# **Chapter 6 A Technological Optimization to Design a Better Gluten-Free Cereal-Based Cake Premix**



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# **6.1 Introduction**

Broadly driven by consumers who voluntarily avoid or limit gluten in their diets, the demand for gluten-free (GF) products continues strong in the foodservice, as well as in retail sectors, and products labeled as GF are showing up more often during in-home meal occasions (Schierhorn [2018\)](#page-24-0). However, the formulation and the manufacture of baked goods without gluten results in considerable technological problems for both cereal technologists and bakers (Gallagher et al. [2004](#page-23-0); Matos et al. [2014\)](#page-24-1). The development of food products from other cereals to replace those containing gluten might lead: (1) to attractive aerated products for the section of the population who consumes GF products; and (2) to increase utilization of these cereals for human food, and sorghum is particularly appealing because it can be grown in areas not suitable for wheat (MacRitchie [2010](#page-24-2)).

Sorghum (*Sorghum bicolor* (L.) Moench), a tropical cereal belonging to the tribe of *Andropogoneae*, is the ffth most important cereal crop worldwide, with more than 57 million tons produced from approximately 41 million ha of land in 2017 (FAOSTAT [2019](#page-23-1)). It is yet considered of low value to humans and often used as animal feed (Stefoska-Needham et al. [2015\)](#page-24-3). For millions of people in parts of

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Africa and Asia, sorghum is a vital food crop, while it is an underutilized resource in most developed countries (Taylor et al. [2006\)](#page-24-4). Still, it has peaked commercial interests in developed economies on how to make sorghum-based food products due to a growing consumer movement dedicated to "healthy living" (Stefoska-Needham et al. [2015\)](#page-24-3). Nevertheless, unlike wheat proteins, sorghum proteins are not highly functional (Mesa-Stonestreet et al. [2010\)](#page-24-5) because they are encapsulated in protein bodies, and this fact probably turns into unavailable its participation in a viscoelastic dough fbril formation (Duodu et al. [2003\)](#page-23-2). Therefore, a modifcation of sorghum proteins could be one way to get around the technological properties challenge. In this context, it is mandatory to employ an economical, food-compatible, safe, and scaled up to commercial level method to modify the functional properties of sorghum proteins, to enlarge their applications in food formulations (Mesa-Stonestreet et al. [2010\)](#page-24-5).

As GF has become an expectation for consumers seeking clean label products (Schierhorn [2018\)](#page-24-0), thermoplastic extrusion can be a process used to satisfy this public. The reason is due to its absence of effuents (Guy [2001\)](#page-23-3), and to improve the protein functionality of sorghum (Mesa-Stonestreet et al. [2010\)](#page-24-5), because it is a nonwet cooking process. It is a method in which starch and/or protein and/or dietary fber are transformed into plastic polymers by adding little water, and they are cooked with a high degree of mechanical energy. The precooking of sorghum four promoted by thermoplastic extrusion, to produce pregelatinized starch, seems to be effective to reduce the rate of gas loss from the batters, which has an important role in the fnal cake quality (Taylor et al. [2006\)](#page-24-4). Limited research has been performed on the heat treatment of whole-grain sorghum four.

Specifcally for cakes, reach both oven spring and specifc volume similar to a cake with gluten in GF cakes is generally a technological challenge itself, as well as achieving a similar texture. Particle size profle and fber, protein, and starch contents and types, which impact on hydration and pasting properties, could have a great role in technological characteristics of cakes (Gómez et al. [2008;](#page-23-4) Wilderjans et al. [2008;](#page-25-0) Oh et al. [2014;](#page-24-6) Dhen et al. [2016](#page-23-5)).

There are many studies, summarized by Di Cairano et al. [\(2018](#page-23-6)), showing the importance of the combination of GF fours on the enhancement of dough and biscuits properties, such as technological, nutritional and sensorial characteristics. This relevance can be easily extrapolated to cakes since gluten plays a minor role in both biscuits and cakes. As designing an experiment typically involves selecting among several candidate designs, a very common choice is a mixture design, which is largely used whenever a multi-component system is concerned. In this type of experimental design methodology, the sum of the proportions of the mixture components is always 1, and the other components that are not part of the design must not change. The application of a mixture design can allow fnding the optimal composition to achieve a desirable response (Larrosa et al. [2013\)](#page-24-7), using mixture contour plots methodology. Rice offers numerous benefts to be a cereal to take part in a

mixture design, considering its natural bland taste, colorless and hypoallergenic properties (Arslan et al. [2019\)](#page-22-0).

Because of its ready-to-eat nature, availability in many types, and affordable cost, cakes are the most popular bakery items consumed nearly by all levels of society (Jeddou et al. [2017\)](#page-23-7). This fact could explain the reason why there are countless researches focused on the technological quality of GF cakes. On the other hand, studies based on cake premixes are scarce, despite their comparatively good shelf-life. Since consumers do not usually have easy access to GF fours (especially if it is made of an ancient whole-grain) suitable for bakery and confectionery products, it becomes important to offer a carefully industrial prepared cake premix. Therefore, costumers fnd the convenience of rapidly prepare a GF cake at home.

As the naturally GF flours, such as sorghum, millet, or buckwheat, are not mainstream items in GF foods, because commercial starches are preferably used in these products (Lee et al. [2019](#page-24-8)), researches into the development of products using GF whole-grains becomes especially important. GF products consumers are progressively demanding GF foods comparable to the long-established gluten ones (Matos et al. [2014\)](#page-24-1). This fact is stimulating much product innovation because GF products do not match with products containing gluten in terms of technological attributes (Arslan et al. [2019](#page-22-0)). Hence, the present study was focused on optimizing a cake premix formulation, based on GF cereals (sorghum—*in natura* and pregelatinized and rice) and using orange pomace four as a fber-rich coproduct, investigating the technological characteristics of GF batter high ratio type cakes, assessed by the mixture contour plots.

# **6.2 Material and Methods**

#### *6.2.1 Ingredients*

The ingredients used in the preparation of the cake premixes and of the cakes were: pregelatinized blend four (E) (prepared as described in Sect. [6.2.1.2](#page-3-0)), red sorghum whole-grain (Empório Figueira, São Paulo, Brazil), whey powder (Laticínios Porto Alegre, Ponte Nova, Brazil), vegetal shortening (FRS Alimentos, Barueri, Brazil), alkalized and natural cocoa powders (Pryme Foods, Sorocaba, Brazil), emulsifer (polyglycerol fatty acid esters, mono- and diglycerides of fatty acid and polysorbate 80; Corbion, Araucária, Brazil), sodium acid pyrophosphate and monocalcium phosphate (Diadema Agro-Industrial Ltda, Itapecerica da Serra, Brazil) and chocolate and orange aromas (Frutarom do Brasil Indústria e Comércio Ltda, Porto Feliz, Brazil). Polished rice, wheat four, corn starch, crystal sugar, salt, sodium bicarbonate, whole milk, egg and margarine were purchased in local markets in the city of Rio de Janeiro (Brazil).

#### **6.2.1.1 Preprocessing of Raw Material**

Red sorghum whole-grain four (S) and polished rice four (R) were produced through milling these cereals on the disc mill Laboratory Mill 3600 (Perten Instruments AB; Huddinge, Sweden), set to aperture 6.

The by-product of orange juicing (favedo, albedo, and seed), was ground on a multiprocessor Cadence Mix for You MPR853 (JCS Brasil Eletrodomésticos SA; Balneário Piçarras, Brazil), right after collecting at the food processing industry Bora Bora Comércio e Indústria Ltda (Rio de Janeiro, Brazil), dispersed on trays and dehydrated in a turbo-electric oven FTT 390G (Tedesco; Caxias do Sul, Brazil) at 60 ºC for 5 h. After dehydration, the by-product was milled in a disc mill Laboratory Mill 3600 (Perten Instruments AB; Huddinge, Sweden), set to aperture 6. This product will henceforth be called orange pomace four.

#### <span id="page-3-0"></span>**6.2.1.2 Thermoplastic Extrusion Conditions**

S (87.8%), orange pomace four (7.2%), whey powder (5%), and water (the necessary amount for end conditioning moisture of 16.2%) were mixed on a powder homogenizer Chopin (Tripette & Renaud; Villeneuve la Garenne, France) to prepare E. It was subsequently stored into sealed polyethylene bags, in refrigeration, for 17 h to equilibrate the water distribution. The extrusion was conducted using a single-screw extruder DO-CORDER Brabender (Brabender; Duisburg, Germany). The extrusion profle was: feed section speed: 20 rpm; shear rate: 4:1; screw speed: 160 rpm; feed section temperature: 50 °C; compression section temperature 90 °C; circular die diameter: 3 mm. The extrudate was dehydrated in a fan oven Macanuda Hauber DMS-G (Macanuda Hauber; Joinville, Brazil) at 60 °C for 4 h and subsequently milled on a disc mill Laboratory Mill 3600 (Perten Instruments AB; Huddinge, Sweden), set to aperture 1, and, afterward, on a roller mill Brabender Jr. (Brabender; Duisburg, Germany). The formulation of E and processing conditions of thermoplastic extrusion were established according to the result obtained for an optimization based on better pasting properties of pregelatinized blend four for cake production employment (unpublished results).

# *6.2.2 Flours Analyses and Measurements*

To obtain a standard four with gluten suitable for cake production, domestic wheat four of the market was mixed with corn starch in a 4:1 ratio. The goal of this mixture was to have the protein content approximately 7–8%, as recommended by Yamazaki and Kissell ([1978\)](#page-25-1). This four will henceforth be called reference flour (RF).

Red sorghum whole-grain flour (S), polished rice flour (R), pregelatinized blend flour (E) and reference flour (RF) were characterized by the following analyses:

### **6.2.2.1 Proximate Composition**

The proximate composition was determined according to the offcial methods of analysis of the Association of Offcial Analytical Chemists (AOAC [2005](#page-22-1)), in duplicate measurements: moisture content (Method 925.09), total nitrogen (Method 2001.11, a conversion factor of 5.75 was used to convert total nitrogen to protein content), lipid content (Method 945.38), ash content (Method 923.03), and total dietary fber (Method 985.29) (analysis without repetitions). Carbohydrates were determined by difference.

## **6.2.2.2 Particle Size Distribution**

The particle size distribution measurement was carried out according to the AACC method n° 66–20.01 (AACC [2010\)](#page-22-2) in a ROTAP sieve shaker RX-29-10 (W.S. Tyler, St. Albans, USA) in duplicate. Seven screen sieve sizes (Newark, USA) were selected (425, 355, 250, 212, 180, 106 and 75μm) and a pan, to obtain a normal distribution of particles from 100 g of sample sieved for 10 min.

## **6.2.2.3 Hydration Properties**

Water solubility index (WSI) and water absorption index (WAI) were determined according to the methodology described by Anderson et al. [\(1969](#page-22-3)), in quadruplicate. WSI indicates the amount of sample that is solubilized in water at 30 °C relative to the initial sample, after shaking and centrifuging. In comparison, WAI reports the amount of water absorbed at 30 °C related to the sample that did not solubilize under shaking and centrifugation.

#### **6.2.2.4 Pasting Properties**

Viscosity was determined, in an aqueous medium, under heating and stirring, using the equipment *Rapid Visco Analyser* (RVA) (*Newport Scientifc*; Warriewood, Australia), according to AACC method n° 76–21.01 [\(2010](#page-22-2)), with modifications. The flour was initially stirred during 10 s, at the initial temperature of 25  $\degree$ C and 960 rpm. The remainder of the procedure, which lasted 20 min, was conducted at 160 rpm. The initial temperature was held for 2 min and gradually raised, heating to 95 °C, at a constant rate of 14 °C min<sup>-1</sup>. Then the temperature was held constant for 3 min and cooled to 25 °C, at a constant rate of 14 °C min−<sup>1</sup> , which was held during the last 5 min of the analysis.

The readings from the pasting curve generated were: pasting temperature (PT), maximum viscosity at 25 °C (MV25), peak viscosity (PV), trough (TR), breakdown (BKD), fnal viscosity (FV), and setback (SB), obtained through the register realized by the software Thermocline (Newport Scientifc; Warriewood, Australia). The measurements were conducted in duplicate.

#### **6.2.2.5 Differential Scanning Calorimetry (DSC)**

Starch gelatinization was studied using a calorimeter (TA Instruments, Q200, USA). Indium (In) standards were used to calibrate the energy and temperature of the equipment, and nitrogen was used as the purge gas. Briefy, approximately 3 mg of four were weighed in hermetic aluminum pans with the aid of a precision scale (Mettler Toledo, Mx5, USA), followed by addition of 6μL of water using a microliter syringe, to maintain hydration. Before the thermal scan, the pans were then hermetically sealed and allowed to equilibrate for 24 h at room temperature. The pans were scanned from 5 to 110  $\degree$ C at a scanning rate of 10  $\degree$ C/min using an empty pan as a reference. The determination of the temperatures of onset, peak, and conclusion, as well as the enthalpy of gelatinization, was analyzed by the Universal V4.5A® software (TA Instruments, USA). The measurements were conducted in duplicate.

## <span id="page-5-0"></span>*6.2.3 Cake Premix Production*

Preliminary baking tests were made to adjust the cake premix formula. Reference cake premixes were prepared by using the reference four (RF). The cake premix formula, based on the four blend weight, was: 120% crystal sugar, 30% vegetal shortening, 10% alkalized cocoa powder, 5% natural cocoa powder, 2.2% sodium bicarbonate, 2% emulsifer, 1.1% sodium acid pyrophosphate, 1.1% monocalcium phosphate, 1% salt, 0.5% chocolate and 0.5% orange aromas. GF cereal-based cake premixes were carried out by replacing four with gluten with mixtures of S, R, and E according to simplex-centroid mixture design (SCMD), which gave 7 experimental assays (Table  $6.1$ ). All variables had the same range (between 0 and 1), and there were no constraints on the design space.

To produce the cake premix, sugar and shortening were mixed during 10 min at medium speed, using a cake batter whip in a planetary mixer (HMT, China). After that, all other ingredients were added and homogenized for 10 min under the same conditions.

# <span id="page-5-1"></span>*6.2.4 Cake Production*

Batter high ratio type cakes were made by adding, based on the cake premix weight, 33.3 % whole milk, 33.3 % egg, and 13.3 % margarine to the cake premix (as recommended by commercial cake premixes sold in Brazil), by following an all-in mixing procedure. The aim was to simulate a homemade preparation. They were homogenized in a planetary mixer (HMT, China), using a cake batter whip, for 10 min at medium speed. Subsequently, the batter was poured  $(45 \pm 1 \text{ g})$  for each paper mold which was supported by a stainless cup (7 and 4.5 cm radius at top and bottom, respectively, and 4 cm height). The cup trays containing batter were then baked in an electric oven (Suggar, China) at 170  $\degree$ C for 20 min. Finally, the cakes



<span id="page-6-0"></span>formity index



were removed from the cup and kept at room temperature  $(25 \pm 2 \degree C)$  for 1 h before analyses.

#### **6.2.4.1 Batter Specifc Gravity**

Specific gravity (SG) of cake batter at  $25 \pm 2$  °C was measured by dividing the weight of a certain volume of batter by the weight of an equal volume of water (Matos et al. [2014\)](#page-24-1).

#### **6.2.4.2 Technological Characteristics of the Cakes**

#### **Oven Spring**

The oven spring (OS) was determined from the difference in the central height of the batter and the central height of the baked cake. A pachymeter was used to obtain these measurements, made in six replicates.

#### **Specifc Volume**

The specifc volume (SV) of the cake was determined by volume/weight. The apparent volume (mL) was measured using the millet seed displacement method according to the AACC method n° 10-05.01 (AACC [2010\)](#page-22-2), and the weight (g) was determined with a semianalytical scale SB12001 (Mettler Toledo, Greifensee, Switzerland). SV was determined in six replicates.

#### **Volume, Symmetry and Uniformity Indices**

Volume, symmetry, and uniformity indices were measured according to the AACC approved method  $n^{\circ}$  10-91.01 (AACC [2010\)](#page-22-2), with template adaptations to the size of cakes (Fig.  $6.1$ ). The heights of the cakes were measured with a pachymeter at three points (B, C, and D) along with the cross-sectioned samples. Indices, which were determined in triplicate, were calculated by the Eqs.  $(6.1)$  $(6.1)$  $(6.1)$ – $(6.3)$  $(6.3)$  $(6.3)$ .

Volume index (VI) = 
$$
B + C + D
$$
 (6.1)

$$
Symmetry index (SI) = 2C - B - D \tag{6.2}
$$

$$
Uniformity index (UI) = B - D \tag{6.3}
$$

#### <span id="page-7-1"></span><span id="page-7-0"></span>**Cake Texture**

Crumb frmness was determined according to the AACC method n° 74-09.01 (AACC [2010](#page-22-2)), and an adaptation of this method was used to measure springiness, according to Sangnark and Noomhorm [\(2004](#page-24-9)). Texture testing was accomplished on a Texture Analyzer TA.XT Plus (Stable Micro Systems, Surrey, England) and

<span id="page-8-0"></span>

**Fig. 6.1** General illustration of standardized cake height measurement points for volume, symmetry, and uniformity indices evaluation for cupcakes. Adapted from AACC International [\(2010](#page-22-2))

ftted with a 30 kg load cell and a P/36 cylindrical aluminum sensor probe. The parameters established were: test option and mode = measurement of the compression force, hold until time; pre-test speed  $= 2.0$  mm/s; test speed  $= 1.7$  mm/s; posttest speed = 10 mm/s; distance =  $40\%$  and time =  $60$  s. In cake texture determinations, the crust was removed by the lowest height of the crust, obtaining a linear surface, and it was assessed the crumb of the sample remainder. Ten replicates were carried out for each cake assay.

#### **Crumb Structure Analysis**

To qualitatively evaluate the crumb of cakes, they were cut in half lengthwise. Images were acquired using a fatbed scanner (HP Scanjet G3110, Hewlett-Packard Development Co, Canada). A black box was used to cover the cakes and avoid light contamination.

# <span id="page-8-1"></span>*6.2.5 Quality During Shelf Life*

Moisture (M) and water activity  $(a<sub>w</sub>)$  of the cake premixes were measured on the day they were baked (Day 0), while the specifc volume (SV) of the cake was calculated on the Day 0 (the results of the Sect. [6.2.6](#page-9-0)) and after 180 days (Day 180) of storage of the cake premixes, into sealed polyethylene bags, at  $25 \pm 2$  °C and dark place.

M was determined using Method n° 925.09 of the Association of Official Analytical Chemists (AOAC [2005\)](#page-22-1).  $a_w$  was measured in AquaLab LITE (Decagon; Pullman, USA). Both measurements were conducted in duplicate.

# <span id="page-9-0"></span>*6.2.6 Statistical Analyses*

Differences between the mean values for the analyses made on fours were assessed by the analysis of variance (ANOVA;  $p < 0.05$ ) and followed by the Tukey test  $(p < 0.05)$ , only when significant differences were observed in ANOVA, using Statistica software 13.0 for Windows (StatSoft Inc.; Tulsa, USA).

The results from the assessment of the technological characteristics of the cakes, as well as the responses of shelf-life study of the cake premixes, were analyzed by the mixture contour plots generated from the ftted regression models obtained by Response Surface Methodology (ANOVA; *p* < 0.05), performed by the same statistical program.

#### *6.2.7 Validation of the Mathematical Models*

To validate the ftted regression models, one formulation, which was different from the formulations of the SCMD, was chosen within the ranges studied. It consisted of  $S = 0.03$ ,  $R = 0.39$  and  $E = 0.58$ . Cake premixes and cakes were prepared as described in Sects. [6.2.3](#page-5-0) and [6.2.4,](#page-5-1) and batter specifc gravity and technological parameters of the cakes were evaluated following the procedures described in Sects. [6.2.5](#page-8-1) and [6.2.6.](#page-9-0) Subsequently, the experimental values were compared with the predicted values by the single sample Student's t-test ( $p = 0.05$ ). The analysis of the relative deviation, obtained using Eq. [\(6.4\)](#page-9-1), was also performed.

Relative deviation

<span id="page-9-1"></span>
$$
= \left| \left( \frac{\text{experimental value} - \text{value predicted by the coded model}}{\text{experimental value}} \right) \right| \times 100 \tag{6.4}
$$

# *6.2.8 Technological Optimization*

Numerical optimization is suggested, also using the abovementioned software. Considering the validated models of technological parameters of the cakes, it was employed the desirability function, according to Derringer and Suich ([1980\)](#page-23-8), to obtain the best cake in terms of the technological attributes, maximizing the values of OS, SV, VI, SI and springiness and minimizing the values of frmness and UI. The target was maximizing the overall desirability value of the sample.

# **6.3 Results and Discussion**

# *6.3.1 Batter Specifc Gravity*

The SG (Table [6.1](#page-6-0)) is a very important physical property affecting the product quality since it is a measurement of the total air-holding capacity, which is initially incorporated into the batter during the mixing time (Turabi et al. [2008;](#page-24-10) Zhou et al. [2011](#page-25-2)). It is important to get proper air incorporation in the batter during mixing because the air bubbles serve as nuclei for other leavening agents (Stauffer [1990](#page-24-11)). Regrettably, the SG gives little information about bubble size or dispersion (Zhou et al. [2011](#page-25-2)).

The SG has a direct infuence over the fnal cake volume (Kim and Walker [1992\)](#page-24-12). Low SG is desired in cake batter because it indicates that more air was incorporated into the batter (Turabi et al. [2008](#page-24-10)), and, therefore, it has been related to a higher cake volume (Gómez et al. [2007\)](#page-23-9). Similarly, the increase in the number of air bubbles in the batter system seems to translate into a tender baked product (Marston et al. [2016\)](#page-24-13), as the change in the density of the batter affects its crumb attributes (Yildiz and Dogan [2014\)](#page-25-3).

For the SG values it was verifed, through the mathematical model (Table [6.2\)](#page-10-0), that the increase in S, R and E caused increments in this batter parameter. In contrast, the interaction between R and E displayed a decrease in the SG. It is noticed

Parameters	Fitted regression model
Batter specific gravity	$SG = 0.71 S + 0.83 R + 0.96 E - 0.56 R E$
(SG)	$[r^2 = 0.7218; p = 0.0419; p_{\text{(lack of fit)}} = 0.0647]$
Oven spring $OS(%)$	$OS = 56.94 S + 88.27 R + 53.11 E$
	$[r^2 = 0.7203; p_{(model)} = 0.0116; p_{(lack of fit)} = 0.3543]$
Specific volume (SV)	$SV = 2.24 S + 2.35 R + 1.31 E + 1.32 S R + 0.54 R E$
(mL/g)	$[r^2 = 0.9917; p = 2.1825 \times 10^{-5}; p_{\text{dark of fit}} = 0.2059]$
Volume index (VI) (mm)	$VI = 105.01 S + 120.53 R + 79.81 E + 29.14 S E + 51.33 S R E$
	$[r^2 = 0.9995; p = 2.2582 \times 10^{-8}; p_{\text{dark of fit}} = 0.3181]$
Symmetry index (SI)	$SI = -0.61 S + 6.29 R + 3.19 E - 9.95 S R + 7.85 S E - 5.15 R E$
(mm)	$[r^2 = 0.9930; p = 0.0002; p_{\text{(lack of fit)}} = 0.7198]$
Uniformity index (UI)	$III = 0.80 S + 1.44 R + 0.74 E - 2.67 S R$
(mm)	$[r^2 = 0.9327; p = 0.0007; p_{\text{(lack of fit)}} = 0.5963]$
Firmness $(N)$	Firmness = $15.83 S + 8.82 R + 24.00 E$
	$[r^2 = 0.9364; p = 6.5000 \times 10^{-5}; p_{\text{(lack of fit)}} = 0.3226]$
Springiness $(\% )$	Springiness = $44.85 S + 48.68 R + 41.85 E + 3.88 S R E - 1.86 S$
	$E + 2.72 R E$
	$[r^2 = 0.9974; p = 2.9266 \times 10^{-5}; p_{\text{(lack of fit)}} = 0.3654]$

<span id="page-10-0"></span>**Table 6.2** Fitted regression models for the batter specific gravity and the technological parameters of the cakes

*S* red sorghum whole-grain flour, *R* polished rice flour, *E* pregelatinized blend flour,  $r^2$  coefficient of determination

<span id="page-11-0"></span>

**Fig. 6.2** Mixture contour plot for (**a**) specifc gravity (SG), (**b**) oven spring (OS), (**c**) specifc volume (SV), (**d**) volume index (VI), (**e**) symmetry index (SI), (**f**) uniformity index, (**g**) frmness, and (**h**) springiness. *S* red sorghum whole-grain four, *R* polished rice four, *E* pregelatinized blend four

that the SG was affected more strongly by E variation (Fig. [6.2a\)](#page-11-0). This fact is justified because E is a pregelatinized flour (Fig.  $6.3$ ) and contributed as a viscosity increasing agent (thickener-like) at the time of mixing, while S and R contributed to increasing viscosity during baking. Since S is the four that presented the highest amount of lipids in its composition (Table [6.3](#page-13-0)), it is plausible that S had shown the highest air-holding capacity during mixing, represented by the lowest coefficient value in the mathematical model (Table [6.2](#page-10-0)). Lipids contribute to the interface stabilization that favors gas entrapment in the batter matrix (Gularte et al. [2012\)](#page-23-10).

# *6.3.2 Technological Characteristics of the Cakes*

### **6.3.2.1 Oven Spring**

Starch and protein in combination mainly with sugar and fat are fundamental to the structure and other technological properties of bakery products (Hesso et al. [2015\)](#page-23-11). During baking, starch gelatinization, protein denaturation (especially egg protein

<span id="page-12-0"></span>

**Fig. 6.3** Pasting properties profles of the fours employed in the cake premixes

coagulation) and air bubbles expansion occur, and cake structure sets as a result of the harmonization of these processes (Yang and Foegeding [2010](#page-25-4); Hesso et al. [2015\)](#page-23-11). In the case of chemically leavened cakes, leavening is understood as being due to carbon dioxide  $(CO<sub>2</sub>)$  produced by chemical reactions, but the expansion of air incorporated into the batter during high-temperature baking also leads the cake to enlarge and form an open interior texture to some extent. Nevertheless, if  $CO<sub>2</sub>$  is released too early or too late, the fnal product has a small height and volume (Stauffer [1990\)](#page-24-11). A more viscous batter with lower SG prevents large air,  $CO<sub>2</sub>$  or steam bubbles from coalescing and leaving the batter from the surface (Marston et al. [2016\)](#page-24-13). The baking step underlines the physical and chemical changes in the product components prompting a stabilized crumb structure. During the cooling process, the joined effect of starch gelation and protein coagulation is responsible for the framework setting of the end product (Hesso et al. [2015\)](#page-23-11).

The amount of time available for batter expansion before the structure sets is one important factor for the magnitude of OS (Stauffer [1990](#page-24-11)). Therefore, as crumb is partially created during baking (Conforti [2014\)](#page-23-12), two important parameters related to the OS are the range of gelatinization temperature and the pasting properties of the fours (Table [6.3](#page-13-0) and Fig. [6.3\)](#page-12-0). The starch gelatinization at low temperatures would prevent the correct expansion of batters (Gómez et al. [2008\)](#page-23-4). As higher is the gelatinization temperature, the longer is the development of the crumb (Cauvain and



<span id="page-13-0"></span>**Table 6.3** Proximate composition, hydration properties, pasting properties and thermal characteristics of the fours used to prepare the cake premixes  $\frac{1}{2}$ l, ं ł,  $\cdot$  $\ddot{\cdot}$  $\ddot{\phantom{a}}$ l, l,  $\ddot{\cdot}$ −. á  $\ddot{\cdot}$ Toble

Young  $2000$ ), which is dependent on the number and size of air,  $CO<sub>2</sub>$  or steam bubbles (Conforti [2014](#page-23-12)). Hence, it is important to have a batter with a suitable viscosity to retain both the air incorporated during mixing and the expanding air nuclei produced by the baking powder during baking, likewise to ensure the uniform dispersion of the ingredients for optimum performance (Wilderjans et al. [2008](#page-25-0)). In the mathematical model of the OS (Table [6.2\)](#page-10-0), it is showed that increments in S, R, and E increased OS values. Nevertheless, it is observed that R variation affected more intensely the OS than S and E variations (Fig. [6.2b\)](#page-11-0). Meantime, thermal analysis data show that the onset and the peak temperatures of R gelatinization were lower than the S and E ones (Table [6.3\)](#page-13-0). However, the values of PT, PV, TR and BKD of R, are higher than the S and E ones (Table [6.3](#page-13-0) and Fig. [6.3](#page-12-0)), suggesting that, in this study, the viscosity provided by the fours is more important for crumb development than their gelatinization temperatures. Among the three vertex points of SCMD (Fig. [6.2b\)](#page-11-0), only R presented the pasting properties profle suitable to be used alone in a GF cake formulation, to achieve the best OS. S and E must be mixed with another source of starch to reach better values of OS, hence justifying the experimental design adopted in this study. Analyzing the pasting properties profles of the fours employed in the cake premixes (Fig. [6.3\)](#page-12-0), it can be inferred that the choice of R as another source of starch of SCMD was successful. The aim was to achieve at a GF mixed four that more closely approached the pasting parameters of the four with gluten. As R has fewer coarse particles  $(> 250 \mu m)$  (Fig. [6.4\)](#page-14-0), this may also

<span id="page-14-0"></span>

**Fig. 6.4** Particle size distribution of the fours used to prepare the cake premixes. Columns with different script letters, within the same sieve opening, are signifcantly different according to Tukey's test  $(p < 0.05)$ 

have contributed to the higher increase in the OS, because the smaller particles are lighter and therefore easier to be carried by air,  $CO<sub>2</sub>$  or steam bubbles. Choi and Baik [\(2013](#page-23-14)) stated that small four particles with a larger total surface area could trap more air bubbles during batter mixing than could coarse particles. This effect was not observed in our results since R did not contribute to decreasing the SG. Nevertheless, R may have contributed to the better bubble distribution, resulting in increased OS. Further investigations would be necessary to corroborate both effects described for the relationship among the four particle size with the SG and OS.

# *6.3.3 Specifc Volume*

Similar to the OS, the mathematical model of the SV (Table [6.2\)](#page-10-0) shows that increases in S, R, E and in the interactions between S and R and R and E caused an increment in the SV. It is noticed that the SV was affected more strongly by R and S variations (Fig. [6.2c\)](#page-11-0). The volume of cake is partly a consequence of the air incorporated in the batter, which agrees with the described results on the SG. Gómez et al. [\(2008](#page-23-4)) explained that cake ingredients with higher protein content resulted in a SV value decrease. Among GF flours used, R presented the lowest content of protein and being not signifcantly different from the four with gluten (Table [6.3](#page-13-0)). This result may contribute to diminish the collapse of the structure during the fnal stages of the baking process or cooling in the cakes with higher amounts of R. Moreover, dietary fber usually contributes to decreasing the cake volume (Gómez et al. [2010;](#page-23-15) Itthivadhanapong and Sangnark [2016\)](#page-23-16). This reduction in the volume might be due to the collapse caused by the fibers in the air,  $CO<sub>2</sub>$  or steam bubbles during baking (Walker et al. [2014\)](#page-25-5). On the other hand, the increase in the dietary fber in a cake formulation might increment SV because of its great properties of water absorption and swelling power, which cause high water retention and subsequent increase in SV (Oh et al. [2014](#page-24-6)). It is also possible that dietary fbers may increase this process temperature by reducing the available water for this process, as well as interacting with starch molecules and, thus, promote cake expansion (Majzoobi et al. [2016\)](#page-24-14). Dietary fbers can also interact with proteins of GF fours and strengthen the overall structure and, therefore, preserve the volume of GF cakes. Further investigations are required to prove these changes. As GF fours used in this study have different dietary fber content and WAI value (Table [6.3\)](#page-13-0), all of them contribute to the enhancement of the SV. Generally, low SV indicates a dense and less attractive crumb, although high SV does not always mean a desirable cake (Brooker [1993](#page-22-4)). A cake with high SV may not be associated with a desirable texture, since variations in bubble size and the presence of large bubbles result in an unstable mix, producing cakes of coarse and uneven texture (Conforti [2014\)](#page-23-12). If the bubble does not reach it to the surface, it may instead form a tunnel in the cake (Stauffer [1990](#page-24-11)), which can generate a cake with high SV, but too porous. For the cake, greater contact and interaction between starch and water resulted in higher volumes (Wilderjans et al. [2008\)](#page-25-0). Due to this, the interaction

between a flour (R), which presented the lowest values for dietary fiber content and WAI, with flours  $(S \text{ and } E)$  that possess higher values of these parameters (Table  $6.3$ ) promoted an increase in the SV. It is as if the fours act synergistically to increase the SV, that is, as if there were a compensatory effect of the characteristics abovementioned among R, S, and E. Another feature of the fours that may have contributed to enlarge SV was their particle size distributions because the larger quantities of the flours were in the finer particle size fractions ( $\leq 180 \mu m$ ) (Fig. [6.1](#page-8-0)). Changes in volume may be related to differences in air retention and expansion during the baking process (Segundo et al. [2017](#page-24-15)), in which the size of particles could impact. This result is in agreement with a study of de la Hera et al. [\(2013\)](#page-23-17), where it was shown that the fnest rice fours produced higher volume GF cakes. It is known that the reduction of four particle size brings about signifcant improvements in cake quality, e.g., volume and softness, primarily when the four is used in high ratio cake recipes (Cauvain [2009\)](#page-22-5). Remarkably, most of the cake formulations reached SV values higher than 2.0 mL/g, approaching the reference cake closely, although some assays presented SV values lower than 2.0 mL/g (Table  $6.1$ ). This result emphasizes the importance of SCMD in the technological study of GF cereal-base cake formulation.

# *6.3.4 Volume, Symmetry and Uniformity Indices*

As expected, the VI followed a similar tendency as the SV, because the VI is an indicator of cake volume as well. So, the same parameters that infuenced the SV infuenced the VI too. VI indicates the amount of air entrapped in cake through the cake crumb (Rahmati and Tehrani [2014\)](#page-24-16). The mathematical model of the VI (Table [6.2](#page-10-0)) shows that increases in S, R, and E caused an increment in the VI. The difference between the SV and VI models was on the interaction factors: they were between S and E and S, R and E, on the VI. This distinction may be related to the different approaches used to measure these responses. Nonetheless, it can be seen in Fig. [6.2](#page-11-0) (c and d) that both cake volume measurement responses have similar profles, that is, they are similarly infuenced by the characteristics of R, S, and E discussed above.

The SI shows height differences between central and lateral areas of a cake (De La Hera et al. [2013\)](#page-23-17), expressing how flat the surface of a cake is. If the sum of the heights B and D (laterals) were close to twice the height C (central), then we have a SI value near to zero, indicating a fatter surface. The SI values greater than zero indicate a peak neighboring the center of the surface contour of the cake, while SI values less than zero indicate a collapse on the center of the cake surface contour. This collapse would seem to suggest that the cake structure is not stable enough to support its weight after baking (De La Hera et al. [2013\)](#page-23-17). Consequently, SI is related to gas retention in the fnal baking phase (Gómez et al. [2008](#page-23-4)). As it is possible to observe in Table [6.1,](#page-6-0) all cakes presented SI greater than zero, revealing that the crumb structure of none of them collapsed. It is desirable a the SI greater than zero in batter type cakes, in which they present the "grandma's classic cake format", that is, a slight peak in the center. In the mathematical model of the SI (Table [6.2](#page-10-0)), it is verifed that increases in R and E caused an increment in SI. In contrast S presented a negative interference in this response. The positive interference of R was greater than that of E (Fig. [6.2e\)](#page-11-0). De la Hera et al. ([2013\)](#page-23-17) showed that the fnest rice fours produced higher SI in GF cakes. More than 50% of S presented coarse particles  $(>=250 \mu m)$  (Fig. [6.4](#page-14-0)) and this may also have contributed to its negative influence in the SI. The larger particles of S may not have been uniformly distributed in the batter during the baking process, thus hindering both the retention of the formed gas and the water vaporization from the mass, decreasing the SI values. Generally, cakes with higher volumes exhibit higher central loaf height (Rahmati and Tehrani [2014\)](#page-24-16), suggesting a causal relationship between the SV and SI. However, this tendency was not observed in this study (Fig. [6.2c, e](#page-11-0)).

The UI evaluates the distribution of heat inside the oven during the baking process, expressing if the sides of the cakes grew equally. So, the closer the UI value is to zero, the more uniform the contour of the cake. All of the cakes assessed in this study presented satisfactory uniformity (Table [6.1\)](#page-6-0), that is, close to zero. In the mathematical model of the UI (Table  $6.2$ ), it is verified that increases in S, R, and E displayed increment in the UI. In contrast, the interaction between S and R displayed a decrease in the UI. Hence, it is possible to notice that the most uniform cakes were those that had higher values of S and R together (Fig. [6.2f](#page-11-0)).

# **6.4 Cake Texture**

During a product development process, texture analysis is one of the most helpful analytical methods, as it is suitable to quantify the effects of four blends on the physical properties of the crumb of the bakery products (Jeddou et al. [2017](#page-23-7)). The texture can be affected by the cake structure (Pizarro et al. [2013](#page-24-17)), with ingredients' infuence the ingredients on the size and distribution of the air cells within the product structure, which consists of air cells distributed throughout its matrix (Sozer et al. [2011\)](#page-24-18). Its tender texture characterizes a desirable cake.

The firmness (Table [6.1](#page-6-0)) test simulates the gentle squeezing by the hand that consumers apply to bakery products and many other food items (Bourne [1990](#page-22-6)). The mathematical model of the frmness (Table [6.2](#page-10-0)) shows that increases in S, R and E caused an increment in frmness. It is noticed that the frmness was affected more strongly by E variation and followed by the S one (Fig. [6.2g](#page-11-0)). The infuence of fours on the frmness was inversely proportional to the OS, SV and VI (Fig. [6.2b, c,](#page-11-0)  [d, g\)](#page-11-0), a theoretically expected result, since as higher are the OS, SV and SI, the lower the frmness presented by the cake. This result indicates that the improvement in the softness was due both to an increase in height and cake volume. Springiness (Table [6.1\)](#page-6-0) measures the elastic recovery of a sample and it was determined as a ratio of constant force during time holding to peak force before the holding time (Sangnark and Noomhorm [2004](#page-24-9)). According to this defnition, it is possible to infer that a good cake is one that presents higher springiness values. The mathematical model of springiness (Table [6.2\)](#page-10-0) shows that increases in S, R, and E and, additionally, in the interactions between S and R and S and E, displayed increment in springiness. It is also showed a negative interference of the interaction between R and E, as it was expected, since the infuence of fours on springiness was directly proportional to OS, SV and VI (Fig, 6.2b, c, d, h), while it was inversely proportional to firmness (Fig.  $6.2g$ , h). It is remarkable that: (1) R leaves the cake less firm and more elastic, and that: (2) among the three vertex points of SCMD (Table [6.3](#page-13-0)), R was the only four that most approached the frmness of the reference cake. These observations showed the importance of R in this study once again.

# *6.4.1 Crumb Structure Analysis*

The contour, symmetry, and shape of samples can be seen in Fig. [6.5](#page-18-0). The quality perceived in a GF cake is considerably related to the appearance of the crumb (Gambuś et al. [2009\)](#page-23-18), as a good cake should show a multitude of evenly distributed minute cells without any large holes (Bennion and Bamford [1997](#page-22-7)). All the cakes in this study presented good OS, SV and UI, if they are compared with the reference cake, except for the assay 3 in the OS and SV perceived on the images. This

<span id="page-18-0"></span>

**Fig. 6.5** Images of sliced cakes. *Ref* reference. See Table [6.1](#page-6-0) for more information about the samples' descriptions

technological problem occurred because the formulation of the cake premix and the ingredients used to prepare the cake itself, i.e., milk, eggs and margarine, were based on industrial formulations which are commercially available already in Brazil market. The amount of liquids added to the premix is practically standardized on the ingredient list of the commercial premixes, and it was chosen to maintain these quantities in this study. Hence, it can be visually concluded that liquids were lacking so that the assay 3 would obtain better technological parameters, considering it is a batter high ratio type cake. Visually, the assay 3 resembles a brownie cake.

# *6.4.2 Quality During Shelf Life*

In a shelf life study of cake premixes, three important parameters should be considered: the M and  $a_w$  of the cake premixes themselves and the SV of the cakes. The M and  $a_w$  are crucial ingredient characteristics because they will dictate the spoilage speed of a food product. As lower these both parameters are, the lower the pace of microbial activities and chemical reactions in the food matrix. Moreover, the rate of reaction of leavening agents is governed by their rate of dissolution (Stauffer [1990\)](#page-24-11). In this way, the contact with the cake premix moisture already initiates the chemical reaction processes by which the chemical leavening act on bakery products. Cakes prepared from premixes present fuctuation in their quality by the day of their preparation and the day of the premix production. The main reason is that time contributes to decreasing the quality of the fnal product. Especially for cake premixes, it is important to sustain the capacity to form a cake with the SV as higher as possible, throughout its storage period.

The M varied from 3.16% to 5.16% and *a*w ranged from 0.46 and 0.62 (Table [6.4\)](#page-19-0). Mathematical models for the M,  $a_w$  and SV-Day180 as a function of S, R, and E

Assays	S	R	E	RF	$M^a(\%)$	$a_w^a$	SV (Day $180$ <sup>b</sup> (mL/g)
	1	$\Omega$	$\Omega$		$5.1629 \pm 0.1039$	$0.623 \pm 0.001$	$1.83 \pm 0.05$
2	$\Omega$		$\Omega$		$4.4858 \pm 0.0592$	$0.557 \pm 0.003$	$2.06 \pm 0.08$
3	$\Omega$	$\Omega$	1		$3.1585 \pm 0.0379$	$0.463 \pm 0.001$	$1.31 \pm 0.10$
$\overline{4}$	1/2	1/2	$\Omega$		$4.7367 \pm 0.1221$	$0.581 \pm 0.001$	$1.88 \pm 0.08$
.5	1/2	$\Omega$	1/2		$3.9941 \pm 0.1134$	$0.530 \pm 0.001$	$1.71 \pm 0.05$
6	$\Omega$	1/2	1/2		$3.8175 \pm 0.0202$	$0.525 \pm 0.001$	$1.94 \pm 0.09$
7	1/3	1/3	1/3		$4.2278 \pm 0.0769$	$0.540 \pm 0.002$	$1.95 \pm 0.11$
8	1/3	1/3	1/3		$4.3096 \pm 0.0232$	$0.549 \pm 0.001$	$1.93 \pm 0.08$
9	1/3	1/3	1/3		$4.3136 \pm 0.0267$	$0.541 \pm 0.001$	$1.96 \pm 0.06$
10	1/3	1/3	1/3		$4.3890 \pm 0.0775$	$0.541 \pm 0.001$	$1.94 \pm 0.06$
Reference					$2.0380 \pm 0.1132$	$0.561 \pm 0.001$	$2.52 \pm 0.05$

<span id="page-19-0"></span>**Table 6.4** Results from the assessment of the shelf-life parameters of the SCMD and reference cake premixes

 $^{\circ}$ Mean  $\pm$  standard deviation, n = 2;  $^{\circ}$ mean  $\pm$  standard deviation; n = 6; *S* red sorghum whole-grain four, *R* polished rice four, *E* pregelatinized blend four, *RF* reference four (gluten-containing), *M* moisture,  $a_w$  water activity, *SV* specific volume

Parameters	Fitted regression model
Moisture $(M)$ $(\%)$	$M = 5.16 S + 4.48 R + 3.16 E - 0.35 S R - 0.66 S E + 4.17 S R E$
	$[r^2 = 0.9950; p = 0.0001; p_{\text{dark of fit}} = 0.9583]$
Water activity $(aw)$	$a_w = 0.62$ S + 0.56 R + 0.46 E – 0.04 S R – 0.05 S E + 0.06 R E
	$[r^2 = 0.9951; p = 0.0001; p_{\text{dark of fit}} = 1.0000]$
Specific volume	$SV$ -Day $180 = 1.83 S + 2.06 R + 1.31 E - 0.26 S R + 0.56 S E + 1.02$
Day 180 (SV-Day180)	$R E + 1.76 S R E$
(mL/g)	$[r^2 = 0.9988; p = 0.0002; p_{\text{(lack of fit)}} = -]$

<span id="page-20-0"></span>Table 6.5 Fitted regression models for the shelf-life parameters of the cake premixes

*S* red sorghum whole-grain flour, *R* polished rice flour, *E* pregelatinized blend flour,  $r^2$  coefficient of determination

<span id="page-20-1"></span>

**Fig. 6.6** Mixture contour plot for (**a**) moisture (M), (**b**) water activity  $(a_w)$ , and (**c**) specific volume of the gluten-free cakes after 180 days of storage (SV-Day180) of the cake premixes. *S* red sorghum whole-grain four, *R* polished rice four, *E* pregelatinized blend four

were found and showed interaction effects (Table [6.5\)](#page-20-0). In the M and *a*w, the related impacts to E were the lowest. Hence, an increment in E is the least contributing to increase these responses (Fig.  $6.6a$ , b), which is desirable to heighten shelf-life related to microbiological, enzymatic, or chemical activities.

When comparing the SV in Day 0 (Fig. [6.2c](#page-11-0)) with the SV after 180 days of cake premixes storage (Fig.  $6.6c$ ), through mixture contour plots it is concluded that: (1) in Day 0, an increment in any range of E entails a decrease of SV; (2) in Day 180, an increment of until 50% of E contributes to maintaining SV in the highest values region. Thus, to retain food safety and technological aspects of these cake premixes, E was shown to be a possible option. Complementarily, Karaoğlu et al. ([2001\)](#page-23-19) found that pregelatinized starch extends shelf life to the cakes by delaying the staling process.

### *6.4.3 Validation of the Mathematical Models*

It can be seen that the experimental values for the SG, OS, SV, VI, SI, UI, frmness and springiness and these values predicted by the models did not present a signifcant difference in the single sample Student's t-test  $(p > 0.05)$  (Table [6.6](#page-21-0)). Therefore, we may consider as validated the models for the SG, OS, SV, VI, SI, UI, frmness and springiness.

	Experimental values	Values predicted by the fitted regression models	Relative deviation
Batter specific gravity (SG)	$0.75 \pm 0.03^{\circ}$	0.73a	$2.33\%$
Oven spring $OS(%)$	$64.29 \pm 6.42^{\circ}$	66.94 <sup>a</sup>	4.12%
Specific volume (SV) (mL/g)	$1.91 \pm 0.08^{\text{a}}$	1.88 <sup>a</sup>	$1.51\%$
Volume index (VI) (mm)	$97.9 \pm 0.8^{\text{a}}$	97.3 <sup>a</sup>	0.63%
Symmetry index (SI) (mm)	$2.8 \pm 0.4^{\circ}$	3.1 <sup>a</sup>	10.40%
Uniformity index (UI) (mm)	$0.6 \pm 0.7^{\circ}$	1.0 <sup>a</sup>	73.57%
Firmness $(N)$	$17.62 \pm 0.79$ <sup>a</sup>	$17.83$ <sup>a</sup>	1.22%
Springiness $(\%)$	$45.41 \pm 0.31$ <sup>a</sup>	$45.25$ <sup>a</sup>	$0.35\%$

<span id="page-21-0"></span>**Table 6.6** Experimental values and the values predicted by the ftted regression models for the batter specifc gravity and the technological parameters of the cakes

a Means followed by the same letter on a row are statistically equal at 5% signifcance according to single sample Student's t-test

# *6.4.4 Technological Optimization*

At the established conditions for the optimization, the optimal four formulation for the cake premixes was:  $S = 20\%$  and  $R = 80\%$ , with a desirability value of 0.516 (Fig. [6.7\)](#page-22-8). Despite not having any E, this optimized formulation is one way to insert sorghum in industrialized foodstuffs, considering the growing demand for GF, more sustainable, convenient and high-quality foods.

# **6.5 Conclusions**

R was shown to be an important cereal in this study, as it improved many of the technological properties of cake (OS, SV, VI, SI, UI, frmness, and springiness) discussed here. Therefore, if the main objective is the introduction of an unconventional cereal, such as sorghum, in industrialized food products, the use of R is suggested to increase the technological quality of GF cereal-based cakes. Regardless of this fact, it is important to highlight that the use of a higher amount of liquid ingredients in the cake preparation could improve the technological characteristics of the products in which there were more S and E.

One optimized technological formulation for GF cake premixes have been suggested, in which the optimal flour formulation for the cake premixes was  $S = 20\%$ and  $R = 80\%$ . Although S and E have presented worse technological yields than R, they have great potential in cake production. It is essential to carry future studies that increase the amount of liquids in the formulations, for example, mainly because it was proved that E extends the shelf-life of the cake premix. These technological

<span id="page-22-8"></span>

**Fig. 6.7** Profle of predicted values and desirability for the technological optimization

evidences could also act as a catalyst for the uptake and demand for sorghum by the food industry and consumers.

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