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KINETICS OF DRYING IN A *MENTHA CRISPA* FOAM LAYER AND ADJUSTMENT OF MATHEMATICAL MODELS

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ABSTRACT

Mentha crispa is a plant commonly used in folk medicine to treat illnesses and has wide industrial application. Dehydration has been used for several types of plants in order to reduce the levels of water content and water activity, so that it would allow na adequate storage due to the conservation of the active principles. The objective of this work is to investigate the process of drying mint in a foam layer. The foam was physically characterized in terms of density, expansion percentage, air incorporation capacity and stability. Drying was carried out according to a factorial experimental design, in which the input variables were: temperature (50, 60 and 70 °C), concentration of foaming agent (3, 5 and 7 %) and stirring time (3, 5 and 7 min); and the output variables were: drying time and water content (X_{bs}) of the product. The physical properties of the foam depended only on the foaming concentration. The rise in the temperature significantly reduced the drying time and the water content of the foam. The Page, Henderson & Pabis, Newton and Modified Page mathematical models were adjusted to the experimental data and Page's model was the one that showed to be the most adequate to represent the drying phenomenon.

Palavras-chave:
Desidratação
Hortelã
Mentha spicata L.
Modelagem matemática
Teor de água

CINÉTICA DE SECAGEM EM CAMADA DE ESPUMA DA *MENTHA CRISPA* E AJUSTE DE MODELOS MATEMÁTICOS

RESUMO

A hortelã-da-folha-miúda (*Mentha crispa*) é uma planta comumente utilizada na medicina popular para tratamento de enfermidades e possui vasta aplicação industrial. A desidratação vem sendo empregada para diversos tipos de plantas com o intuito de diminuir os níveis de teor de água e atividade de água, a fim de permitir um armazenamento adequado em função da conservação dos princípios ativos. O trabalho tem como objetivo estudar o processo de secagem em camada de espuma da hortelã-da-folha-miúda. A espuma foi caracterizada fisicamente em termos de densidade, porcentagem de expansão, capacidade de incorporação de ar e estabilidade. A secagem foi realizada de acordo com um planejamento experimental fatorial, no qual as variáveis de entrada foram: temperatura (50, 60 e 70 °C), concentração de agente espumante (3, 5 e 7 %) e tempo de agitação (3, 5 e 7 min); e as variáveis de saída foram: tempo de secagem e teor de água (X_{bs}) do produto. As propriedades físicas da espuma apresentaram dependência apenas da concentração de espumante. O aumento da temperatura reduziu significativamente o tempo de secagem e o teor de água da espuma. Os modelos matemáticos Page, Henderson & Pabis, Newton e Page Modificado foram ajustados aos dados experimentais e o de Page foi o que se mostrou mais adequado para representar o fenômeno de secagem.

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INTRODUCTION

Historically, medicinal plants are important as herbal medicines and in the discovery of new drugs, with the plant kingdom making the greatest contribution of medicines. Researchers in the area of natural products are impressed by the fact that these products found in wild reveal an enormous range of diversity in terms of structure and physical-chemical and biological properties (BRASIL, 2012; GUERRA & NODARI, 2001).

The *Labiatae* family includes about 220 genera and 3,300 species widely used for several purposes around the world. Plants belonging to the *Labiatae* family are rich in polyphenolic compounds and a large number of them are well known for their antioxidant properties, being widely used in traditional medicine for exhaustion, weakness, depression, improving memory, improving circulation, strengthening fragile blood vessels, inflammation, infection, indigestion and gastritis (BENABDALLAH *et al*., 2016; SHEKARCHI *et al*., 2012).

The genus *Mentha* is an important member of the *Labiatae* family, which includes eighteen species and eleven hybrids (BENABDALLAH *et al*., 2016). *Mentha crispa*, also known as smalll leaf mint in Brazil, originates from the cross between *Mentha spicata L.* and *Mentha suaveolens* Ehrh (ZEMIANI et al., 2021). *Mentha crispa* is one of the economically significant species of the genus and is distributed throughout the world, in tropical and temperate regions. According to the modern classification, *Mentha crispa* is synonymous with mint *Mentha spicata L*., and these are species that are characterized by a strongly wavy leaf blade (MALANKINA *et al*., 2022).

The economic interest in *Mentha* species is particular because of the commercial exploitation of essential oils, which are complex substances with great chemical polymorphism (DESCHAMPS *et al*., 2008). One of the predominant compounds found in species of the genus is the monoterpene rotundifolone (piperitenone oxide). In addition, the essential oil of *Mentha crispa* contains about 70% of this secondary plant compound (DE SOUSA *et al*., 2016). Further, *M. crispa* essential oil has several biological properties, including antinociceptive, spasmolytic, antimicrobial, larvicidal, trypanocidal, cytotoxic and antitumor (TURKEZ *et al*., 2018).

Medicinal plants are used in various forms, but the pharmaceutical industry is more interested in dry plant extracts as solid forms offer greater advantages over conventional liquid forms, such as greater concentration and stability of active substances, as well as ease of use, standardization and handling, contributing to guarantee the homogeneity of pharmaceutical preparations (GALLO *et al*., 2013). Humidity control in the conservation of plant species is also extremely important, as it has a direct influence on the metabolic activity, multiplication, resistance and survival of microorganisms (RIGUETO *et al*., 2018).

The reduction of free water can be obtained through drying, which contributes to the conservation and prolonged use of biological materials (SILVA & PEDRO, 2018). This unit operation aims to reduce and inhibit the chemical, enzymatic and microbiological activities that are responsible for food deterioration (BALDI *et al*., 2021). This implies lower transport, packaging and storage costs for food, as products can be stored at room temperature without the need for refrigeration (OLIVEIRA *et al*., 2020).

Foam layer drying is a technique that converts liquid or semi-liquid foods into stable foam through cooperation with foaming or stabilizing agents, and can be applied to various types of foods. This method is relatively simple and can be performed at a lower cost than spray drying and lyophilization, in addition to being performed at lower temperatures, which helps to maintain nutrients in the vegetables (MEI LING & SULAIMAN, 2018). Another advantage of this method is that it provides a shorter drying time due to the greater surface area exposed to heat during drying and the provision of uniform heating, thus accelerating the moisture diffusion rate. The powders obtained through this process

reduce water activity, extend shelf life, have high economic potential and can be easily reconstituted (MOHAMED *et al*., 2022).

In the work on drying, optimization, determination of commercial viability and improvement and sizing of equipment, simulation and obtaining theoretical information regarding the behavior of each product during water removal are of fundamental importance (COELHO *et al*., 2019). The drying conditions, the type of dryer and the characteristics of the material to be dehydrated influence the drying kinetics (ONWUDE *et al*., 2016). Several mathematical models have been used to describe the water loss during the drying process, under the conditions under which the experimental data were obtained.

Consequently, the objective of this work was to investigate the foam layer drying process of mint (*Mentha crispa*) through the physical characterization of the foam, obtaining kinetic curves and fitting mathematical models to the experimental data and evaluation of the influence of independent variables of the process on the drying time and water content of the product.

MATERIAL AND METHODS

The experiments were conducted at the Thermodynamics Laboratory, owned by the Technology Center of the Federal University of Paraíba, Campus João Pessoa – state of Paraíba, Brazil. *Mentha crispa* was obtained fresh, in the local market in the city of João Pessoa, state of Paraíba from the Hortaliças Sempre Verde Company and taken to the Thermodynamics Laboratory, where they were sanitized in accordance with Resolution RDC No. 218/2005 of ANVISA (BRASIL, 2005), which regulates hygienic-sanitary procedures for handling food and beverages prepared with vegetables. Branches of *Mentha crispa* used in the preparation were subjected to selection to remove deteriorated parts and/or units, vectors, dirt and other foreign matter and then washed and disinfected. Next, the stems and branches of the plant were removed and a vegetable derivative of the leaves was prepared, using a percentage of 15% m/v of leaf/distilled water, through grinding in a Metvisa high-speed industrial blender, at a maximum power of 800 W, and speed of 18000 rpm for about one (01) minute, to obtain a homogeneous mixture.

Experimental design and statistical analysis

The complete factorial experimental design was used to evaluate the efficiency of the foam layer drying process of *Mentha crispa*, in order to verify significant effects of the input variables on the response variables. The definition of the independent variables of the process was based on studies of the foam drying of the blackberry leaf (SILVA, 2019), soursop leaf (OLIVEIRA *et al*., 2020) and the inner bark of *juazeiro* (MOREIRA *et al*., 2021). The assessed planning matrix verified the influences of the input variables: stirring time for foam production, percentage concentration of foaming agent and drying temperature; on the response variables: drying time and final water content. The non-coded values were defined from preliminary tests and are shown in Table 1.

The complete factorial experimental design used in this work was of the 2^3 type (2 x 2 x 2) with three repetitions at the central point, resulting in a matrix with 11 experiments. In order to comply with the randomness of the experimental design, the tests were not carried out in the order in which they are found in the adopted planning matrix, described in Table 2.

In the statistical analysis of the factorial experimental design, the computational package Statistica version 7.0 was used. The program was used to calculate the principal effects and interactions of the independent variables on the responses and for analysis of variance (ANOVA), enabling the optimization of the drying process.

Foam production

Preliminary tests with *Mentha crispa* were based on the experiment by Bag et al. (2011), who report that foams that do not collapse for at least 1 hour at room temperature are considered

Table 1. Coded and real values of independent variables

Independent values	Level (-1)	Central Point (0)	Level (+1)
Concentration (%)	3	5	7
Temperature (ºC)	50	60	70
Stirring time (min)	3	5	7

Source: The author

Table 2. Experimental design matrix

Experiment	C (%)	T (°C)	t_{ag} (min)
1	3 (-1)	50 (-1)	3 (-1)
2	7 (+1)	50 (-1)	3 (-1)
3	3 (-1)	70 (+1)	3 (-1)
4	7 (+1)	70 (+1)	3 (-1)
5	3 (-1)	50 (-1)	7 (+1)
6	7 (+1)	50 (-1)	7 (+1)
7	3 (-1)	70 (+1)	7 (+1)
8	7 (+1)	70 (+1)	7 (+1)
9	5 (0)	60 (0)	5 (0)
10	5 (0)	60 (0)	5 (0)
11	5 (0)	60 (0)	5 (0)

C = foaming agent concentrations; T = drying temperature; t_{ag} = stirring time

mechanically stable for the drying process. Sodium carboxymethylcellulose, albumin, gelatin and Portogel® emulsifier were evaluated in terms of foam stability and concentration. The foaming agent that proved to be the most efficient and suitable for the process requirements was the synthetic food additive Portogel®, which has a chemical composition of water, propylene glycol, distilled monoglycerides, polyglycerol ester and potassium stearate. To produce the foam, the plant derivative obtained, together with the emulsifier, was stirred in a 400-W Philco Paris Cristal Maxx mixer at stirring speed 6. The stirring speed was the maximum of the equipment and was fixed for the performance of all experiments.

Foam physical Properties analysis

According to the method of Brock *et al*. (2008), foam density should be measured at room temperature (25±1°C) and foam samples weighed in graduated cylinders. After weighing, the density was determined using the ratio between the quantified mass and the volume of the cylinder used, previously checked with distilled water at room temperature (Equation 1).

$$\rho = \frac{m}{v} \quad (1)$$

Where,
ρ = foam density (g/cm³);
m = foam mass (g);
v = volume occupied by the foam (cm³).

The percentage of foam expansion resulted from the previously calculated density values of the vegetal derivative of the leaves and their respective foams, according to Equation 2 and methodology described by Feitosa *et al*. (2017).

$$\%expansion = \left(\frac{\frac{1}{\rho_e} - \frac{1}{\rho_s}}{\frac{1}{\rho_s}} \right) x100 \quad (2)$$

Where,
$\%expansion$ = foam expansion percentage (%);
ρ_e = foam density (g/cm³);
ρ_s = juice density (g/cm³).

The air incorporation capacity is related to the volume of air introduced into a solution. It is determined by the increase in volume of the formed foam, and obtained according to the methodology of Moreira *et al*. (2021). The calculation of the

percentage of air incorporation capacity was performed from the values of the mixture volume before beating and after beating, according to Equation 3.

$$\%over\ run = \left(\frac{V_f - V_i}{V_i}\right) x100 \quad (3)$$

Where,
$\%over\ run$ = percentage of air incorporation capacity (%);
V_f = foam volume after stirring (cm^3);
Vi = initial volume before stirring (cm^3).

The stability of the foam was evaluated according to the methodology cited by Karim and Cheewai (1999), adapted from an old method described by Sauter and Montoure (1972). The drainage method is based on depositing the foam in a nylon filter supported by a funnel in a graduated cylinder and subjecting it to conditions identical to those found during drying.

All analytical determinations of foam physical properties were performed in triplicate. The experimental data from the characterization of the samples were statistically analyzed using a completely randomized design with comparison among the means using the test of Tukey ($p < 0.05$), and ASSISTAT version 7.7 Beta computer program (SILVA & AZEVEDO, 2016).

Drying on foam layer

The foams were arranged in aluminum trays with a fixed thickness of 0.5 cm and placed in an oven with air circulation and renewal SL 102/221 Solab, at the temperatures defined by the experimental design (Table 2). The reduction in water content was determined by weighing the sample on a semi-analytical scale, at time intervals of 5, 10, 30 and 60 minutes. Three consecutive measurements were adopted as a criterion for determining constant mass at regular 60-minute intervals, without changing the mass of the samples (MATOS *et al*., 2022). The dry material was removed from the trays with the aid of spatulas, weighed and then packed in airtight polyethylene bags. The water content of the samples was determined in an oven at 105 °C, in triplicate, according to the methodology Adolfo Lutz (2008).

According to Equation 4, the ratio of water content of the *Mentha crispa* leaf was determined during oven drying, under different temperature conditions. Using data obtained experimentally through drying kinetics, drying curves were plotted using Origin version 6.0.

$$RX = \frac{X - X_e}{X_i - X_e} \quad (4)$$

Where,
RX = water content ratio (dimensionless);
X = water content of the sample at a given drying time (bs);
Xi = initial water content of the sample (bs);
Xe = moisture content in the sample balance (bs).

Several mathematical models have been used to describe the drying process; and although several theories have been proposed to predict the behavior of some foods, in most cases, semi-empirical and empirical relationships have been the best options to describe the drying process (BROOKER *et al*., 1992). Different models proposed in the literature were adjusted to predict the drying kinetics in a *Mentha crispa* foam layer (Table 3).

The non-linear regression analysis was performed using the Gauss-Newton estimation method and the models were adjusted using the Statistica computer program version 6.0. To evaluate the mathematical fit of the drying kinetics curves, the models were selected considering the

Table 3. Mathematical models used to predict the drying phenomenon

Model designation	Model	Equation
PAGE	$RX = \exp(-k.t^n)$	(5)
HENDERSON & PABIS	$RX = a.\exp(-k.t)$	(6)
MIDILLI & KUCUK	$RX = a.\exp(-k.t^n) + b.t$	(7)
NEWTON	$RX = \exp(-k.t)$	(8)
MODIFIED PAGE (MANGUEIRA ET AL., 2020)	$RX = a.\exp(-k.t^n)$	(9)

Where, RX = Product water content ratio (dimensionless); t = drying time (h); k = drying coefficients; A, n = constants of the mathematical models

magnitude of the determination coefficient (R^2) (Equation 10), standard error of the estimate (S) (Equation 11), mean squared error (MSE) (Equation 12) and chi-square test (χ^2) (Equation 13).

$$R^2 = 1 - \left(\frac{\sum_{i=1}^{n} \left(RX_{pred,i} - RX_{exp,i} \right)^2}{\sum_{i=1}^{n} \left(RX_{exp,i} - RX_{pred,i} \right)^2} \right) \quad (10)$$

$$S = \sqrt{\frac{\sum_{i=1}^{n} \left(RX_{exp,i} - RX_{pred,i} \right)^2}{GLR}} \quad (11)$$

$$MSE = \frac{\sqrt{\sum \left(RX_{pred,i} - RX_{exp,i} \right)^2}}{n} \quad (12)$$

$$\chi^2 = \frac{\sum_{i=1}^{n} \left(RX_{pred,i} - RX_{exp,i} \right)^2}{DFM} \quad (13)$$

Where,

$RX_{pred,i}$ = ratio of water content predicted by the model;

$RX_{exp,i}$ = experimental moisture content ratio;

n = number of experimental observations;

DFM = degrees of freedom of the model.

The drying rate was also calculated from the relationship between the moisture content (d.b.) and the drying time and drying area of the foam, according to Equation 14.

$$W = -\frac{m_{ss} \cdot dX_{bs}}{A \cdot dt} \quad (14)$$

Where,

W = drying rate (g/cm².min);

m_{ss} = mass of dry solids in the sample (g);

A = drying area (cm²);

X_{bs} = moisture on a dry basis; t = time (min).

RESULTS AND DISCUSSION

The physical properties of the foam are shown in Table 4. As the temperature variable has no influence on the physical characterizations of the foam because they are analyzes that precede drying, its effect was neglected in the statistical analyses. The results of each statistical analysis, for the 95% confidence level, were expressed in Pareto graphs.

It can be seen on Table 4 that experiment 2 had the highest density value and experiment 5 had the lowest value. Such observations can be explained due to the variable concentration of emulsifier (%), which is found in lower concentration in higher density experiments and in higher concentration in lower density experiments. This behavior is compatible with the premise that the smaller the volume occupied by a particular mass, the greater its density.

The foam density is a very important property to evaluate, as it exerts a great influence on the stability as well as the efficiency of the drying process. Kudra and Ratti (2006) report that foams with lower density present greater stability and facilitate the removal of water during drying, thus allowing them to be dehydrated at milder temperatures.

According to the results obtained, it can be seen that the density variation range of *Mentha crispa* foam between 0.1457 and 0.1777 g/cm³ presents good stability, showing to be viable for the use of the foam layer drying technique. These values are within the range predicted by Van Arsdel and Copley (1964), who suggest values between 0.1 and 0.6 g/cm³ for an ideal foam for dehydration.

The analysis of the Pareto Chart (Figure 1), shows that only the variable foaming concentration and the interaction between concentration and stirring time are statistically significant for the 95% confidence level ($p < 0.05$), that is, that significantly affect foam density. The analysis also shows that the concentration and stirring time exert a negative sign influence on the density, so that the smaller their values are, the higher the density value will be. Based on the statistical analysis it was possible to generate a model, through linear regression of the experimental data of the process, of the correlation

Table 4. Physical properties of *Mentha crispa* foam

Experiment	Density (g/cm³)	Expansion (%)	Air incorporation (%)
1 (C 3%; t_{ag} 3 min)	0.1649 ± 0.0089 a	415.52 ± 36.31 a	480.00 ± 16.33 c
2 (C 3%; t_{ag} 7 min)	0.1777 ± 0.0024 a	376.78 ± 12.79 a	506.67 ± 9.43 c
3 (C 5%; t_{ag} 5 min)	0.1542 ± 0.0069 a	453.01 ± 26.34 a	682.50 ± 26.69 ab
4 (C 7%; t_{ag} 3 min)	0.1625 ± 0.0002 a	421.15 ± 10.37 a	616.67 ± 30.91 b
5 (C 7%; t_{ag} 7 min)	0.1457 ± 0.0189 a	489.79 ± 68.01 a	713.33 ± 33.99 a

The values represent an mean ± standard deviation. Different letters on the same line differ significantly by the test of Tukey ($p < 0.05$). C = concentration of foaming agent; Tag = stirring time.

of the density with the independent variables foaming concentration (C) and stirring time (T_{ag}). This model had a percentage of explained variation of 80.13% and a standard error of 0.0000166 and is represented by Equation 15.

$$Density = 0.1603 - 0.0171C - 0.0148t_{ag}\,C \quad (15)$$

The percentage of foam expansion is an important characteristic to be investigated to evaluate the efficiency of the method used for foam formation. A high percentage of expansion indicates that a greater amount of air is trapped in the foam, and a low concentration of foam is not able to form a critical adequate lamella thickness to keep the resulting air bubbles trapped. Susanti *et al*. (2021) state that the percentage of expansion indicates the ability of the emulsifying agent to create bubbles without rupture. Also, according to the authors, very long beating times can cause the rupture of the foam microstructure, through the coagulation of proteins to form insoluble aggregates and decrease the water retention capacity.

It can be seen in Table 4 that the highest mean expansion value was found in Experiment 5 (489.79%), where the maximum emulsifier concentration and maximum agitation time of the experimental design were used. Experiment 2 (376.78%) showed the lowest mean expansion percentage, where the stirring time used was the top level of the planning and the foaming concentration was the minimum. Oliveira et al. (2020) also observed that lower foam densities lead to greater air incorporation and retention, and, consequently, greater foam expansion.

Statistical analysis (Figure 2) shows that only the foaming concentration variable and the interaction between the two variables are statistically significant ($p < 0.05$). Both variables exert a positive influence on the percentage of expansion, so that the higher their values, the greater the expansion

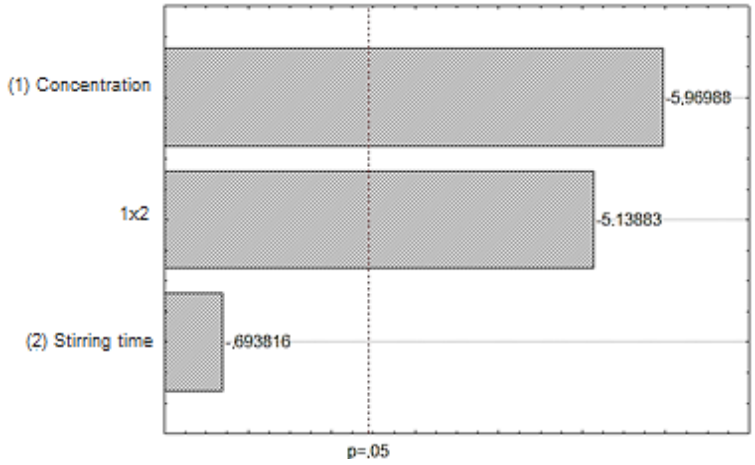

Figure 1. Pareto's Chart: Effect of foaming concentration and stirring time on *Mentha crispa* foam

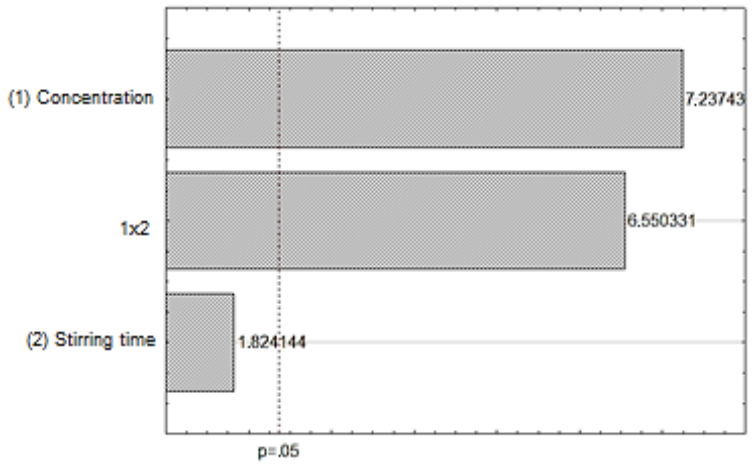

Figure 2. Effect of foaming concentration and stirring time on the percentage of expansion of *Mentha crispa* foam

of the foam. The model generated through linear regression of the experimental data of the process had a percentage of explained variation of 93.37% and can be represented by Equation 16.

$$\%expansion = 430.02+59.32C+53.69t_{ag}\ C \quad (16)$$

Another important physical property of foams is the amount of gas or air that can be incorporated during its change in structure from a viscous liquid to a semi-solid structure. The lowest values of air incorporation capacity for foams are those linked to the minimum level of emulsifier concentration (Experiments 1 and 2). Despite the stirring time between them varying between the upper and lower levels, there was little difference between their %over run results. The highest value obtained was found in Experiment 5, in which the maximum levels of emulsifier concentration and stirring time were used.

According to the Pareto Chart (Figure 3) of the correlation between the air incorporation capacity and the independent variables foaming concentration (C) and stirring time (T_{ag}), it is observed that only the concentration is statistically significant ($p < 0.05$). The air incorporation capacity of the foam follows the opposite tendency to the density, so that to achieve greater and more efficient foaming activity, the best conditions will be those with higher values. The generated model had a percentage of explained variation of 69.5% and can be represented by Equation 17.

$$\%over\ run = 610.30 + 171.66C \quad (17)$$

A difficulty previously experienced with this process and reported by Karim and Cheewai (1999) is the lack of stability of the foam during the heating cycle, as if the foam does not remain stable cell destruction will occur, causing serious deterioration of the operation. According to Cruz (2013), the stability of the foam is of fundamental importance for the success of drying in a foam layer, as well as for the quality of the product, having a directly proportional relation to the addition of foam stabilization agents.

As it can be seen in Figure 4, the stability test curves showed a similar behavior, even with temperature variation. For 50 ºC, the maximum loss of volume was 6.6 mL, while for the 70ºC, the volume released reached 11.4 mL. In both tests, the foams that presented the highest volume release were those with the foaming concentration at the lowest level (3%), with little dependence on the variation in agitation time values. The results indicate that the foam formed with the synthetic emulsifier is destabilized as drying temperature increases.

According to Kandasamy *et al*. (2012), the stability is influenced by the density, thickness and permeability of the liquid-foaming agent interface, by the distribution of the size of the air bubbles and by the surface tension; however, the nature and concentration of the agent used is one of the

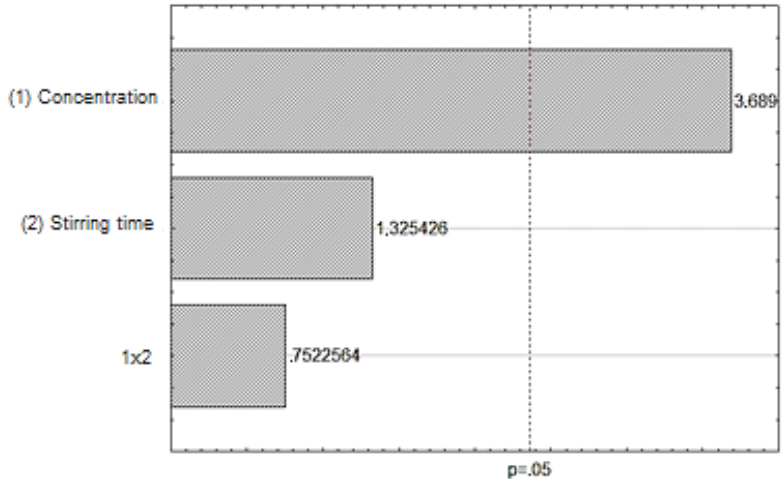

Figure 3. Pareto's Chart: Effect of foaming concentration and stirring time on air incorporation capacity of *Mentha crispa* foam

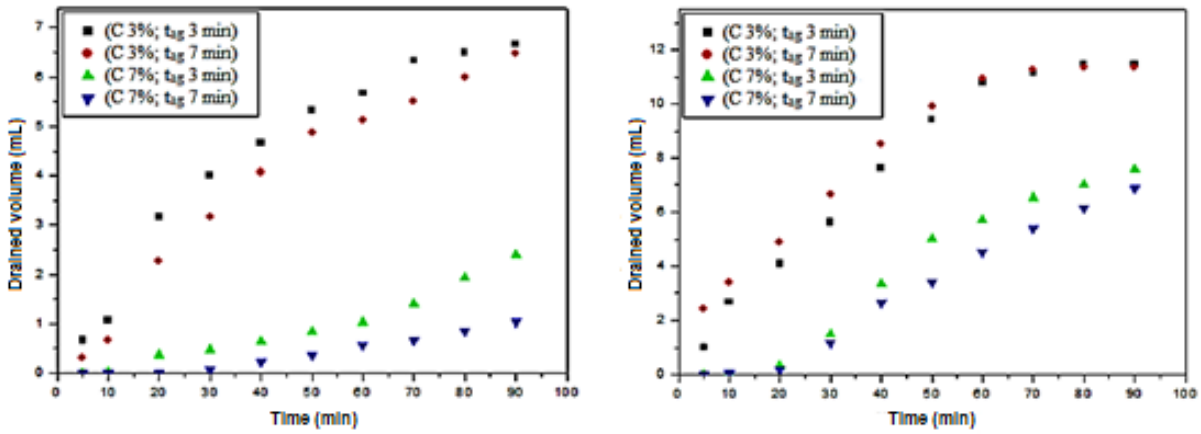

Figure 4. *Mentha crispa* foam stability tests 50 and 70°C

main factors that alter this property. Other factors that may have negatively affected the stability of the foam and should be taken into account in view of this behavior are the high thickness of the foam layer and the low surface area exposed to the drying air during the experiment. Bag *et al*. (2011) also reported that the collapse of foams can also occur due to the force of gravity acting on the foam due to the increase in volume, which results in the fusion of small air bubbles.

The curves of *Mentha crispa* drying kinetics are shown in Figure 5. Data are displayed in the form of dimensionless moisture (RX) as a function of drying time (min) for the temperatures set by the experimental design of 50, 60 and 70 °C respectively.

As it can be seen, the drying time varied according to the temperature used in the experiment, in such a way that the rise in the temperature significantly reduced the time. It is observed that in the experiments submitted to drying at 70°C, the equilibrium humidity was reached after 80 minutes, while at 50°C, only after 180 minutes. Drying occurred quickly and this fact is naturally explained by the fact that *Mentha crispa* is rich in essential oils that evaporate easily the higher the temperature used in the experiment (MATOS, 2002). Furthermore, it is known that higher temperatures lead to greater heat transfer between the foam layer and the air and, consequently, greater evaporation of water from the product, resulting in a reduction in the water content and operating time.

In the work of drying kinetics, most authors mention that food drying has a decreasing rate period, which may or may not be preceded by a constant rate period (FELLOWS, 2006; DANTAS, 2010). It can be seen in the drying kinetics data shown in Figure 5 that the presence of a short heating period in the initial drying stage and in sequence, a constant rate period, with a very short decreasing rate period. In the constant rate period, water is freely available around the solid matrices and can easily be transported by capillary flow and vapor diffusion. The water vapor pressure at the surface during this period is constant and equal to the vapor pressure of pure water at product temperature. The product temperature is also constant and equal to the wet bulb temperature, typical of the fact that heat and mass transfers are compensated, causing a constant drying rate (PARK *et al*., 2014).

The absence of the period of constant rate in drying can occur due to the characteristic of the moisture because even with free surface humidity, the water can be in the form of suspension of cells and solution (sugars and other molecules), presenting a vapor pressure below of pure water vapor pressure (SILVA *et al*., 2008).

Other authors, when working with different methods of drying the plant species, also found similar results. Pachú (2007) observed a similar behavior in the curves of drying kinetics of *Mentha crispa* at 60 and 70°C, obtained under

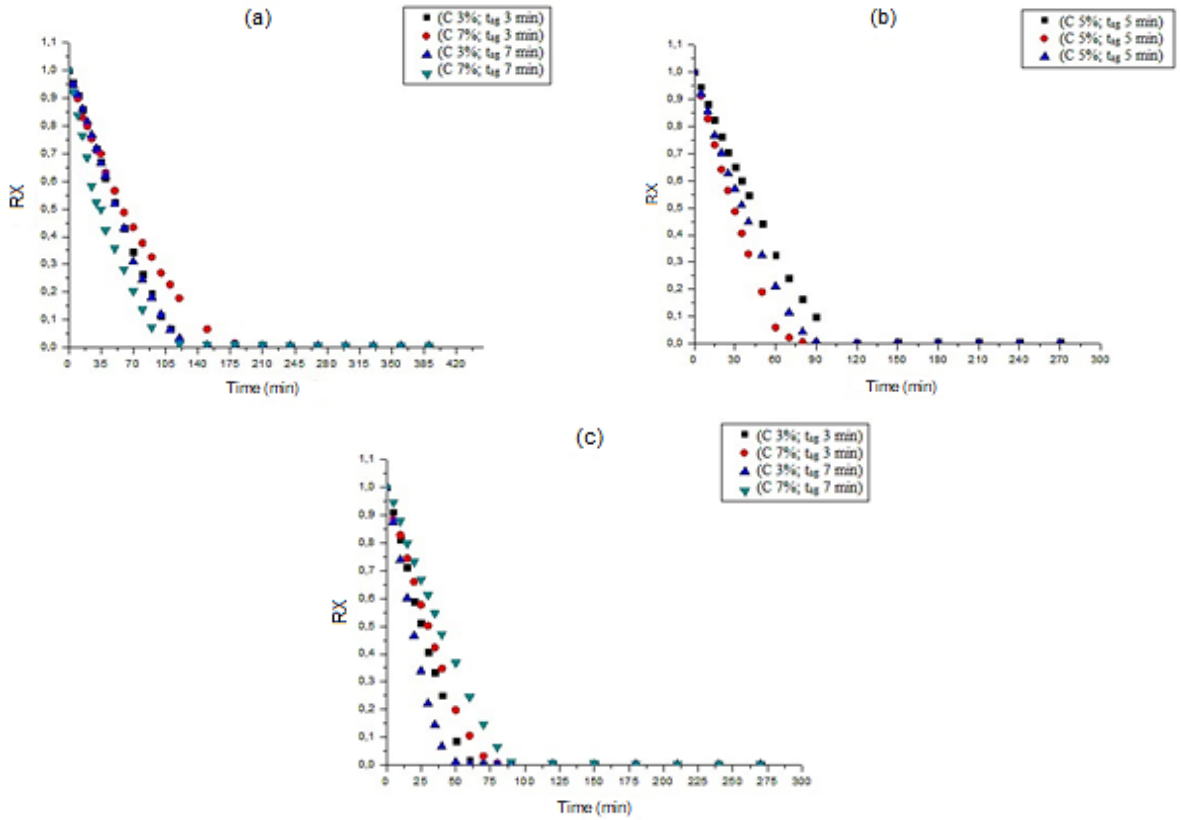

Figure 5. *Mentha crispa* drying curves at (A) 50, (B) 60 and (C) 70°C

conditions of forced convection through a thin layer of fresh leaves. Fernandes *et al*. (2013) determined the behavior of drying mint in an oven with air circulation and obtained drying times of 3 h for a temperature of 45°C, 2 h for a temperature of 55°C and 1 h and 15 min for a temperature of 65 °C, showing that the drying of the leaves is influenced by the drying temperature, with drying occurring in a shorter time at the higher temperature. Gasparin *et al*. (2017) also found that the drying kinetics of peppermint (*Mentha piperita*) in a fixed bed was strongly influenced by temperature and that the loss of moisture content is faster at the beginning of the drying process, a behavior demonstrated by the lack of a constant drying period.

With regard to the technique, Thuwapanichayanan *et al*. (2008) also reported the predominant occurrence of constant rate period in foam layer drying of mashed banana, with higher drying rates at higher drying air temperatures and lower foam densities. Other authors have also described this behavior in foam drying of tamarind pulp (SILVA *et al*., 2008), pulp of mango cv. Haden (SILVA FILHO *et al*., 2016), soursop tree leaf (OLIVEIRA *et al*., 2020) and umbu pulp (SOUZA *et al*., 2021).

Figure 6 shows graphs of the drying rate as a function of water content. It is observed that the temperature of 70°C presented a higher drying rate in relation to the other two treatments, showing the influence of temperature on the rate. According to Fernandes et al. (2013), the effect of temperature is caused by its influence on the potential for water transfer from the solid to the drying air, given that heating the air to higher temperatures implies a reduction in its relative humidity, directly affecting the potential mass transfer and may also change the physical properties.

Another fact that can be observed in the curves is the presence of a short period of decreasing rate, between the range from 0.0 to 15 of the final moisture, opposite to the existence of a long period of constant rate during the course of the drying process, between 15 and 90.

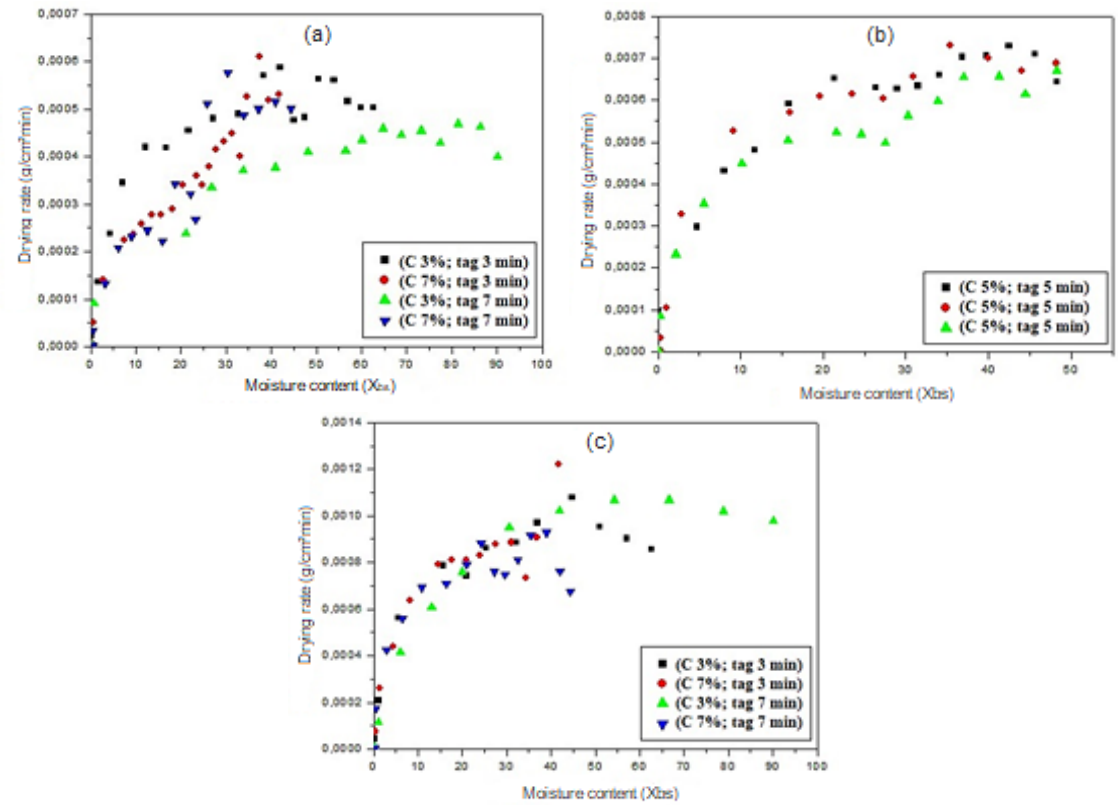

Figure 6. Drying rate curves according to moisture content (X_{bs}) to (a) 50, (b) 60 and (c) 70°C

This observation only confirms the behavior previously shown by the drying kinetics curves in a layer of *Mentha crispa* foam. According to Martins *et al*. (2020), the constant drying rate stage persists until the moisture inside the solid is very low, decreasing the diffusion of water to the surface of the solid. The reduction in the drying rate observed in both graphs occurs because the amount of energy needed to vaporize the water in this period is lower, and the solid starts to increase its temperature. Thus, drying progresses to a limit, represented by the equilibrium moisture content.

The adjustment of the mathematical models to the experimental data was carried out using statistical analysis as a model selection criterion, in case the model presents statistically significant coefficients, in addition to verifying satisfactory adjustment, through the determination coefficient (R^2), standard error of the estimate (S), mean square error (MSE) and chi-square test (χ^2). The results of the adjustments of the non-linear models are shown in Table 5 for each of the temperature ranges used in the drying kinetics. The models satisfactorily adjusted to the experimental drying data, showing a coefficient of determination greater than 0.9566 and a chi-square function with values in the order of 10−2 and 10−3, which indicates adequate model accuracy and high correlation between experimental and predicted (SANTOS et al., 2022), with the exception of the Midilli and Kucuk model, which did not present statistically significant coefficients ($p < 0.05$) for any of the assessed temperatures, and the Modified Page model for drying at 70°C.

For the drying temperatures of 50 and 60ºC, the models that had the best adjustment to the experimental data and the lowest standard error of the estimate were the Modified Page and Page models. When investigating the drying of sugarcane juice in a foam layer (MARQUES, 2009), fruit pulp (DANTAS, 2010), jackfruit pulp (DANTAS et al., 2008) and red rose apple (RIGUETO et al., 2020) and drying starfruit in a tray dryer (SANTOS et al., 2010), several authors also found better fits and high coefficients of determination for Page's model.

Table 5. Adjustment of drying *Mentha crispa* foam drying kinetics models

Model	Parameter							
	T (ºC)	k	n	a	R^2	S	MSE	χ^2
Page	50	0.0058399	1.23767		0.9866	0.3383	0.005703	0.00338
	60	0.0049887	1.40342		0.9866	0.2103	0.007279	0.00345
	70	0.008386	1.35365		0.9645	0.6408	0.01082	0.0089
Henderson & Pabis	50	0.01640		1.0518	0.9839	0.4074	0.006258	0.00407
	60	0.02455		1.07669	0.9797	0.3171	0.008939	0.00520
	70	0.03140		1.0724	0.9591	0.7356	0.01159	0.01022
Midilli & Kucuk	50				ns			
	60				ns			
	70				ns			
Newton	50	0.01535			0.9825	0.4423	0.00652	0.00438
	60	0.02251			0.9768	0.3615	0.009545	0.00583
	70	0.02903			0.9566	0.7801	0.01194	0.01069
Modified Page	50	0.004486	1.2940	0.9772	0.9868	0.3345	0.005671	0.003379
	60	0.00342	1.4920	0.9687	0.9869	0.205	0.007195	0.003425
	70				ns			

ns = not significant

The adjustments for the Henderson & Pabis and Newton models were also satisfactory and had determination coefficients greater than 0.9768, but presented higher values of estimation error, a fact that can be explained by the lack of adjustment to the experimental data in the final period of the drying, close to reaching equilibrium moisture, when compared to the other two models. The particular characteristic of the behavior of the drying curves of the *Mentha crispa* obtained in the absence of a well-defined period of decreasing rate resulted in a fit of the models with relatively high estimation errors. The constant rate period is characterized by equality between the mass transfer rate inside the solid or semi-liquid and the water evaporation rate on its surface, resulting in linearity in the drying kinetics curves.

The results are consistent with those determined by Silva *et al*. (2008), Jabs (2013) and Oliveira (2017), whose determination coefficients were higher for the Page equation when compared to the Henderson & Pabis and Newton models. In the work carried out by Silva (2015), among the models that presented good adjustments to the experimental data, the Page model was selected to represent the phenomenon of drying passion fruit pulp in a foam layer due to its simplicity of application and because, traditionally, it is recommended and applied to predict the phenomenon of drying of various agricultural products.

For the temperature of 70 ºC, the Page model was the one that best adjusted to the behavior of the drying curves. Nevertheless, all the presented models was found not be satisfactorily adjusted to the experimental data, with coefficients of determination lower than 0.97 and high standard error values of the estimate. Similar to the other adjustments, the high standard error of the estimate can be attributed to the behavior of the drying kinetics curves, which show a significant constant rate and an almost imperceptible period of decreasing rate, not properly adjusting to the non-linear mathematical models.

Applications of mathematical models for drying curves are important to describe how moisture transfer occurs inside the solid to evaporate on its surface and which transfer mechanism controls the process (Pereira, 2015). According to Misra and Brooker (1980), parameter k represents the effect of external drying conditions, while parameter n represents the product's internal resistance to drying. In all evaluated models, it is observed that the numerical value of k is considerably negligible if compared to the n parameter, thus demonstrating that drying is a process totally dependent on the internal resistance of the product to drying. It

seems that the parameter k increased as drying temperature increased for the Henderson & Pabis and Newton models, a behavior also observed for the Henderson & Pabis model by Fernandes et al. (2013) when working with drying *Mentha crispa* in a fixed bed (45, 55 and 65 ºC) and by Kaya *et al*. (2007), in apple drying (35, 45 and 55 °C).

It was observed in the statistical analysis of the process that, for the drying time response, with the exception of the drying temperature, the other parameters of the model were not statistically significant. The temperature had a negative influence on the response variable, so that the lower the temperature, the longer the drying time. From the statistical analysis, it was possible to generate a model, through linear regression of the experimental data of the process, with the input variable that was statistically significant ($p < 0.05$). This model had a coefficient of determination of 0.9395 or percentage of explained variation of 93.95%, and it can be represented by Equation 18.

$$t_s = 176.364 - 122.5T \quad (18)$$

When carrying out the variance analysis of the data for the drying time variable, it is observed that the represented model is statistically significant as, through the F Test, it can be seen that the value of $F_{calculated}$ is greater than the F_{tabled}, 62.19 > 4 .46, considering the confidence level and degrees of freedom for the regression and the residual. In addition, still based on Test F, it is observed that the model also does not present a lack of fit, since the same test also considers the confidence level and the degrees of freedom of the lack of fit and pure error, revealing that the calculated F value is smaller than the tabulated F, 0.0666 < 19.33, which characterizes a model with no lack of fit.

Figure 7 shows the response surfaces that show the comparison between the independent variables of the process in relation to the dependent variable drying time.

The influence of stirring time on drying time was minimal, however, even though the variable did not have a significant effect on the response, drying time tended to decrease at lower levels of stirring time. The concentration of foaming agent showed a positive influence, so that, as its levels increase, the values of the drying time also increase. Therefore, it is possible to observe that the drying operation must be carried out at its minimum values, which would promote a reduction in production costs.

The analysis on the water content showed that, with the exception of the emulsifier concentration and the interactions involving the drying temperature, the other parameters of the model were significant. It was found that the temperature exerts a negative sign influence on the response variable, so that the lower the temperature, the greater the water content, while the stirring time exerts a positive sign influence on the response variable, so that the larger it is, the higher the water content.

From the statistical analysis it was possible to obtain a model, through linear regression of the experimental data of the drying process, with the input variables and the interaction that were statistically significant ($p < 0.05$). This model had a coefficient of determination of 0.7049, or percentage of explained variation of 70.49%, and a

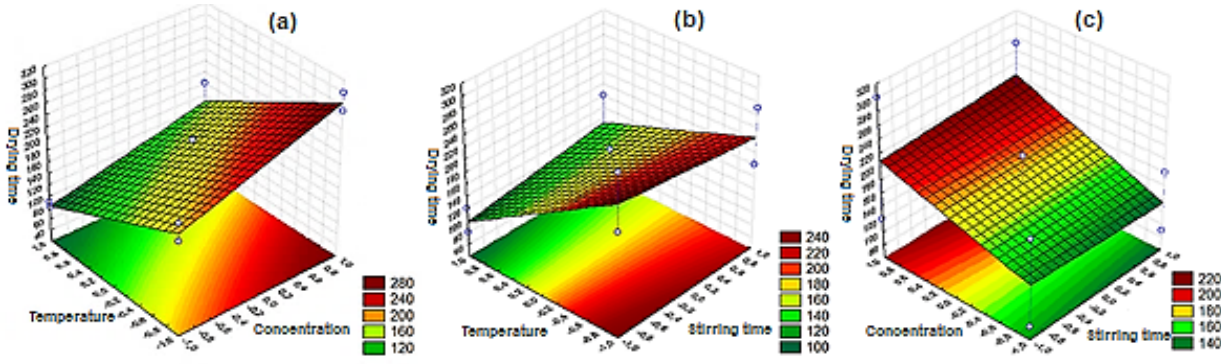

Figure 7. Response surfaces for the drying time variable, keeping the following fixed on average levels: a) stirring time; b) foaming agent concentration; c) temperature

standard error of 0.0007182. In addition, it can be represented by Equation 19.

$$X_{bs} = 0.144707 + 0.153443t_{ag} - 0.094878T - 0.096533t_{ag}\,C \quad (19)$$

The variance analysis of the data for the water content Variable showed that the represented model is statistically significant, since through the F Test it is verified that the value of $F_{calculated}$ is greater than the F_{tabled}, 5.57 > 4 .35, considering the confidence level and degrees of freedom for the regression and the residual. Also, based on the F Test, it was observed that the model does not present a lack of fit as the same test also considers the confidence level and the degrees of freedom of the lack of fit and pure error, revealing that the value of $F_{calculated}$ is smaller than the F_{tabled}, 10.25 < 19.30. However, due to the low value of the coefficient of determination (R^2), it must be concluded that there is a lack of fit.

Figure 8 shows the response surfaces that show the comparison between the independent variables of the process in relation to the dependent variable water content. When analyzing Figure 8a, a greater dependence of the water content on the drying temperature is observed, so that throughout the working range of the concentration there was no significant variation in the water content. It can be seen that the lowest water content of the product is found at the upper level of temperature and lower level of concentration. This observation provides information that the variable emulsifier concentration can be used at its lowest levels with no negative effect on the drying operation and the quality of the final product.

It can be seen in Figure 8c the influence of the interaction between the two independent variables on the response, in addition to a demonstration of greater dependence of the final moisture on the stirring time, observed through the variation along its working range. Both variables have a positive influence, so that the lowest water contents are found at the lowest levels of stirring time and emulsifier concentration. Based on the results of response surfaces for final moisture, it is observed that the influence of the positive sign of stirring time may have caused a masking of the results of estimating the effect of the other variables. It is possible to suggest that the longer agitation times resulted in a mechanically fragile interfacial film of the foam, so that the stability of the air bubbles in its structure was negatively affected and impaired the removal of moisture through the capillarity of the pores during drying.

CONCLUSIONS

- The density, percentage of expansion, air incorporation capacity and stability of the *Mentha crispa* foam depend only on the concentration of the foaming agent, given that the stirring time did not have a significant influence on the evaluated conditions.
- The rise in the temperature significantly reduces the drying time and the water content of the mint foam. The kinetics and drying rate curves have a behavior with the presence of a long period of constant rate and almost non-existence of decreasing rate. The nonlinear

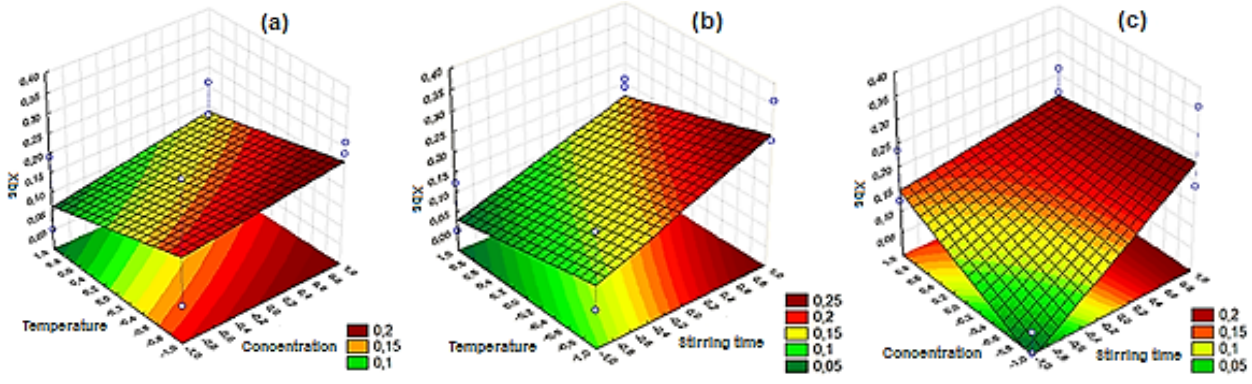

Figure 8. Response surfaces for the dry base water content, keeping the following fixed on average levels: a) stirring time; b) concentration of foaming agent; c) temperature

mathematical models of Page, Modified Page, Newton and Henderson & Pabis present statistically significant coefficients, where the Page model is the one that best fits the experimental data and, therefore, the most suitable to represent the phenomenon of drying in a foam layer of *Mentha crispa*.

- The best drying condition occurs when working with at 70°C, 3% concentration of foaming agent and stirring time of 3 min, resulting in a drying time of 80 min and water content of 0.9%.

AUTHORSHIP CONTRIBUTION STATEMENT

LEITE, A.C.N.: Formal Analysis, Investigation, Methodology, Writing – original draft, Writing – review & editing; **CAVALCANTE, J.A.:** Conceptualization, Methodology, Supervision, Validation, Writing – original draft; **COSTA, N.A.:** Conceptualization, Formal Analysis, Software, Supervision, Validation; **PINHEIRO, W.S.:** Formal Analysis, Investigation.

DECLARATION OF INTERESTS

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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