LWT - Food Science and Technology 152 (2021) 112241

Contents lists available at ScienceDirect

LWT

journal homepage: www.elsevier.com/locate/lwt

Effect of the type and degree of alkalization of cocoa powder on the physico-chemical and sensory properties of sponge cakes

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ARTICLE INFO

Keywords:
Dutching
Bakery
Quality
Consumer's acceptance

ABSTRACT

Alkalization is a crucial process during cocoa processing to reduce its bitterness, improve solubility and develop color. Alkalization can be performed out at several points of the process (nib or cake), with different agents and at various intensities. All these variables may affect cocoa properties, but also physico-chemical and sensory properties of derived products (i.e. cakes). This work aims to evaluate the impact of alkalization type (nib vs. cake), alkalizing agent (K_2CO_3, $NaHCO_3$ and KOH) and process intensity (mild and strong) on the physico-chemical and sensory properties of sponge cakes. For this aim, 8 different alkalized powders were industrially produced and used in the preparation of sponge cakes. Alkalizing conditions significantly affected cocoa properties (pH, color, sensory properties) and those of the corresponding cakes (cake doughs color and rheology, as well as baked cake color and texture). In general, doughs prepared with cocoas alkalized under strong conditions were a 55 % darker and a 15 % less elastic. After baking, the corresponding cakes were a 17 % darker (L*) and a 12 % harder in texture. Despite these differences, all the cakes were equally rated by consumers in sensory terms demonstrating that for this application, alkalization variables do not condition consumer acceptability.

1. Introduction

Cocoa powder is widely used in the food industry as one of the main ingredients in confectionery, bakery and pastry products.

Obtaining cocoa powder begins with cocoa beans, which undergo fermentation, drying, deshelling and roasting processes (Miller et al., 2008). Unshelled and roasted cocoa beans are ground and liquefied by heating to obtain cocoa liquor, which is pressed to obtain a fatty fraction (cocoa butter) and a partially defatted fraction (cocoa cake). After subjecting the cocoa cake to a pulverization process, natural cocoa powder is obtained (Frauendorfer & Schieberle, 2006), which is characterized by its light brown color, and having a slightly acidic pH (5–6) (Kostic, 1997) and astringent taste (Quelal-Vásconez, Lerma-García, Pérez-Esteve, Talens & Barat, 2020).

In order to broaden their technological applications, cocoa nibs, cocoa liquor, cocoa cake and cocoa powder can be subjected to direct treatment with alkali, which is known as alkalization or the Dutching process (Kealey et al., 2005; Miller et al., 2008; Minifie, 2012; Rodríguez, Pérez, & Guzmán, 2009). This process consists of mixing any of the above-mentioned cocoa sources with an alkali solution, and treating this mixture with the combined effects of temperature and pressure.

During the alkalization process, pH increases to a range from 6 to 8 (Kostic, 1997). In addition to neutralizing the acids present in cocoa, the reactions between cocoa pigments and the alkali in the presence of oxygen and heat allow cocoa color to develop. In this way, the alkalized cocoa powder color darkens and its flavor is milder than that of natural cocoa powder. If cocoa powder is highly alkaline, it provides an even more intense flavor and a darker color, which is consistent with consumers' expectation of dark chocolate (Chau Loo, Tesén, & Valdez, 2013). For this reason, medium- and strong-alkalized cocoa powders are generally used in bakery applications.

In short, it can be stated that the alkalization process improves solubility, develops a darker color, removes acidity, astringency and bitterness (Valverde-Garcia, Perez Esteve, & Barat Baviera, 2020). However, the intensity of these changes depend on the cocoa process

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https://doi.org/10.1016/j.lwt.2021.112241
Received 8 April 2021; Received in revised form 28 July 2021; Accepted 30 July 2021
Available online 3 August 2021

stage in which alkalization takes place (nib, liquor, cake or powder), the type and concentration of alkali, and the intensity of other process variables (time, pressure, temperature, water percentage, etc.) (Dyer, 2003).

Alkalizing agent is generally established insofar as potassium carbonate and sodium hydroxide are the most widely employed alkali agents during alkalization. In line with the World Health Assembly's commitment to reduce the mean population sodium chloride intake by 30 % to <5 g/day by 2025 (World Health Organization (WHO), 2013) in the cocoa industry, sodium hydroxide has been replaced with potassium hydroxide. However, other alkaline agents are authorized, such as ammonium hydroxide, ammonium or magnesium carbonate, sodium, potassium or ammonium bicarbonate, ammonia gas, magnesium oxide, and mixtures of these (Codex Alimentarius, 1981). The alkali agent added during the cocoa alkalization process can impact baking properties in the same way as baking soda does. For this reason, sodium bicarbonate alkali is a good option in bakery products (Olam Cocoa, 2017).

The most economical option is cake alkalization as cocoa butter does not enter the production process and does not undergo any alteration process due to high temperatures and saponification processes: changes of fatty acids profile or setting characteristics, modification of unsaponifiable fraction... However, these cakes are traditionally less appreciated because they are considered to have a lower coloring capacity and a less intense aroma (Dyer, 2003). Moreover, during alkalization with nibs, more aromatic products are obtained whose colors are more reddish and intense, and less alkali is used in many cases (Olam Cocoa, 2017). For this reason, and although the product is more expensive because butter can alter and lose part of its commercial value, nib-alkalized cocoas are usually considered a better product (Moser, 2015).

Despite the large body of scientific literature on the effects of alkalinization on cocoa's nutritional, functional, microbiological and sensory characteristics (Valverde-Garcia, Perez Esteve, & Barat Baviera, 2020), there is still a research gap in the field to clarify the impact of cocoa powder characteristics on the physico-chemical and sensory properties of the different applications in which it can be used to, thus, evaluate how the food matrix empowers or masks these attributes.

In this context, the present work studies the effect that cocoa type has according to its processing (alkalization type, degree and type of alkali) on the physico-chemical and sensory properties of dough and cake whose formulations contain alkalized cocoa powder.

2. Materials and methods

2.1. Chemicals and ingredients

2.1.1. Ingredients for preparing cocoa powder

All the cocoa powders herein employed were supplied by Olam Food Ingredients S.L. (Cheste, Spain), who especially prepared these products for our study. The origin of the cocoas used as raw materials for the different alkalization treatments (cocoa cake and cocoa nibs) was Côte d'Ivoire. Three different alkalis were utilized: potassium carbonate (K_2CO_3) (Quimialmel, Castellón, Spain), sodium bicarbonate ($NaHCO_3$) (Barcelonesa, Cornellà de Llobregat, Spain) and potassium hydroxide (KOH) (Quimialmel, Castellón, Spain).

2.1.2. Cake preparation ingredients

The essential ingredients for preparing cakes, such as wheat flour (Aragonesa, Huesca, Spain), butter (Hacendado, Pozoblaco, Spain), white sugar (Pfeifer & Langer, Colonia, Germany), eggs (Huevos Guillen, Paterna, Spain), baking powder (Hacendado, Novelda, Spain) and pasteurized semi-skimmed milk (Hacendado, Urnieta, Spain), were bought in a local supermarket.

2.2. Cocoa alkalization process

Eight different alkalized cocoa powders were specifically prepared for this study at a pilot plant scale. They differed in terms of: a) the process stage in which alkalization took place: cocoa nibs (N) and cocoa cake (C-); b) alkalizing agents (potassium hydroxide -KOH-, potassium carbonate -K_2CO_3- and sodium bicarbonate -$NaHCO_3$-); c) alkalization intensity (mild and strong). Both alkalization process were done using batches of 100 kg of cocoa (nibs or cake), previously roasted. Nib alkalization was performed exclusively with potassium carbonate (K_2CO_3) as it is the most widespread one. Cake alkalization was performed with K_2CO_3, sodium bicarbonate ($NaHCO_3$) and potassium hydroxide (KOH) to study the effect of the alkaline agent on the properties of both the resulting cocoa powder and the final product. Processing parameters conditions such as amount of water or alkalizing agent, solution temperature, reaction time, pressure, drying time… were set to assure equivalent alkalization degrees between the nib and cake-alkalized processes. Values of these parameters are shown in Table 1.

In order to allow the equivalent raw material and manufacturing treatments in all the samples, they were especially produced for this study in various processing plants belonging to Olam Cocoa. Cake alkalization was performed in the pilot plant of Olam Food Ingredients Spain (Cheste, Valencia, Spain). The nib-alkalized cocoa powder processed under mild conditions (NM) was produced at the Unicao factory (Abidjan, Ivory Coast). Finally, the nib-alkalized cocoa powder processed under strong conditions (NS) was prepared at the deZaan factory (Koog aan de Zaan factory, The Netherlands). All the products were dried by using hot air (80 °C) until a moisture content of 35–45 g/kg was obtained.

2.3. Cocoa powder characterization

2.3.1. Extractable pH

The extractable pH in the cocoa powder and cocoa liquor was determined by following the methodology described in The deZaan Cocoa Manual (Olam Cocoa, 2017). For this purpose, a suspension of 2.5 g of cocoa powder/liquor was prepared in 12.5 mL of distilled water at 80 °C and stirred. Once powder was dispersed, another 12.5 mL-quantity of water at room temperature was added and the suspension was left to cool down to room temperature. Data collection was carried out in triplicate ($n = 3$) using a Crisonbasic 20+ pH meter (Barcelona, Spain), previously calibrated with technical solutions of known pH (4.01, 7.00 and 9.21 at 25 °C, Crison Instruments SA, Barcelona, Spain).

2.3.2. Extrinsic and intrinsic color

Extrinsic and intrinsic color was measured in the cocoa powder samples. To determine extrinsic color (dry cocoa powder color), a portion of dry cocoa powder was placed inside a methacrylate cuvette (20 mm thick) to ensure uniform powder distribution to guarantee sample opacity. To determine intrinsic color (cocoa dispersed in water), 5 g of the cocoa powder sample were suspended in 15 mL of distilled water at 60 °C and were stirred for 1 min. The mixture was left to cool

Table 1
Processing conditions for medium and strong alkalization intensities.

Parameter	Medium conditions	Strong conditions
Batch weight (kg)	100	100
Alkalizing salt (kg/100 kg of cocoa)	3	6
H_2O (kg/100 kg of cocoa)	20	30
Solution temperature (°C)	75	75
Reaction time (min.)	50	60
Pressure range (mbar)	1400–1600	1800–2000
Reaction set point (°C)	110	140
Drying time (min. after reaching 100 °C)	5	5

down to room temperature. Then it was vigorously stirred to resuspend possible settled particles and placed inside a methacrylate cuvette. A CM-3600d (Konica Minolta, Azuchi Machi, Japan) spectrocolorimeter was used to measure both, extrinsic and intrinsic color.

The results were expressed according to the CIE-Lab system (D65 illuminant and 10° viewing angle). Measurements were taken with an 8-mm diameter diaphragm inset. The measured parameters were L* (L* = 0 (black), L* = 100 (white)), a* (+a* = red) and b*(+b* = yellow). Using the a* and b* values, we calculated hue (h*ab) and chroma (C*ab) by the following equations:

$$h^*_{ab} = \arctan\frac{b^*}{a^*} \quad (1)$$

$$C^*_{ab} = \sqrt{a^{*2} + b^{*2}} \quad (2)$$

The mean values and standard deviation from three replicate measurements (n = 3) were calculated.

2.3.3. Sensory evaluation

Sensory analyses were performed according to the ISO 6658:2017 (International Organization for Standardization, 2017) by a trained panel made up of nine people (n = 9). The members of the panel were selected and trained according to the ISO 8586:2012 (International Organization for Standardization, 2012). To this end, 40 g of cocoa powders were mixed with 1L of tap water at 60 °C and stirred until a homogeneous suspension was obtained. Around 50 mL of this suspension were poured into coded cups. Samples were tested at a temperature close to 50 °C and seven different markers were analyzed: cocoa and chocolate taste, acidity, astringency, alkalinity, body, bitterness. The intensity of each attribute was scored from 1 to 5, where 0 represented no taste and 5 denoted a very intense taste. The coded samples were shown simultaneously and evaluated in a random order among the panelists.

2.4. Cocoa cake preparation

A typical cocoa sponge cake recipe was followed to prepare the different formulations. Each dough was prepared in a Thermomix TM 31 food processor (Vorwerk, Wuppertal, Germany) by mixing 150 g of sugar and 240 g of eggs at room temperature for 1 min at the speed of 3. Subsequently, 30 mL of milk and 250 g of butter were added to the mixture, which was stirred for 2 min at the same speed. Then 240 g of flour and 60 g of alkalized cocoa (4 soft-alkalized and 4 strong-alkalized) were added, and the dough was mixed for 3 min by increasing the speed to 4. Finally, 16 g of baking powder were added and was mixed for 3 min at the speed of 4.

Once doughs were obtained, 700 g of each one were transferred to a previously greased metal mold (14.5 cm × 11.0 cm), and were cooked for 50 min with medium dry air ventilation at 165 °C in a Self-CookingCenter® scc 62 oven (Rational, Landsberg am Lech, Germany). After baking, cakes were cooled to room temperature (approx. 2 h). Then the physico-chemical and sensory analyses were performed.

Each formulation was repeated 3 times on different days to include the maximum preparation variability. Every formulation was characterized the day of preparation following the procedures described in sections 2.5 and 2.6.

2.5. Dough characterization

2.5.1. pH

The pH in doughs was determined by a CrisonBasic 20+ pH meter (Crison, Barcelona, Spain) by embedding a puncture probe directly into dough. The pH measurements of doughs were taken in triplicate for each of the three independent formulation repetitions (n = 9).

2.5.2. Rheology

The rheological properties of the different cocoa doughs were determined under the steady state and dynamic conditions by a controlled stress rheometer (RheoStress, Thermo Haake, Karlsruhe, Germany) using plate-plate sensor geometry (60 mm in diameter) with a 1 mm gap. A stress sweep from 0.1 to 200 Pa at 1 Hz and 25 °C was first performed to establish the linear viscoelastic region (LVR). Then a frequency sweep from 0 to 15 Hz at 1Pa and 25 °C was performed to record the values of the elastic and viscous moduli and parameters G′ and G″, respectively. Tests were done in triplicate for each of the 3 independent formulation repetitions (n = 9).

2.5.3. Color

The color determination in sponge cake doughs was measured in triplicate following the procedure described in Section 2.3.2 for extrinsic color (measuring the sample after being transferred to the methacrylate cuvette). Tests were done in triplicate for each of the 3 independent formulation repetitions (n = 9).

2.6. Cake characterization

2.6.1. Moisture, water loss, density and rise

Moisture was determined by AOAC method 650.46 (AOAC, 1997). For this purpose, 3 g of sponge cake cooled to room temperature were weighed in duplicate and taken to an FD 115 heat/drying chamber (Binder, Tuttlingen, Germany), which was previously programmed at a temperature of 100 ± 1 °C overnight. Based on the difference in sample weight before and after drying, moisture content was determined by the following expression:

$$Moisture\ (g/100\ g) = \frac{Wi - Wf}{Wm} * 100 \quad (3)$$

where Wi = weight of the crucible, plus the wet sample (g); Wf = crucible weight, plus the dry sample (g); Wm = sample weight.

The total water loss during baking was determined by the following expression:

$$Water\ loss\ (g/100g) = \frac{Wi - Wf}{Wi} * 100 \quad (4)$$

where Wi = weight before baking (g); Wf = weight after baking (g).

In order to determine density, cakes were cut into 30 mm diameter x 30 mm high cylinders with a metal punch. Density was calculated by the ratio between the weight of cylinders and their volume.

The rise of cakes was determined by measuring the height of the cakes at the highest point of three central sections with the help of a graduated measuring tape.

Moisture determination, density and rise determinations were done in triplicate for each of the 3 independent cake repetitions (n = 9).

2.6.2. Color determination

The baked cake color was measured with the help of a CM-700d spectrocolorimeter (Konica Minolta, Ramsey, USA), conditioned with a CM-A181 standard mask for 3 mm diameter SAV (with no license plate). For measurements, a 10° visual angle, a D65 illuminator and the excluded specular component mode (SCE) were selected. Color was measured at seven different locations on the crust and crumb for each of the 3 independent cake repetitions (n = 21). The results were expressed in accordance with the equations described in Section 2.2.2.

2.6.3. Texture

The texture parameters hardness (N), elasticity (dimensionless), cohesiveness (dimensionless), and Resilience (dimensionless) defined by Bourne (1978) of sponge cakes were determined by a double compression test (TPA) in the TA-XT plus texture analyzer (Stable Micro Systems, Godalming, England), equipped with a SP/75 compression plate. The probe speed was 1 mm/s (pre-test and test) and 2 mm/s (post-test), with 50 % deformation and a 30-s waiting time between compressions. For this analysis ten pieces of each cake were cut (30 mm diameter x 30 mm height) and analyzed (n = 30).

2.7. Sensory analysis

The degree of acceptance of the different sponge cakes by a panel of consumers was evaluated by hedonic tests. The panel of tasters was formed by 40 students and research staff members from the Food Technology Department of the Universitat Politècnica de València (Spain), who were frequent chocolate cake eaters. Samples were evaluated in a tasting room equipped with individual cabins (International Organization for Standardization, 2007). They were coded with randomly chosen 3-digit numbers. Acceptance tests were performed on a 7-point hedonic scale (1 I absolutely dislike, 2 I considerably dislike, 3 I somewhat dislike, 4 I neither dislike nor like, 5 I somewhat like, 6 I considerably like, 7 I absolutely like). This scale was used to describe all the following parameters defined: color, sponginess, taste, chocolate aroma and overall appreciation. To minimize any residual effects, consumers were asked to rinse their mouths with water before testing each sample.

2.8. Statistical analysis

Normality was tested using Shapiro-Wilk test ($\alpha < 0.05$) and homogeneity of variance was tested using Levene's test. The differences between samples due to cocoa type (alkalization type, alkali type and the intensity of the alkalization process) employed in the formulation of the different cakes were determined by an analysis of variance (ANOVA), followed by Tukey's post hoc test were used when the data was normally distributed and the variance was homogeneous. For nonparametric statistics, the Kruskal-Wallis test and Dunn's multiple comparison test were used. Data were analyzed by XLSTAT 2020.3.1 (New York, USA). ANOVAs were done by using the STATGRAPHICS Centurion XVI v. 17.2.04 statistical software (Manugistics Inc., Rockville, MD, USA). All data presented in tables are expressed as as averages and pooled standard deviations. Pooled standard deviations make no assumption about the distribution and are much more robust than standard deviations calculated on only part of the data. For its calculation it was followed the procedure suggested by Renard (2021).

3. Results and discussion

3.1. Cocoa powder characterization

3.1.1. Extractable pH

The pH value is an indicator of the degree of cocoa alkalization (Valverde-Garcia, Perez Esteve, & Barat Baviera, 2020). This determination allows samples to be classified into different categories: light alkalized cocoa (pH 6.0–7.2), medium alkalized cocoa (pH 7.2–7.6) and strong alkalized cocoa (pH > 7.6), according to the guidelines of Miller et al. (2008). As shown in Table 2, the minimum pH in the alkalized samples was 6.6 ± 0.1. Bearing in mind that the pH of the raw materials was 5.4 ± 0.1, it can be stated that the alkalization process was successful in both the different alkalization treatments (nib and cake).

According to Miller's classification (Miller et al., 2008), by applying mild-alkalizing conditions, two different alkalization levels were achieved: light-alkalized ($NaHCO_3$, K_2CO_3) and medium-alkalized (KOH). However, under strong alkalizing conditions, the obtained products were classified as medium- ($NaHCO_3$) and strong-alkalized (K_2CO_3 and KOH). When K_2CO_3 was used as the alkalizing salt, no significant differences were found in pH between the samples alkalized in nib and cake, which confirmed the equivalent conditions of treatments (Olam Cocoa, 2017).

In this way, at the same alkali concentration, the alkalization salt

Table 2
pH values of the obtained cocoa powders.

	Mild conditions	Strong conditions
N_K_2CO_3	6.7^{aA}	8.0^{bB}
C_K_2CO_3	6.8^{aA}	7.9^{bB}
C_KOH	7.4^{bA}	8.1^{bB}
C_$NaHCO_3$	6.6^{aA}	7.1^{aB}
Pooled standard deviation (n = 3)	0.1	0.1

N#_K_2CO_3 = Nib-alkalized cocoa with K_2CO_3; C#_K_2CO_3 = Cake-alkalized cocoa with K_2CO_3; C#_KOH = Cake-alkalized cocoa with KOH; C#_$NaHCO_3$=Cake-alkalized cocoa with $NaHCO_3$; # = M = mild alkalization conditions; # = S = strong alkalization conditions. Values with different lowercase letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoa powders with different alkalizing agents or type of alkalization (Nib (N) or Cake (C)) for the same percentage of alkali. Values with different capital letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoas with different percentages of alkali for the same alkalizing agent or type of alkalization. Data were analyzed with Kruskal-Wallis test and Dunn's multiple comparison test.

determined the final cocoa pH (Moser, 2015). The salt with the highest capacity to increase pH was potassium hydroxide (KOH), followed by potassium carbonate (K_2CO_3) and sodium bicarbonate ($NaHCO_3$). This ability of hydroxides to increase the pH of cocoa during alkalization to a greater extent than carbonates or bicarbonates is because hydroxides are stronger bases (Srivastava & Misra, 2015).

3.1.2. Extrinsic and intrinsic color

In order to evaluate the effect of alkalization type (cake or nib), alkalization intensity (mild or strong) and alkali type (KOH, K_2CO_3 and $NaHCO_3$) on cocoa powder color, this property was measured by two different approaches. On the one hand, the dried powder color (extrinsic color) was determined because it is the parameter that normally characterizes cocoa specification. However, this parameter is conditioned by the amount and polymorphic forms of cocoa butter (Olam Cocoa, 2017). Thus, two powders may appear different in terms of their extrinsic color and look like a similar color in a certain application. For this reason, the color of the cocoa suspended in water (intrinsic color) was also determined as it is considered to correlate better with the color of the powder in an application (Dyer, 2003). This has also been described by Moser (2015), who advises that, in order to properly evaluate cocoa impact when using cocoa in an application, it is always necessary to examine cocoa's true intrinsic color. In fact, early indicators can be misleading, such as extrinsic color.

Table 3 shows the color coordinates for both extrinsic and intrinsic colors. As we can see, the three processing variables (salt type, concentration and alkalization type) significantly affected both color types.

The sample made with $NaHCO_3$ was the lightest in color (highest L* value), in both the extrinsic and intrinsic colors at the mild- and strong-alkalization intensities. The darkest sample was that made with KOH. Significant differences were found between the mild-and strong-alkalizing intensities when comparing the same alkalizing salt.

For chroma (C *), differences were found between the alkalization intensities (mild and strong) in the extrinsic and intrinsic colors. The sample with the most saturated color was that formulated with $NaHCO_3$ for the mild- and strong-alkalizing conditions. The lower color purity was for the samples made with KOH.

In the values shown for h*, and also for extrinsic and intrinsic colors, the mild-alkalization conditions provided a higher h* value compared to the strong-alkalization conditions, which reaffirmed the capacity of alkalization to go from yellowish to reddish tones due to the polymerization of polyphenols (Valverde-Garcia, Perez Esteve, & Barat Baviera, 2020).

These differences between KOH, K_2CO_3 and $NaHCO_3$ were due to the different salt strengths (Rodríguez et al., 2009).

The correlation between extrinsic and intrinsic colors was quite

Table 3
Extrinsic and intrinsic color of the obtained cocoa powders.

	Extrinsic color			Intrinsic color		
	L*	C*	h*	L*	C*	h*
NM_K_2CO_3	36.8^{bB}	26.4^{bB}	55.2^{bB}	16.3^{cB}	19.2^{cB}	49.75^{bB}
CM_K_2CO_3	36.1^{aB}	26.1^{bB}	54.7^{bB}	15.5^{bB}	18.5^{bB}	49.2^{bB}
CM_KOH	35.7^{aB}	23.8^{aB}	52.7^{aB}	13.6^{aB}	16.2^{aB}	45.5^{aB}
CM_$NaHCO_3$	39.6^{bB}	28.2^{cB}	55.0^{bB}	18.6^{dB}	19.9^{dB}	51.4^{cB}
NS_K_2CO_3	25.5^{bA}	20.2^{bA}	47.7^{bA}	11.0^{cA}	9.8^{cA}	36.25^{bA}
CS_K_2CO_3	24.8^{bA}	18.6^{aA}	48.4^{cA}	9.1^{bA}	8.7^{bA}	36.1^{bA}
CS_KOH	22.0^{aA}	18.2^{aA}	46.0^{aA}	7.0^{aA}	7.9^{aA}	34.7^{aA}
CS_$NaHCO_3$	28.8^{cA}	20.7^{bA}	49.9^{dA}	12.8^{dA}	9.5^{cA}	41.4^{cA}
Pooled standard deviation (n?=?3)	0.3	0.2	0.4	0.3	0.3	0.4

N#_K_2CO_3 = Nib-alkalized cocoa with K_2CO_3; C#_K_2CO_3 = Cake-alkalized cocoa with K_2CO_3; C#_KOH = Cake-alkalized cocoa with KOH; C#_$NaHCO_3$ = Cake-alkalized cocoa with $NaHCO_3$; # = M = mild alkalization conditions; # = S = strong alkalization conditions. L * = luminosity; C * = chroma; h * = hue. Values with different lowercase letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoa powders with different alkalizing agents or type of alkalization (Nib (N) or Cake (C)) at medium (M) or strong (S) alkalization conditions. Values with different capital letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoas with different percentages of alkali for the same alkalizing agent or type of alkalization. Data were analyzed with Kruskal-Wallis test and Dunn's multiple comparison test.

notable. Regarding the L* value, the correlation was 0.80 for the cocoa powders alkalized under mild conditions and 0.95 for those processed under strong ones. Similar values were obtained for C* and h* with correlation values of 0.94 and 0.87 or 0.84 and 0.80 for the mild- and strong-alkalization conditions, respectively. Although a correlation existed, it was not 100 %. Therefore, it was not possible to accurately predict intrinsic color from extrinsic color, so both parameters should always be measured.

3.1.3. Sensory profile

Fig. 1 shows the sensory profiles of the obtained cocoa powders. The cocoas alkalized under mild conditions presented more cocoa, chocolate, body, acidity and astringency, and less alkalinity tones than those alkalized under the strong conditions.

Of the samples alkalized under the mild conditions, NM_K_2CO_3 and CM_K_2CO_3 cocoas presented more chocolate flavor, body, astringency, and alkali taste, while CM_KOH and NM_K_2CO_3 presented more bitterness. For the samples alkalized under the strong conditions, all the samples had a similar flavor profile. This finding highlights that CS_K_2CO_3 and CS_KOH had more intense bitterness and alkali taste, and that cocoa flavor was more present in the sample alkalized in nibs. These results confirmed the impact of the alkalization conditions of the final sensory attributes and also provide one of the few examples of published data on how the cocoa sensory profile is modified according to different alkalization parameters.

3.2. Dough characterization

3.2.1. pH and color

Table 4 shows the pH and color values of the cake doughs prepared with the different alkalized cocoa powders. Any type and color of cocoa can be added to a bakery recipe, but much attention should be paid to pH (Moser, 2015). Generally, bakery doughs with a lower pH, linked with lactic acid production during fermentation, require a slightly shorter mixing time and are less stable than doughs with a normal pH level (Delcour & Honseney, 2010; Wehrle, Grau, & Arendt, 1997). In our case, although the pH of cocoas varied between 6.7 and 8.1, the pH of the doughs ranged between 7.4 and 7.7. These data confirmed that the cake dough pH is generally established by the main ingredients creating a

Table 4
pH and color of the doughs prepared with different alkalized cocoa powders.

	pH	Color		
		L*	C*	h*
NM_K_2CO_3	7.5^{A}	18^{abB}	16.8^{abB}	47.4^{abB}
CM_K_2CO_3	7.5^{A}	19^{bB}	$16.^{abB}$	46.4^{aB}
CM_KOH	7.5	17^{aB}	15.8^{aB}	46.3^{aB}
CM_$NaHCO_3$	7.6	25^{cB}	18.8^{bB}	51.9^{bB}
NS_K_2CO_3	7.7^{bB}	13^{bA}	9.1^{bA}	39.9^{aA}
CS_K_2CO_3	7.7^{bB}	13^{abA}	9.0^{abA}	40.1^{abA}
CS_KOH	7.5^{ab}	11^{aA}	8.8^{aA}	40.1^{abA}
CS_$NaHCO_3$	7.4^{a}	14^{cA}	9.7^{bA}	42.9^{bA}
Pooled standard deviation (n = 9)	0.1	2	0.5	0.6

N#_K_2CO_3 = Nib-alkalized cocoa with K_2CO_3; C#_K_2CO_3 = Cake-alkalized cocoa with K_2CO_3; C#_KOH = Cake-alkalized cocoa with KOH; C#_$NaHCO_3$ = Cake-alkalized cocoa with $NaHCO_3$; # = M = mild alkalization conditions; # = S = strong alkalization conditions. L * = luminosity; C * = chroma; h * = hue. Values with different lowercase letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoa powders with different alkalizing agents or type of alkalization (Nib (N) or Cake (C)) at medium (M) or strong (S) alkalization conditions. Values with different capital letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoas with different percentages of alkali for the same alkalizing agent or type of alkalization. Data were analyzed with Kruskal-Wallis test and Dunn's multiple comparison test.

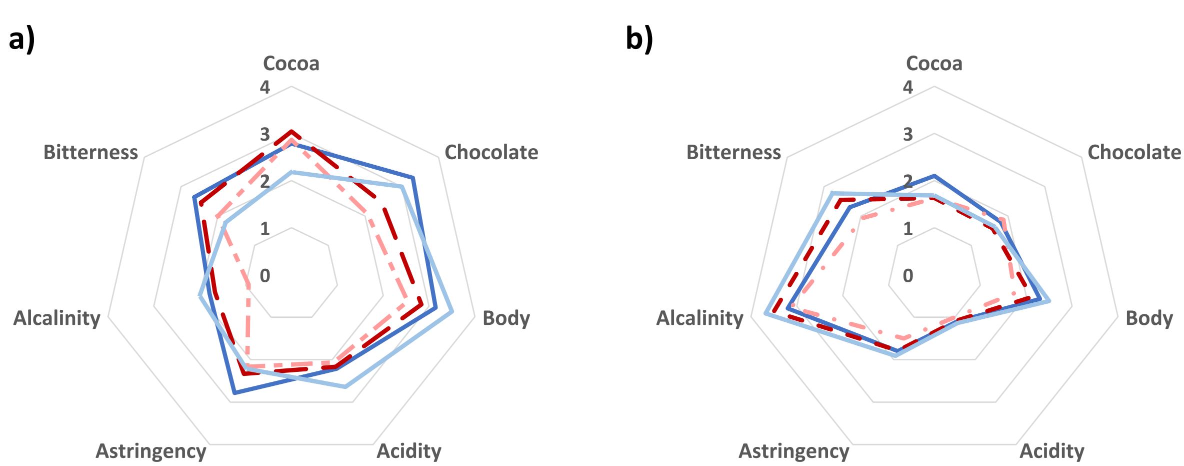

Fig. 1. Sensory profiles of cocoa samples alkalized in mild (a) and strong (b) conditions. N_K_2CO_3 (dark blue squares), C_K_2CO_3 (light blue circles), C_KOH (dark red rhombuses), C_$NaHCO_3$ (light red triangles). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

buffering effect (Baik, Marcotte, & Castaigne, 2000) and are merely affected by the employed cocoa. Thus, the type of cocoa should not affect to the needed mixing time.

For color, statistically significant differences ($p < 0.05$) were observed between the samples due to the three factors: the degree, type of alkalization and the alkali agent.

In agreement with the color of the cocoa powders used in the formulation, the dough with the lighter color (lower L* value) was that prepared with the C#_ $NaHCO_3$ cocoa, followed by that prepared with N#_K_2CO_3 under both the mild (M) and strong (S) alkalizing conditions. In all cases, and as expected, the samples formulated under the strong-alkalization conditions were darker than those made under the mild ones.

Regarding color purity values (C*), the dough formulated with cocoas alkalized using a mild alkalization intensity exhibited a purer color than those prepared with the cocoas alkalized under strong conditions. The dough made with the C#_ $NaHCO_3$ cocoa was purer in color under the mild- and strong-alkalization conditions. The sample with less color purity was made with KOH.

Significant differences appeared in the h* values between the mild and strong alkalization conditions when each alkalized cocoa powder was employed. In accordance with cocoa powders (see Table 3), in all the cases strong conditions gave a more reddish hue. At the two alkalization conditions (M and S), statistically significant differences ($p < 0.05$) were found between the hue of the doughs made with the alkalized cocoa in the cakes with sodium bicarbonate (C#_$NaHCO_3$) and the rest. No statistically significant differences ($p < 0.05$) were observed between alkalization in nib (N#_K_2CO_3) and cake (C#_K_2CO_3) for either alkalization conditions.

When comparing the dough color values with the cocoa powder color results, the correlation between them was high. The correlation for the L* value was 0.86 if extrinsic color was compared and was 0.87 if intrinsic color was compared. The correlation values for C* and h* were 0.95 and 0.85 and 0.98 or 0.92 for extrinsic or intrinsic color, respectively. In short, the correlation between cocoa powder color and dough was quite relevant and higher if intrinsic color was compared. However, lack of a total correlation between cocoa powder and the corresponding cocoa dough indicates that cocoa color in a certain application not only depends on its own interaction, but also on its interactions with other food constituents, such as proteins, fat or polysaccharides.

3.2.2. Rheology

In addition to color and flavor, alkalization type is believed to affect the water-holding capacity of cocoa in pastry and confectionery products, and that nib-alkalized cocoa retains much more water than cake-alkalized cocoa to, thus, affect cocoa rheology (Olam Cocoa, 2017).

In order to evaluate this possible affects, the dynamic strain sweep data of all the doughs were studied. Fig. 2 depicts the evolution of the storage modulus values (G′, which measures the elastic component) and the loss module (G″, which measures the plastic component) according to the frequency with which cakes are prepared with the different types and degrees of cocoa powder alkalization. This measurement is important because cake volume and texture correlate with dough rheological properties (Sahin, 2008).

The doughs prepared with cocoas under the mild alkalization conditions (Fig. 1a) obtained higher G' values than those for G″, which indicates greater elastic behavior. This behavior was also evident in the sponge cakes under the high alkalization conditions (Fig. 1b).

Among the doughs prepared with the alkalized cocoa powders under the mild conditions (Fig. 2a), the dough prepared with the N#_K_2CO_3 cocoa showed dominating elastic and viscous modulus than the dough prepared with C#_K_2CO_3. When comparing the rheology of the doughs prepared with alkalized cake cocoas, alkalization with K_2CO_3 conferred dough more elasticity than the other alkalis. No statistical differences ($p > 0.05$) in viscous modulus were observed.

Under the strong conditions (Fig. 2b), alkalization type (nib and cake) did not affect either elastic or viscous modulus. However, when comparing the values of the doughs containing alkalized cake powders, the dough containing the cocoa powder alkalized with KOH (CS_KOH) displayed more elasticity than the other doughs. No differences were found in viscous modulus among the different formulations. These data indicate that the degree of alkalization is the factor that most affects dough rheology when cocoa is incorporated, followed by alkalization type or the employed alkalizing agent.

By taking the rheology results into account, it can be concluded that alkalization type (nib or cake) affects dough rheology. However, in this formulation, this process variable could influence rheology to the same extent as the degree of alkalization or alkali type.

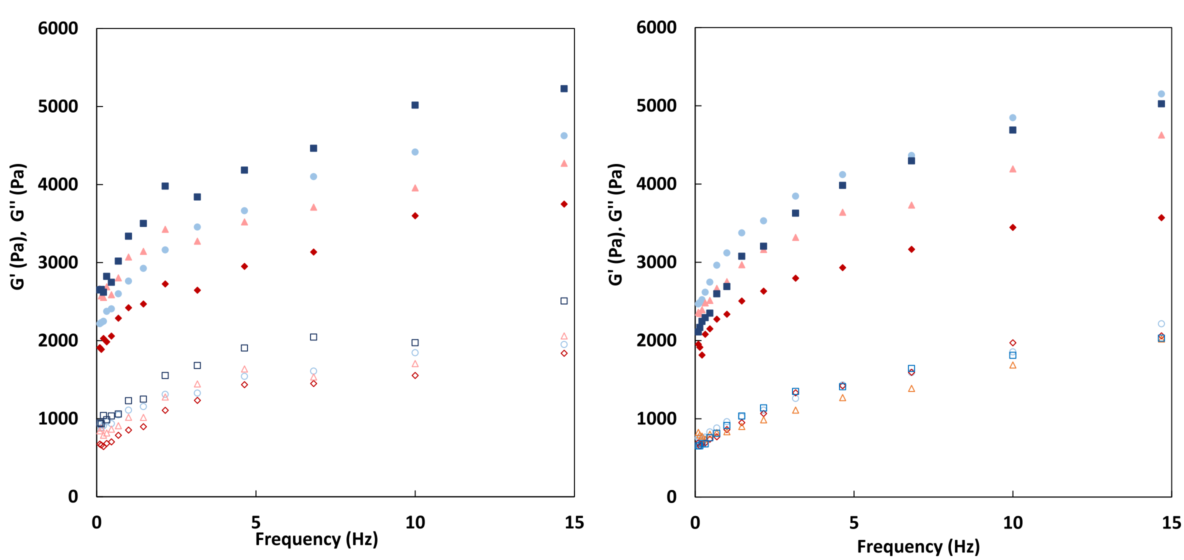

Fig. 2. Viscoelastic properties of sponge cakes made cocoa samples alkalized in mild (a) and strong (b) conditions. G' (filled symbols), G'' (white symbols). N_K_2CO_3 (dark blue solid line), C_K_2CO_3 (light blue dotted line), C_KOH (dark red dashed line), C_$NaHCO_3$ (light red dash-dotted line). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3.3. Cake characterization

3.3.1. Water loss, moisture, density and rise of cakes

Table 5 offers the values obtained for water loss, moisture, density and rise of cakes. It is important to study these variables because the alkali concentration might affect final product properties. For example, baking products can be affected by introducing certain alkalized cocoas due to the alkalizing agent's leavening effect (Olam Cocoa, 2017). However as observed, neither the type or degree of alkalization of the cocoa powder used in the sponge cake formulation had a statistically significant effect ($p > 0.05$) on the water loss, moisture, rise noted for doughs during baking and density. These data also confirmed that the possible water-holding capacity modification due to different alkalization process did not affect moisture or water loss after baking.

3.3.2. Color determination

Table 6 shows the L *, C *, h* values of cake crust and crumb. When the cake-alkalized cocoa with sodium bicarbonate under the mild and strong alkalization conditions (C#_ $NaHCO_3$) was used, sponge cake crust became lighter and had yellowish color with higher saturation. Under the mild-alkalization conditions, the darkest color with less color purity was obtained when using KOH as alkalizing salt. No statistically significant differences were found among cake- and nib-alkalized cocoas with K_2CO_3 for most of the analyzed parameters. These results agree with the color trends observed in the powder in which they were prepared.

Similarly, with sponge cake crumb, the use of cake-alkalized cocoa under the mild conditions of sodium bicarbonate (CM_ $NaHCO_3$) generated a lighter, more saturated and yellower crust. Under the strong alkalizing conditions, the cake-alkalized cocoa with potassium hydroxide (CS_KOH) conferred crumb more darkness and less color purity. The cake alkalinized with sodium bicarbonate (CS_ $NaHCO_3$) gave a more yellowish tone than the other samples.

When these data were correlated with the color coordinates of the cocoa powders, the correlation was around 0.95 for the L* value, 0.97 for the C* value and 0.86 for the h* value when the intrinsic color values were used.

3.1.1. Texture

Regarding cake texture, the texture profile analysis (TPA) found that the type of alkalization and alkali used to manufacture cocoas had a statistically significant effect ($p < 0.05$) on the hardness, cohesiveness, resilience and elasticity of cakes. Table 7 shows that the cakes with the cocoas prepared under the mild alkalization conditions presented greater hardness than those prepared under the strong alkaline conditions. For the mild alkalization conditions, the use of alkalized cocoa with potassium carbonate (K_2CO_3) in nib or cake conferred cake greater hardness than the other alkalis, and no statistically significant differences were observed between both. Under the strong alkalizing conditions, the hardness of the cake formulated with NS_K_2CO_3 was greater than when CS_K_2CO_3 was used.

Cakes prepared with cocoa alkalized with K_2CO_3 obtained lower cohesiveness (internal food structure resistance) and resilience (ratio of recoverable energy as the first compression is released) values than those prepared with KOH or $NaHCO_3$. Statistical differences were found ($p < 0.05$) for alkalization intensities (mild or strong).

Regarding elasticity, the cakes prepared with cocoas alkalized with mild conditions presented less elasticity than those prepared with cocoas alkalized in strong ones. Under the mild alkalization conditions, the

Table 5
Moisture, water loss, rise and density of the cakes prepared with different alkalized cocoa powders after baking.

	Moisture	Water loss	Rise	Density
NM_K_2CO_3	26.0	10.2	7.3	0.47
CM_K_2CO_3	26.8	10.6	8.0	0.48
CM_KOH	26.7	10.5	8.5	0.472
CM_$NaHCO_3$	25.9	10.5	8.3	0.47
NS_K_2CO_3	26.6	11.0	9.0	0.47
CS_K_2CO_3	26.5	10.7	8.7	0.46
CS_KOH	26.4	10.6	8.0	0.47
CS_$NaHCO_3$	26.1	10.2	8.6	0.46
Pooled standard deviation (n = 9)	0.9	0.4	0.4	0.02

N#_K_2CO_3 = Nib-alkalized cocoa with K_2CO_3; C#_K_2CO_3 = Cake-alkalized cocoa with K_2CO_3; C#_KOH = Cake-alkalized cocoa with KOH; C#_$NaHCO_3$ = Cake-alkalized cocoa with $NaHCO_3$; # = M = mild alkalization conditions; # = S = strong alkalization conditions. The differences found between cakes prepared with cocoa powders with different alkalizing agents or type of alkalization (Nib (N) or Cake (C)) at medium (M) or strong (S) alkalization conditions were not statistically significant ($p > 0.05$) using the Kruskal-Wallis test.

Table 6
Color of the crust and crumb of the cakes prepared with different alkalized cocoa powders.

	Crust			Crumb		
	L*	C*	h*	L*	C*	h*
NM_K_2CO_3	35^{bB}	17.0^{aB}	49.2^{aB}	21^{bB}	12.5^{aB}	42^{bB}
CM_K_2CO_3	34^{bB}	17.0^{aB}	50.1^{abB}	21^{bB}	12.6^{aB}	42^{abB}
CM_KOH	33^{aB}	16.4^{aB}	52.3^{bB}	19^{aB}	11.7^{aB}	41^{a}
CM_$NaHCO_3$	37^{cB}	18.3^{bB}	55.9^{cB}	22^{bB}	15.7^{bB}	46^{cB}
NS_K_2CO_3	29^{aA}	11.3^{bA}	42.7^{aA}	17^{bA}	9.5^{cA}	37^{aA}
CS_K_2CO_3	29^{aA}	10.0^{bA}	45.0^{bA}	17^{abA}	6.9^{bA}	39^{abA}
CS_KOH	29^{aA}	8.5^{aA}	46.2^{bA}	16^{aA}	5.5^{aA}	40^{b}
CS_$NaHCO_3$	32^{bA}	11.3^{bA}	49.0^{cA}	18^{bA}	10.0^{cA}	42^{cA}
Pooled standard deviation (n = 21)	2	0.5	0.9	2	0.6	1

N#_K_2CO_3 = Nib-alkalized cocoa with K_2CO_3; C#_K_2CO_3 = Cake-alkalized cocoa with K_2CO_3; C#_KOH = Cake-alkalized cocoa with KOH; C#_$NaHCO_3$ = Cake-alkalized cocoa with $NaHCO_3$; # = M = mild alkalization conditions; # = S = strong alkalization conditions. L * = luminosity; C * = chroma; h * = hue. Values with different lowercase letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoa powders with different alkalizing agents or type of alkalization (nib or cake) for the same percentage of alkali; Values with different capital letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoas with different percentages of alkali for the same alkalizing agent or type of alkalization. Data were analyzed with Kruskal-Wallis test and Dunn's multiple comparison test.

Table 7
TPA parameters for sponge cakes prepared with different alkalized cocoa powders.

	Hardness	Cohesiveness	Resilience	Elasticity
NM_K_2CO_3	13^{bB}	0.57^{aA}	0.23^{aA}	0.89^{aA}
CM_K_2CO_3	13^{bB}	0.58^{a}	0.24^{aA}	0.90^{aA}
CM_KOH	11^{aB}	0.63^{bB}	0.27^{bB}	0.92^{bB}
CM_$NaHCO_3$	10^{aB}	0.63^{bB}	0.27^{b}	0.91^{abA}
NS_K_2CO_3	11^{bA}	0.60^{aB}	0.25^{aB}	0.92^{bB}
CS_K_2CO_3	10^{aA}	0.60^{a}	0.26^{bB}	0.92^{bB}
CS_KOH	10^{aA}	0.59^{aA}	0.26^{bA}	0.90^{aA}
CS_$NaHCO_3$	10^{aA}	0.62^{bA}	0.27^{b}	0.92^{bB}
Pooled standard deviation (n = 30)	2	0.03	0.02	0.02

N#_K_2CO_3 = Nib-alkalized cocoa with K_2CO_3; C#_K_2CO_3 = Cake-alkalized cocoa with K_2CO_3; C#_KOH = Cake-alkalized cocoa with KOH; C#_$NaHCO_3$ = Cake-alkalized cocoa with $NaHCO_3$; # = M = mild alkalization conditions; # = S = strong alkalization conditions. Values with different lowercase letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoa powders with different alkalizing agents or type of alkalization (nib or cake) for the same percentage of alkali. Values with different capital letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoas with different percentages of alkali for the same alkalizing agent or type of alkalization. Data were analyzed with Kruskal-Wallis test and Dunn's multiple comparison test, since only Hardness passed the normality and homoscedasticity tests.

alkalinized cake cocoa with potassium hydroxide (C#_KOH) provoked greater elasticity in sponge cakes than the other employed alkalis. Cakes prepared with cocoas alkalized under the strong conditions showed a lower elasticity. No statistically significant differences ($p > 0.05$) were observed for elasticity between cakes prepared with cocoas alkalized in nib (N#_K_2CO_3) and cake (C#_K_2CO_3) at either alkalization intensity.

3.1.2. Sensory analysis

In parallel to the instrumental evaluation, cake samples were sensory-evaluated by a panel of untrained consumers ($n = 40$). To measure product liking and preference, the hedonic scale is a unique scale that provides reliable and valid results (Stone, Bleibaum, & Thomas, 2012). Consumers were asked to evaluate six sample attributes on a 7-point hedonic scale. The sensory test results are shown in Table 8. Despite the number of testers could limit the reliability of results, the statistical analysis revealed no statistically significant differences ($P > 0.05$) in color, sponginess, taste, chocolate flavor, mouthfeel and overall appreciation terms, and demonstrated the degree of similarity between the samples that differed in the process stage in which alkalization was produced (cocoa nibs and cocoa cake) with alkalizing agents (KOH, K_2CO_3 and $NaHCO_3$) and alkalization intensity (mild and strong). These results were surprising because it was considered that the selection of specific cocoas to prepare certain products would be important to guarantee consumer acceptance (Shankar, Levitan, Prescott, & Spence, 2009). In this case, and despite color and sensory profile determinations being significantly different among samples, consumer acceptance was similar which, thus, indicates in this case that the matrix can mask most cocoa attributes.

However, the data revealed that despite there being no correlation between instrumentally measured color and sensory preference, the instrumental color crumb values showed a good correlation ($r = 0.77$) for the global evaluation. In this way, darker cocoa cakes (lower L* values) obtained a better global evaluation value (KOH alkalizing agent), which confirmed what was previously established: darker products are generally better appreciated by consumers as color has a strong impact on perceived flavor (Spence, 2015).

Table 8
Sensory attribute scores for sponge cakes prepared with different alkalized cocoa powders.

	Color	Sponginess	Taste	Chocolate aroma	Global evaluation
NM_K_2CO_3	7^a	5	6	6	6
CM_K_2CO_3	7^a	6	5^A	6	6
CM_KOH	7^a	6	6	6	6
CM_$NaHCO_3$	5^b	6	5^A	6	6
NS_K_2CO_3	6	6	7	6	6
CS_K_2CO_3	7	6	7^B	6	6
CS_KOH	7	6	7	6	7
CS_$NaHCO_3$	7	6	7 ^{B}	6	6
Pooled standard deviation (n = 40)	2	2	2	2	2

N#_K_2CO_3 = Nib-alkalized cocoa with K_2CO_3; C#_K_2CO_3 = Cake-alkalized cocoa with K_2CO_3; C#_KOH = Cake-alkalized cocoa with KOH; C#_NaH-CO_3=Cake-alkalized cocoa with $NaHCO_3$; # = M = mild alkalization conditions; # = S = strong alkalization conditions. Values with different lowercase letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoa powders with different alkalizing agents or type of alkalization (nib or cake) for the same percentage of alkali; Values with different capital letters in the same column indicate statistically significant differences ($p < 0.05$) between cocoas with different percentages of alkali for the same alkalizing agent or type of alkalization. Data were analyzed with ANOVA and LSD post hoc test.

4. Conclusions

This is one of the first works aiming to understand the effect of alkalization type (nib or cake), alkali (K_2CO3, KOH and $NaHCO_3$) and alkalization intensity (mild and strong) not only on cocoa powder quality parameters, but also on those of the corresponding doughs and cakes where they are included. The study evidences that KOH provides the darkest, most reddish and least saturated cocoa, while $NaHCO_3$ yields the lightest one. This general behavior is kept after the preparation of the dough and the cake. Comparing nib-and cake-alkalized powders with K_2CO3, it can be noted that color differences are not significant for most of the parameters in both, cocoa powders and corresponding cakes. The same happens to texture, but not for rheology, where the doughs prepared with the nib-alkalized powders in mild conditions exhibit the higher elastic modulus. Despite these differences, consumers' satisfaction with different cakes was very similar, although darker products were perceived as tastier. By considering these attributes, the best conditions for processing cocoa if used to prepare cakes should be alkalization in cake (processing is more economical and does not reduce consumer perceived sensory quality). Moreover, KOH and strong alkalizing conditions should be preferred to obtain a product as dark as possible and thus, to allow consumers to perceive a more intense cocoa flavor.

CRediT authorship contribution statement

Marta Puchol-Miquel: Methodology, Formal analysis, Data curation, Writing – original draft. **César Palomares:** Investigation, Resources, Supervision. **Isabel Fernández-Segovia:** Data curation. **José Manuel Barat:** Validation, Funding acquisition, Resources. **Édgar Perez-Esteve:** Conceptualization, Formal analysis, Data curation, Investigation, Writing – review & editing.

Declaration of competing interest

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

Acknowledgements

The authors wish to acknowledge the financial assistance provided by the Ministerio de Economía, Industria y Competitividad (Spanish Government) and the European Regional Development Fund (Project RTC- 2016-5241-2). We also thank Helen Warburton for editing the English style.

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