between filling and shell as previous showed, which gave rise to a more flowing and creamy filling (Afoakwa et al., 2009). These results showed the ability of beeswax oleogel barrier to preserve optimal rheological properties in filling pralines, that maintained values similar to initial ones even after two months of storage, demonstrated migration delaying properties, that can affect in positive way pralines shelf-life.

3.3.2. Measurements in oscillatory mode

In all samples the storage modulus (G′) (Fig. 4a), was higher than the dissipative module (G″) (Fig. 4b), indicating that the filling internal structure under non-destructive conditions is dominated by the elastic component (Bayod & Tornberg, 2011). In general, higher values of the conservative modulus, compared to the dissipative, are due to the presence of more aggregate structure (Glicerina et al., 2016). Oscillatory

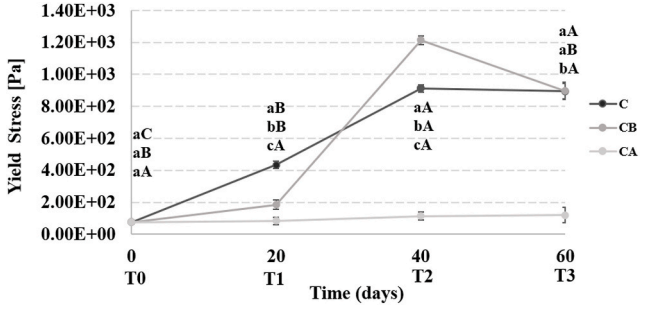

Fig. 3. Yield stress values of samples (C, CB and CA) at different storage times, measured evaluating the stress at 5 Pa of shear rate.
[a-c] letters significantly differ at $p < 0.05$ for different samples at the same time of storage.
[A–B] letters significantly differ at $p < 0.05$ for the same sample at different time of storage.

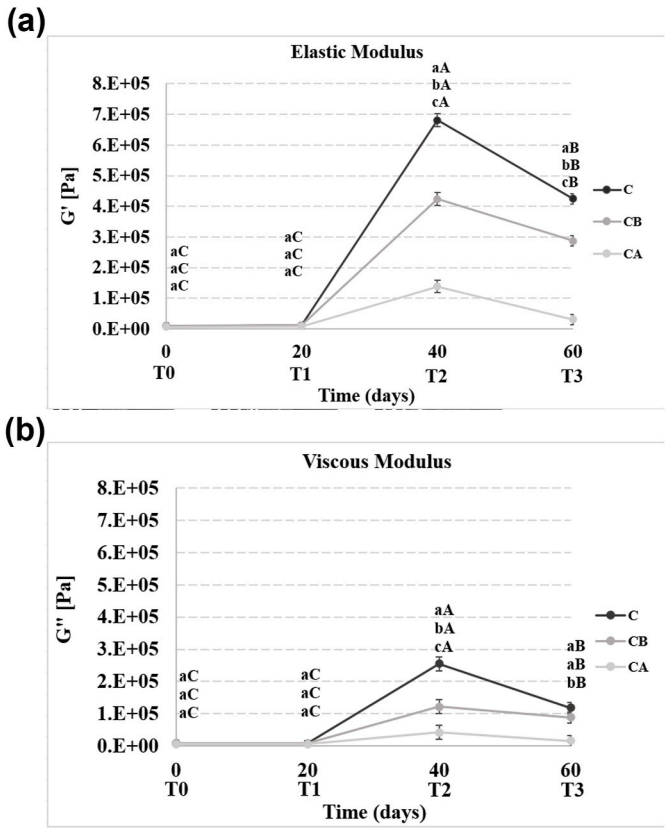

Fig. 4. (a,b). The storage (G′) and viscous modulus (G″) related to samples over time obtained in oscillatory mode.
[a-c] letters significantly differ at $p < 0.05$ for different samples at the same time of storage, for the same rheological parameter.
[A–B] letters significantly differ at $p < 0.05$ for the same sample at different time of storage, for the same rheological parameter.

analysis confirms the trend observed in stationary ones, (Fig. 4a) which underlines an increase in the elastic modulus (G′) during storage for all samples, especially for C and CB ones compared to CA, with initial values ranging from $7.01*10^3$ of C to $7.08 *10^3$ of CB in line with previous findings (Peressini et al., 2006; Glicerina et al., 2013; Aydemir, 2019). Both C and CB samples showed in fact a considerable increase of this parameter after 20 days of storage, with values around $6.3*10^5$ for C and $2.20*10^5$ for CB, tending slightly to decrease as we approach the end of storage, which suggests the presence of progressive matrix aggregation related to presence of a three-dimensional network of rigid particle aggregates and of a cream with a higher gumminess (Peressini et al., 2006). C that presents the highest G' values compared to CB and CA, which is indicative of greater oil migration, resulting in a more "gummy" and sticky filling structure, far from values considered suitable for nut or cocoa filling creams (Peressini et al., 2006; Glicerina et al., 2013; Aydemir, 2019).

3.4. Rheological empirical-imitative analysis

Empirical-imitative analysis, performed on the whole praline system, showed significant differences between samples also at T0, presumably related to the different types of barriers employed in both CB and CA and to their absence in sample C (Table 2). In fact, at time T0, the barrier consisting of high-melting cocoa butter increased the sample's resistance to cutting of CB pralines, that showed the highest significantly values (16.60 ± 0.84) compared to C (6.27 ± 1.68) and CA (1.40 ± 0.37); however, this resistance drastically decreased over time, reaching a percentage decrease at T3 of about 50 %, as a result of the high storage

Table 2
Textural properties of C, CB and CA samples during the storage, expressed as resistance to breakage and linear distance parameter.

		T0	T1	T2	T3
C	**F (N)**	6.27 ± 1.68^{bA}	2.80 ± 0.72^{bB}	7.03 ± 2.68^{aA}	5.40 ± 1.44^{aAB}
	LINEAR DISTANCE	38.62 ± 3.76^{bA}	32.61 ± 5.14^{aB}	40.75 ± 2.90^{aA}	31.65 ± 1.10^{aB}
CB	**F (N)**	16.60 ± 0.84 ^{aA}	7.49 ± 1.76^{aB}	8.12 ± 1.05^{aB}	8.36 ± 1.40^{aB}
	LINEAR DISTANCE	92.31 ± 10.72^{aA}	39.57 ± 4.20^{aB}	33.43 ± 4.24^{bB}	37.40 ± 3.95^{aB}
CA	**F (N)**	1.40 ± 0.37^{cA}	1.09 ± 0.80^{bA}	1.75 ± 0.68^{bA}	1.15 ± 0.22^{bA}
	LINEAR DISTANCE	2.27 ± 0.46^{cB}	19.99 ± 1.34^{bA}	23.42 ± 2.34^{cA}	24.43 ± 1.24^{bA}

$^{a-c}$ letter significantly differ at $p < 0.05$ for different samples at the same time of storage, within the same textural parameter.
$^{A-B}$ letters significantly differ at $p < 0.05$ for the same sample at different time of storage within the same textural parameter.

temperatures that led to a beginning in the partial melting of the cocoa butter layer, despite its higher melting point, but as well as to oil migration that according with literature (Gosh et al., 2002; Lonchampt & Hartel, 2004; Dahelenborg et al., 2015), dissolves some of the crystallized cocoa butter tryacylglycerol presents in the shell which leads to a softening of this part.

The CA sample demonstrated the lowest maximum force values compared to the other samples at each analyzed storage time, mainly due to the oleogel barrier, which reduced the resistance of CA to breakage, in relation to its gel-like and plastic structural characteristics (Martins et al., 2016). Also for this analysis as previously highlighted for yield stress one, no significant changes were observed upon storage indicating good structural stability. The control sample followed a similar pattern to CB, with the exception of T0, which showed a resistance to breakage decrease during storage of approximately 14%.

In terms of "linear distance" parameter, results confirmed what was previously discovered in fundamental rheological analysis. At T0, samples C and CB showed higher values, which are likely due to the presence of a more aggregated and gummy structure present inside the filling, that opposed higher resistance to blade sliding (Mousavi et al., 2019), in contrast to the CA sample, which had lower values, confirming the presence of a more spreadable and creamy filling, following a similar pattern to those shown for resistance to breakage parameters. Moreover an interesting trend was observed for CA sample, during storage in relation to linear distance. In CA samples in fact an increase in these parameters was observed during storage (from 2.27 ± 0.46 to 24.43 ± 1.24) despite the lowest yield stresses, elastic moduli and highest oil retaining values previously observed; this behavior can be probably attributed to the contribution of beewax oleogel that was absent in the previous analysis having been performed just on filling. According to literature (Vreeker, 1992; Omar et al., 2015) in fact, during storage in oleogel matrices, post-hardening or post-crystallization of oils and fats frequently occurs due to secondary nucleation giving rise to new small crystal formation that modifies the linearity of the structure and so its aggregation state involves an increase in linear distance parameter.

3.5. Spectrophotocolorimetric analysis

In Table 3 the lightness (L*) and hue angle (h°) values of pralines shell of analyzed samples are shown. All samples generally exhibit an increase in lightness during storage, with the CA samples showing a more stable trend. C sample showed a higher lightness increase after two months of storage even if not statistically different from CB. The lowest lightness value in CA samples compared to C and CB is probably related to a higher oil retention in the filling that contributes to maintain a more

Table 3
Colorimetric parameters (L* and h°) of shells of pralines during storage.

SHELLS				
Samples	T0	T1	T2	T3
Lightness (L*)				
C	36.18 ± 1.09^{aC}	39.53 ± 0.80^{aB}	39.03 ± 1.02^{aB}	42.38 ± 1.22^{aA}
CB	36.24 ± 1.11^{aC}	38.55 ± 0.76^{aA}	38.20 ± 0.37^{aA}	39.95 ± 1.86^{aAB}
CA	36.15 ± 1.07^{aA}	36.78 ± 0.37^{bA}	36.23 ± 0.13^{bB}	37.19 ± 0.36^{bB}
Hue angle (h°)				
C	54.37 ± 0.37^{aC}	56.34 ± 0.66^{aB}	57.60 ± 0.74^{aB}	59.01 ± 0.82^{aA}
CB	54.89 ± 0.45^{aB}	54.40 ± 0.48^{bB}	56.27 ± 1.11^{aA}	58.48 ± 1.94^{aA}
CA	54.29 ± 0.24^{aA}	54.91 ± 0.29^{bA}	55.38 ± 0.04^{bA}	54.90 ± 0.19^{bA}

$^{a-b}$ letter significantly differ at $p < 0.05$ for different samples at the same time of storage, within the same colorimetric parameter.
$^{A-C}$ letters significantly differ at $p < 0.05$ for the same sample at different time of storage within the same colorimetric parameter.

dark colour. According to literature (Ziegleder & Schwingshandl, 1998; Ghosh et al., 2002; Lonchampt & Hartel, 2004; Dahelenborg et al., 2015), in fact the migrating liquid fat from the filling can dissolve some of the crystallized tryacylglycerol present in cocoa butter, giving rise to the fat bloom phenomenon and so to a lightning of pralines shell, for that lower lightness values can be related to a lower oil migration.

In parallel, in relation to hue angle, in both C and CB pralines this parameter ranged from 54.37 to 59.01 in the first and from 54.89 to 58.48 in the latter after 60 days of storage, highlighting the presence of a more light brown color than CA in which values are quite similar during all the storage time.The addition of oleogel seems to have contributed to maintaining a more dark browning color, as T0, supports its barrier effect, while for the chroma index (data not shown) all samples showed a similar trend during storage.

4. Conclusions

Oil migration, especially at high storage temperatures, represents the main contributor to chocolate pralines quality loss and shelf life reduction, in terms of elasticity, mouthfeel and spreadability loss of filling, shell softening and bloom formation. In this paper, two different strategies were investigated to mitigate and enhance the quality of chocolate pralines in terms of microstructural, oil retention, rheological properties and colour of pralines: a high melting cocoa butter layer (CB) and an oleogel beeswax -based layer (CA) compared with a control (C). In pralines realized with the addition of beeswax oleogel better rheological properties in terms of yield stress, viscoelastic characteristics and gumminess of the filling were highlighted, strictly related to a lower filling oil loss as enhanced by oil retention analysis, as well as by microstructural evaluation. CA samples showed a more linear trend over all storage times, maintaining results more similar to T0 and closest to the optimal commercial ones, even after 60 days of storage. Moreover, in these samples, better colour retention in terms of high browning colour and less lightness reduction strictly related to fat blooming, was observed during all storage time. Pralines realized with cocoa butter high melting barriers presented intermediate performances between CA and C samples, in some cases not significantly different from control ones, especially at the last days of storage, underlining a progressive filling aggregation mass over storage, with an increase in the yield stress and elastic modulus that involve a reduction of spreadability and flowability of pralines filling. At the same time in both C and CB pralines a higher oil loss and lightness increase were observed during storage probably in relation to high oil migration and high fat blooming, which affected the quality characteristics of these products in a negative way.

Overall results demonstrated the efficiency of the oleogel barrier composed of beeswax and sunflower oil in delaying the migration of oil from the filling to the shell of chocolate pralines, compared to those realized with cocoa butter layer and control samples, also during storage. For this reason, on the basis of this preliminary study, the oleogel beeswax barrier can represent a valid strategy to retard oil migration and enhance the quality properties of chocolate pralines also during storage under conditions of thermal abuse, increasing their shelf life. However, starting from these promising results, additional combination of beeswax and oil needed to be investigated in order to highlight how different formulations can affect shelf life extension of pralines, parallel to a deep study focused on the sensory aspects of these products, to assess their consumers acceptability.

CRediT authorship contribution statement

Hazel Dilsad Tatar: Writing – original draft, Formal analysis, Data curation. **Virginia Teresa Glicerina:** Methodology, Investigation, Formal analysis, Conceptualization. **Roberta Foligni:** Writing – original draft, Supervision, Data curation. **Giuseppe Zeppa:** Writing – review & editing, Resources, Data curation.

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

The data that has been used is confidential.

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