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Optimization of processing parameters of a ball mill refiner for chocolate

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Abstract

The aim of this work was to optimize the ball mill based refining process of chocolate, in terms of refining time and energy consumption. Experiments were planned following a central composite design (CCD), considering refining time (*rt*) and agitator shaft speed (*as*) as factors. The experimental variables measured were chosen from the main characteristics that describe unmoulded chocolate. A complete second-order model was fitted to the experimental data. The most significant coefficients were that of energy consumption, iron content and particle size. Optimization consists in a bound minimization of refining time using the desirability function. Before experiments, working conditions were 70 rpm for *as* and 55 min for *rt*. The optimum conditions calculated by optimization were as follows: 58 rpm for *as* and 38.5 min for *rt*. The new working conditions identified for the ball mill considered enabled to rise output from 109 kg/h to 156 kg/h, with a 43% increase in productivity. A control experiment carried out in the optimized conditions to corroborate the results obtained, confirmed calculated expectations of response variables.

Keywords: Chocolate; Ball mill refiner; Experimental design; Optimization; Desirability function

1. Introduction

The current European legislation (Dir. 2000/36/CE; UE, 2000) designates chocolate as the product obtained from cocoa products and sugars, which contains not less than 35% total dry cocoa solids, including not less than 18% cocoa butter and not less than 14% dry non-fat cocoa solids.

The most traditional methods of chocolate-making are based on the mixing of ingredients, grinding by roll refiners (refining phase), conching, and tempering. Above all, conching is carried out in order to remove moisture and undesirable flavours while developing the pleasant ones. In addition, since the previous grinding process will have created many new surfaces not yet covered with fat, the conching phase coats these new surfaces and improves the flow properties (Beckett, 1999). The tempering process is a technique of controlled crystallization that is necessary to induce the most stable solid form of cocoa butter (which is a polymorphic fat) in the finished product (Talbot, 1999). Chocolate refining depends on product type (milk, dark or compound), on process (crumb vs. milk powder) and on ingredients (granulated or powder sugar). Grinding operations may be evaluated on the basis of their costs (capital, maintenance and energy) and the characteristics with which they provide the product (particle size distribution, particle shape and minimization of contamination). The choice of equipment for size reduction depends on many factors, including feed and final particle size, and type of material being processed. Chocolate refining is most often carried out using a five-roll refiner. Four grinding rolls are aligned vertically, while the feed roll is placed at an angle to the lowest stack roll. The feed rate determines the throughput and the final fineness of chocolate,

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Nomenclature

A	current measured by the ball mill amperometer (A)
as	agitator shaft speed (rpm)
CCD	central composite design
$\cos\varphi$	constant of the electric motor (0.85)
D	overall desirability function
d_{90}	particle size (μm)
d_i	individual desirability function
E	electricity consumption (kW h)
Fe	iron content (mg/kg)
L	lower limit in the desirability function
r	weight assigned to each individual desirability function
rt	refining time (min) considered as factor
RT	refining time (min) considered as response variable
T	target value in the desirability function
t_0	time interval (min) in electricity consumption calculation
U	upper limit in the desirability function
V	voltage measured by the ball mill voltmeter (V)
x_1	value of refining time (rt) in the polynomial model
x_2	value of agitator shaft speed (as) in the polynomial model
y	experimental variable value in the polynomial model
β_0	constant value in the polynomial model
β_1, β_2	linear coefficients in the polynomial model
β_{12}	interaction coefficient in the polynomial model
β_{11}, β_{22}	quadratic coefficients in the polynomial model
ε	random error in the polynomial model
$\dot{\gamma}$	shear rate (s^{-1})
τ	shear stress (Pa)
η_{Ca}	Casson plastic viscosity (Pa s)
τ_{Ca}	Casson yield value (Pa)

and is adjusted by changing the feed roll gap at a constant roll speed or by changing the roll speed at a constant gap (Ziegler & Hogg, 1999).

Many minor chocolate manufacturers, in particular, require a compact chocolate-making plant that is smaller than the traditional roll refiner/conching system. Many kinds of these plants have been developed. Perhaps the most common ones are based on re-circulation through a ball mill (Beckett, 1999), which employs the relative motion of loose elements (balls) to generate a grinding action. They are typically vertical or horizontal cylinders, equipped with a rotating shaft with arms, filled to as much as 90% of the available volume with grinding media (steel balls, ceramic beads, etc.). The feed material in the form of a suspension is pumped into the grinding chamber and comminuted between the moving media, the stirrer and the grinding chamber wall by compression and shear. A temperature control system (made up of a water jacket equipped with temperature sensors and thermo-regulators controlled by electric board) allows the initial melting of solid fats, ensures that the product does not suffer thermal damage, such as a burned aroma or milk derivative decay, and it performs the substitutive action of traditional conching (Ziegler & Hogg, 1999).

Since the stirred ball mills born in the first decades of 20th century as refiners for paintings, some works about their use in the mining and powder industries (Gao & Forssberg, 1995; Gao, Forssberg, & Weller, 1996; Kheifets & Lin, 1998; Ma, Hu, Zhang, & Pan, 1998; Tuzun, Loveday, & Hinde, 1995) are present in the literature. On the contrary, very few studies dealing with chocolate refining by means of ball mills have been conducted (Franke, Scheruhn, & Tscheuschner, 2002; Lucisano, Casiraghi, & Mariotti, 2006). However, the use in food industry has modified so much the machines and the process than the technologies are not anymore comparable. For this reason, even if the argumentations of existing documents are attractive, and relations among operating variables and examined physical principles are very interesting, unfortunately they are not suitable to the environment of chocolate refining.

The aim of this study was to optimize working conditions of a ball mill used for chocolate refining process, in order to reduce refining time and energy consumption without forgetting the quality characteristics of the product obtained.

2. Materials and methods

2.1. Experimental design

Experiments were planned following a two-factor, five-level central composite design (CCD), including five central points (Montgomery, 2001). The two factors (independent variables) considered were the refining time ($rt = 33.7$, 40, 55, 70, 76.2 min) and the agitator shaft speed ($as = 41.7$, 50, 70, 90, 98.3 rpm); the extreme levels of the factors were selected on the basis of previous experiments, guidance given by the ball mill manufacturers and the technical limitations of the plant. The 13 combinations obtained are reported in Table 1. The order of experiments was fully randomized to avoid systematic biases. The experimental variables measured were electricity consumption (E), particle size (d_{90}), iron content (Fe), the Casson plastic viscosity (η_{Ca}) and the Casson yield value (τ_{Ca}). Moreover, the refining time factor was also inserted as a response variables

Table 1
Fully randomized experimental order with refining time (*rt*) and agitator shaft speed (*as*) levels

Run order	*rt* (min)	*as* (rpm)
1	55	70
2	55	70
3	70	50
4	55	70
5	55	41.7
6	33.7	70
7	55	70
8	55	98.3
9	76.2	70
10	40	50
11	40	90
12	55	70
13	70	90

(RT) to allow the optimization of the process by the desirability function.

2.2. Chocolate production

Experiments were conducted using the following formulation: 34% cocoa liquor, 17% cocoa butter, 49% icing sugar, 0.4% lecithin and 0.03% vanillin.

For each experiment, a 100 kg batch of dark chocolate was produced by using as refiner a ball mill SOTU-MILL/130 (Packint, Milano, Italy), constituted of a double-jacket cylinder, containing 9.5 mm diameter wear resistant steel balls and a stirring/mixing group. The vertical shaft with horizontal arms, while rotating, puts the steel balls in movement and a recycling pump recycles the chocolate mass through the ball bed. The impact between two contiguous balls or between one ball and the lateral wall of ball refiner provokes the crumbling of the particles existing in the space within the two elements.

The chocolate production starts with the manual feeding of cocoa butter and liquor (both from Barry-Callebaut Italia S.p.A., Assago, MI, Italy) into the mill grinding chamber, pre-heated to 50 °C. Fats were melted at this temperature for about 12 h. On the contrary, refining process was carried out at 60 °C. A temperature control group, by means of a thermostat, controls that product does not suffer flavour damages. Fifteen minutes after the recycling pump is turned on, the agitator shaft starts up and this was considered the starting point of the experiment. During the first 5 min of the experiment, the icing sugar (80 μm; Gelcrem, Bareggio, MI, Italy) was gradually fed in. Lecithin and vanillin (Gelcrem) were added, respectively, 15 and 14 min prior to the end time. After the established refining time, the chocolate was discharged in a tank.

The layout of the plant and a ball mill image are shown in Figs. 1 and 2.

2.3. Electricity consumption

The consumption of electricity was calculated following Eq. (1), used for an asynchronous three-phase motor (Bellini & Figalli, 1994):

$$E = \sum_{i=1}^{n} A_i \cdot V_i \cdot \sqrt{3} \cdot \cos\varphi \cdot t_0 \tag{1}$$

where E (kW h) is the consumption of electricity, i is the ith time interval of 3 min, n is the total number of time intervals, A_i is the current (A) measured by the amperometer at the ith time interval, V_i (V) is the voltage measured by the voltmeter at the ith time interval, $\cos\varphi$ is a constant of the electric motor (0.85 for the motor assembled in the ball mill), and t_0 is the time interval (3 min).

2.4. Particle size

Size distributions of chocolate particles were determined by the laser light-scattering method (McFarlane, 1999), using a particle size analyser MALVERN 2600 (Malvern Instruments Ltd., Malvern, UK). Before the analysis, samples were diluted with acetone and treated in a Branson 2200 ultrasonic system (Branson Ultrasonic Corporation, Danbury, CT, USA) for 15 min. Results are expressed in micrometers as the size of the largest particles (d_{90}, i.e. 90% finer than this size).

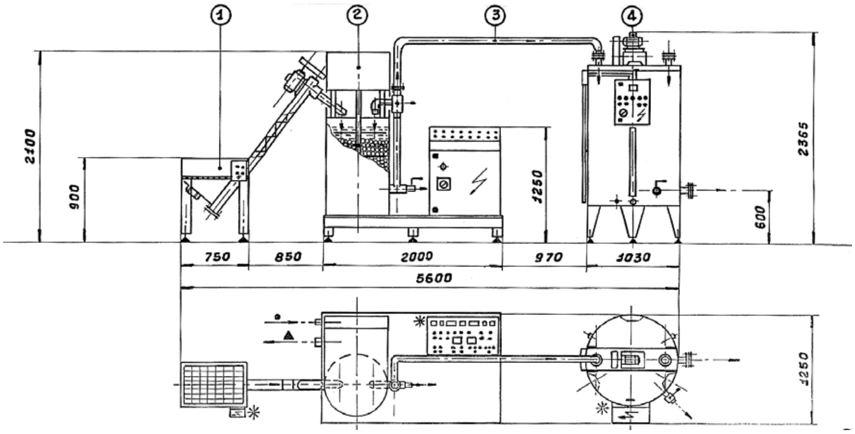

Fig. 1. Layout of the SOTU-MILL/130 plant (Packint, Milano, Italy) used to perform experiments. The line is composed of a powder feeder (1), a ball mill (2) a recycling pump (3) and a tank to store the refined chocolate (4).

Fig. 2. The ball mill (Mill/130, Packint, Milano, Italy) used to perform experiments. Inside the double-jacket cylinder an agitator shaft moves 400 kg of wear resistant stainless steel balls, whose relative movement generates the refining action.

2.5. Iron content

In order to determine the iron content (Fe), samples were burned with a nitric acid and hydrogen peroxide solution employing an Ethos D microwave labstation (Milestone S.r.l., Sorisole, BG, Italy). Then, the absorbance of the extract diluted with deionized water was measured by the atomic absorption spectrometer SpectrAA-10 (Varian Inc., Palo Alto, CA, USA) (McFarlane, 1999). The amounts of mineral were calculated using a standard curve and expressed as mg/kg.

2.6. Rheological parameters

The flow behaviour of melted chocolate samples was studied following the official IOCCC 46 method (2000), using a Bohlin VOR Rheometer (Bohlin Reologi AB Corp., Lund, Sweden), equipped with coaxial cylinders. Shear stress (τ) was determined at shear rates ($\dot{\gamma}$) ranging from 4.63 to 58.2 s^{-1}, holding samples at 40 °C, after a pre-conditioning phase carried out at 55 °C. Yield value (Pa; τ_{Ca}) and plastic viscosity (Pa s; η_{Ca}) were calculated applying the mathematical model of Casson (Chevalley, 1999):

$$\tau^{1/2} = \tau_{\mathrm{Ca}}^{1/2} + \eta_{\mathrm{Ca}}^{1/2} \cdot \dot{\gamma}^{1/2}.$$

2.7. Statistical analyses

Data were analyzed by response surface regression for a second-order polynomial model, which contained linear, quadratic and interaction terms for the two factors. The response surfaces were generated from the Eq. (2) (Montgomery, 2001):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \varepsilon \tag{2}$$

where y is the value of the considered experimental variable, x_1 and x_2 are the values of rt and as, respectively, β_0 is the constant value, β_1 and β_2 are linear coefficients, β_{12} is the interaction coefficient, β_{11} and β_{22} are quadratic coefficients, and ε is the random error. In order to determine the significance of each coefficient, the one way analysis of variance (ANOVA) was carried out.

An overall desirability function was constructed as multi-objective optimization (Eq. (3)). The function is defined as the weighted geometric average of n individual desirability functions (Montgomery, 2001):

$$D = \left(d_{\mathrm{RT}} \cdot d_E \cdot d_{d_{90}} \cdot d_{\mathrm{Fe}} \cdot d_{\eta_{\mathrm{Ca}}} \cdot d_{\tau_{\mathrm{Ca}}}\right)^{1/6} \tag{3}$$

where d_i are the individual desirability functions into which the y_i responses have been converted.

The different d_i vary between the values of 1 and 0, according to whether the respective y_i responses coincide with the fixed target values. If the target for response is minimization (RT, E and Fe) or inclusion within a range of values (d_{90}, η_{Ca} and τ_{Ca}), d_i assumes the values calculated by Eqs. (4) and (5), respectively:

$$d = \begin{cases} 1 & y < T \\ \left(\frac{U-y}{U-T}\right)^r & T \leqslant y \leqslant U \\ 0 & y > U \end{cases} \tag{4}$$

$$d = \begin{cases} 0 & y < L \\ \left(\frac{y-L}{T-L}\right)^{r_1} & L \leqslant y \leqslant T \\ \left(\frac{U-y}{U-T}\right)^{r_2} & T \leqslant y \leqslant U \\ 0 & y > U \end{cases} \tag{5}$$

where T is the target value for response y, U is the upper limit, while L is the lower limit. Finally r are the weights assigned to each desirability function: the higher the value (higher than 1), the more emphasis is placed on the target value, again as regards minimization and range, respectively.

All statistical analyses were performed using Minitab 14 software (Minitab Inc., State College, PA, USA).

3. Results and discussion

Experimental design (CCD) considered refining time (rt) and agitator shaft speed (as) as factors. Chocolate formulation and the quantity processed were kept constant, as was the refining temperature. Indeed, even if temperature particularly affects chocolate, determining its flow parameters and also affecting particle size distribution, it can, however, also cause variations in aroma – at the worst, burning – which must be avoided at all costs. Its value must be high to favour moisture and anomalous flavour elimination and, at the same time, it must be kept below temperatures that might lead to product damage (Pontillon, 1998). For this reason, temperature value is practically fixed, i.e. there is a higher limit (burning) and a lower limit (eliminating moisture and anomalous flavours) which lead to the choice of only one temperature: the one utilized, i.e. 60 °C.

To develop the CCD, measured experimental variables were chosen from the main characteristics that describe unmoulded chocolate. Table 2 shows the results relating to the different experimental variables measured for all experiments carried out.

As far as particle size is concerned, d_{90} of the distribution curve was evaluated, which is usually considered acceptable if lower than 23 μm (Beckett, 1999). It is in fact believed that, over this dimension particles can produce an unpleasant sand effect in the mouth (Beckett, 1994). The d_{90} values obtained, indicated in Table 2, are included between 14.5 and 28.5 μm; some products would therefore prove to be inadequate (experiment nos. 6 and 11). On the other hand, 6 μm is the minimum particle size if optimum rheological properties are to be achieved in the chocolate mass (Kruger, 1999). Indeed, there are also ranges of acceptability for the rheological parameters of chocolate, which depend on the final use for which the product is intended: Casson viscosity (η_{Ca}) from 1 to 2 Pa s, Casson flow limit (τ_{Ca}) from 5 to 10 Pa (Pontillon, 1998). All the values of η_{Ca} obtained in the different experiments range between a very restricted value interval, as indicated in Table 2; this is due to the utilization of lecithin, which is known to attenuate the effect of subfines on rheological properties. Even if less strongly, the same can be stated for τ_{Ca} which, even if it has a higher range, never exceeds the value of 10 Pa, as shown in Table 2.

Table 2
Results relating to response variables, measured for all experiments carried out

Run order	d_{90} (μm)	η_{Ca} (Pa s)	τ_{Ca} (Pa)	Fe (mg/kg)	E (kW h)	RT (min)
1	16.2	1.63	5.73	22.9	3.22	55.0
2	15.7	1.63	6.87	24.2	3.41	55.0
3	19.2	1.68	8.42	23.9	3.10	70.0
4	17.8	1.63	8.76	24.3	3.28	55.0
5	16.8	1.77	6.69	22.5	1.87	55.0
6	25.5	1.79	5.78	25.5	1.88	33.7
7	20.1	1.58	8.07	24.9	3.08	55.0
8	23.0	1.53	7.78	27.6	3.11	55.0
9	17.5	1.57	9.26	25.3	4.20	76.2
10	20.6	1.83	4.70	24.0	1.54	40.0
11	28.5	1.68	6.22	28.7	2.17	40.0
12	14.5	1.71	7.04	24.6	3.03	55.0
13	16.9	1.65	8.20	26.5	3.78	70.0

d_{90}, particle size index; η_{Ca}, Casson viscosity; τ_{Ca}, Casson flow limit; Fe, iron content; E, ball mill energy consumption; and RT, refining time.

Iron concentration in the refined chocolate (Fe) was determined as this metal constitutes the main contamination factor in the case of chocolate processing by ball mill, due to the wear of the moving media. The presence of iron in chocolate, the limits of which are not determined by the law but only suggested in the Codex Alimentarius (20 mg/kg), can cause product shelf-life to be reduced, by catalysing the oxidation of the cocoa butter. In fact, as expected, the data obtained (Table 2) indicate high levels of iron in all experimental samples. However, the analysis of ingredients revealed an iron concentration equal to 23.59 mg/kg and 30.43 mg/kg for sugar and cocoa mass, respectively. Considering that, respectively, 49% and 34% of these two ingredients are contained in the formulation, it can be said that approximately 20 mg/kg of the iron present in the various samples of chocolate was due to ingredients and not to process. This information was considered during elaboration of the target function.

Finally the energy consumption (E) of the ball mill was calculated. This, as expected, increased proportionately to the increase in speed and refining time (Table 2).

From the analysis of effects (not reported), which indicates the importance of the effect of each factor on each measured quality characteristic, it was noticed that *as* and *rt* had opposite effects on d_{90} but the same effect on η_{Ca}. This means that if an increase in *as* diminishes particle size, in the long run, an increase in *rt* causes re-aggregation, i.e. the value of d_{90} increases, while viscosity increases with both the increase in *as* and in *rt*. Interaction among the factors of experiments proved to be particularly significant in the case of d_{90}. Scatterplots analysis confirmed that the results obtained were independent from the run order of experiments (Montgomery, 2001).

A complete second-order model was fitted to the experimental data (Eq. (2)) and the analysis of variance (ANOVA) indicated the acceptability of the various models. The coefficients of the fitted models are shown in Table 3.

As regards as E, results indicate significant but not very high second-order coefficients, meaning an increase of energy consumption as a consequence of *rt* and *as* increasing. The most significant second-order polynomial model coefficients were that of d_{90}, which showed the phenomenon of particle re-aggregation, already described in the literature (Beckett, 1994): prolonged refining time (*rt*) and/or high speed (*as*) determines re-aggregation of sugar crystals in amorphous form. The crossed second-order coefficient, significant even if not very high, indicates that the response curve of d_{90} has a minimum point. Actually Fig. 3 showing the iso-response surface of d_{90}, confirms the expectations of data analysis and ANOVA, showing an area with an important minimum point.

Considering Fe, the coefficients reported in Table 3 indicate that iron content is statistically independent from *as* (coefficient β_2 not significant), whereas second-order coeffi-

Table 3
Coefficients of second-order polynomial model (Eq. (2)) for experimental response variables, with regression coefficients and lack of fit of ANOVA

Response variable	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}	R^2	Lack of fit (P-value)
E	-7.1719^{***}	0.1057^{*}	0.152^{***}	0.0005^{*}	0.0009^{***}	0.0000	0.981	0.581
d_{90}	42.7962^{*}	−0.8166	−0.0304	0.0109^{**}	0.0042^{*}	-0.0084^{*}	0.896	0.914
Fe	29.3472^{**}	−0.2680	−0.0117	0.0034^{*}	0.0014	−0.0017	0.905	0.574
η_{CA}	3.1439^{***}	−0.0273	−0.0151	0.0001	0.0000	0.0001	0.809	0.357
τ_{CA}	−6.4056	0.1949	0.1509	−0.0000	−0.0003	−0.0014	0.727	0.931

Significance levels: * 95%; ** 99%; *** 99.9%.
d_{90}, particle size index; η_{Ca}, Casson viscosity; τ_{Ca}, Casson flow limit; Fe, iron content; and E, ball mill energy consumption.

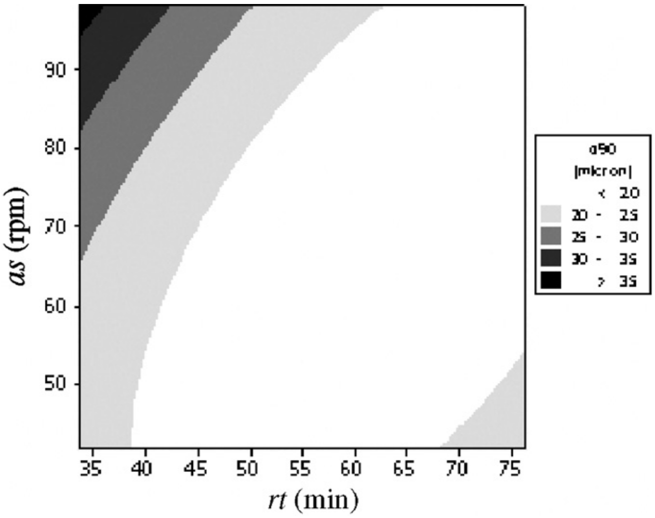

Fig. 3. Iso-response surface of particle size (d_{90}).

cient for rt (β_{11}) is significant. Actually, the Fe iso-response surface presented in Fig. 4 shows an area of minimum, confirming the curvature suggested by β_{11}.

Finally, η_{Ca} and τ_{Ca} coefficients reported in Table 3 are statistically not significant indicating that both parameters remain quite constant with the variation of rt and as.

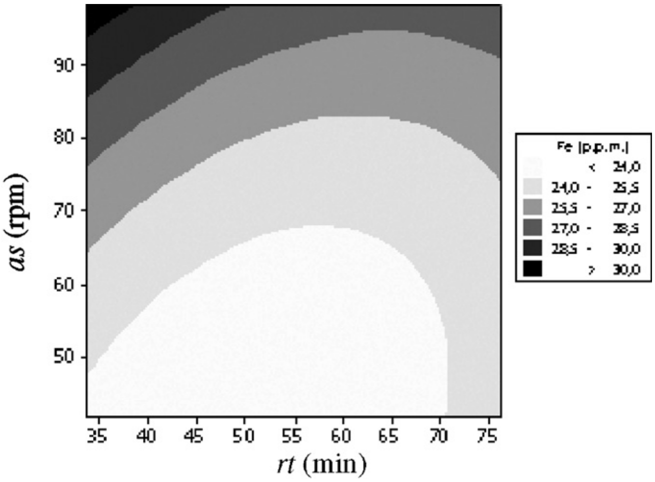

Fig. 4. Iso-response surface of iron content (Fe).

Optimization consists in a bound minimization of variable RT using the desirability function (Montgomery, 2001).

In our case the objective was to achieve a minimum RT with the following specifications:

$$\text{Min}(E)$$
$$20\ \mu\text{m} \leqslant d_{90} \leqslant 23\ \mu\text{m}$$
$$1\ \text{Pa s} \leqslant \eta_{Ca} \leqslant 2\ \text{Pa s}$$
$$5\ \text{Pa} \leqslant \tau_{Ca} \leqslant 10\ \text{Pa}$$
$$\text{Min}(\text{Fe})$$

In Table 4, the lower, upper and target values are listed, together with the weight of each variable for the desirability function. A target value of 30 min and an upper limit of 45 min were assigned to RT. The longer working time proposed by the producer (55 min) was considered to be excessive, since it was established on the basis of particle size measurement carried out by a manual micrometer. In fact, the uncertainty of this method obliged the producer to refine chocolate more than necessary, in order to be sure that the particle size of the final product would not exceed the desired value. The accuracy introduced by light-scattering method allowed to cut this extra-time. Since the reduction of RT was the main aim of this study, the maximum weight was assigned to this variable, i.e. 10.

The weight assigned to E was practically zero (0.1). E was in fact been measured and included in the target function solely because, other variables being equal, the combination of as and rt that cost less energy was chosen, owing to problems connected with the environment and responsible consumption and because it may be possible to reutilize the data collected in future optimizations.

Table 4
Desirability function values (lower, upper and target) and weight of each variable

Response variable	Aim	Lower	Target	Upper	Weight
RT (min)	Minimize		30	45	10
E (kW h)	Minimize		3	6	0.1
d_{90} (μm)	Target	20	21	23	5
Fe (mg/kg)	Minimize		20	30	1
η_{Ca} (Pa s)	Target	1	1.5	2	1
τ_{Ca} (Pa)	Target	5	8	10	1

RT, refining time; E, ball mill energy consumption; d_{90} = particle size index; Fe, iron content; η_{Ca}, Casson viscosity; and τ_{Ca}, Casson flow limit.

The easiest choice to make consisted in establishing the best value of d_{90}, for which limits are fixed in the literature (Beckett, 1994; Pontillon, 1998), i.e. 20 μm and 23 μm. The target value and the weight of this variable were set at 21 μm and 5, respectively, in order to be sure that the percentile value was not too close to the maximum limit.

As already stated, as approximately 20 mg/kg of the iron present in the final product came from the ingredients, the target value and the upper limit for Fe was fixed at 20 and 30 mg/kg, respectively. Thus it was ensured that the chocolate production process minimizes the release of iron from the balls in the mill.

As regards η_{Ca}, in view of the fact that results were acceptable even when the process was carried out under the most extreme conditions, it was decided that strict restrictions should not be imposed as, for most uses, chocolate viscosity values between 1 and 2 Pa s are more than acceptable (Pontillon, 1998). Similar considerations can be made for τ_{Ca}, the values of which vary more with respect to the other Casson parameter, but are in any case contained within the range advised in the literature, equal to 5–10 Pa (Pontillon, 1998).

The overall desirability function gave an optimum of 58 rpm *as* and 38.5 min *rt*. At these coordinates, response variable values are as follows:

$E = 1.85\ \mathrm{kW\,h}$

$d_{90} = 21.0\ \mu\mathrm{m}$

$\mathrm{Fe} = 24.2\ \mathrm{mg/kg}$

$\eta_{Ca} = 1.80\ \mathrm{Pa\,s}$

$\tau_{Ca} = 5.31\ \mathrm{Pa}$

This solution fulfils all the restrictions imposed on the construction of the individual functions and complies with the specifications for achieving maximum global desirability.

The choice of the weights assigned to each quality characteristic in optimization is only apparently arbitrary, as it is defined by production requirements and by the knowledge and experience of the technicians working with these plants. On the other hand, the analysis of sensitivity to variations in the assigned weights demonstrated that the results are constantly replicable.

Sensitivity analysis was carried out on the RT which, as it is the most important variable for optimizing and measuring the production capacity of the machine, proved to be an interesting cost variable. A further aim of the analysis was to evaluate the trend in the cost variable with respect to d_{90} weight variations, as the experiments carried out have demonstrated that acceptable particle size dimensions are not always obtained. Variables E, Fe, η_{Ca} and τ_{Ca} were excluded from the sensitivity analysis: their values proved to be already very stable and it would therefore make no sense to examine the influence of the weight assigned to them on response variable RT.

The analysis was carried out applying variations of one unit to the weights assigned to RT and to d_{90} and evaluating the performance of the mathematical model. It was established a priori that the mathematical model is to be considered stable if, for each weight variation made, the cost variable RT value remains within a semi-neighbourhood of 5 min with respect to the value calculated in the original optimization. The whole area around the original combination was explored, i.e. situations implying a contemporary variation of the two weights were included. Observing the results obtained (Table 5), and bearing in mind that the initial optimization solution was 38.5 min *rt*, it can be affirmed that all weight variations considered lead to a value of the RT variable that respects the conditions whereby the semi-neighbourhood should be less than 5 min. It can be therefore affirmed that, in view of the decided limits (weights of Table 4), in a neighbourhood consonant with the original conditions the solution remains stable. The model with weight 10 for RT and weight 5 for d_{90} is thus further corroborated.

Table 5
Sensitivity analysis results as a function of weight variations

RT weight	d_{90} weight	RT (min)
10	6	40.31
10	4	43.54
9	4	43.54
9	5	38.15
9	6	39.68

RT, refining time and d_{90}, particle size index.

At this point an experiment was carried out to corroborate the results obtained, maintaining the same conditions and procedures of previous trials, setting 58 rpm for *as* and 38.5 min for *rt*. The results obtained confirmed the expectations:

$E = 1.84\ \mathrm{kW\,h}$

$d_{90} = 21.1\ \mu\mathrm{m}$

$\mathrm{Fe} = 24.0\ \mathrm{mg/kg}$

$\eta_{Ca} = 1.71\ \mathrm{Pa\,s}$

$\tau_{Ca} = 5.24\ \mathrm{Pa}$

4. Conclusions

Starting out from a company's need to ensure that the working conditions of its machine were ideal for the production required, an experimental design was employed to optimize the working process. Referring to the considered chocolate recipe, before experimentation and statistical analysis, the working conditions for the ball mill used were 55 min at 70 rpm for a batch of 100 kg. However, the new optimal conditions prove to be 38.5 min at 58 rpm, again for a 100 kg batch. By making these changes, output rises from 109 kg/h to 156 kg/h, with a 43% increase in productivity. Thus, the new working conditions confirmed the refining capability of the ball mill alternative technology in chocolate production. Another important results was low iron content of the final product, meaning the good quality of anti-wear steel balls.

The optimization procedure can be extended to other recipes, but a second important step must be a serious statistical analysis and optimization of ball mill constructive characteristics, such as diameter of the inner cylinder of the mill, height of the inner cylinder, and diameter of the balls.

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