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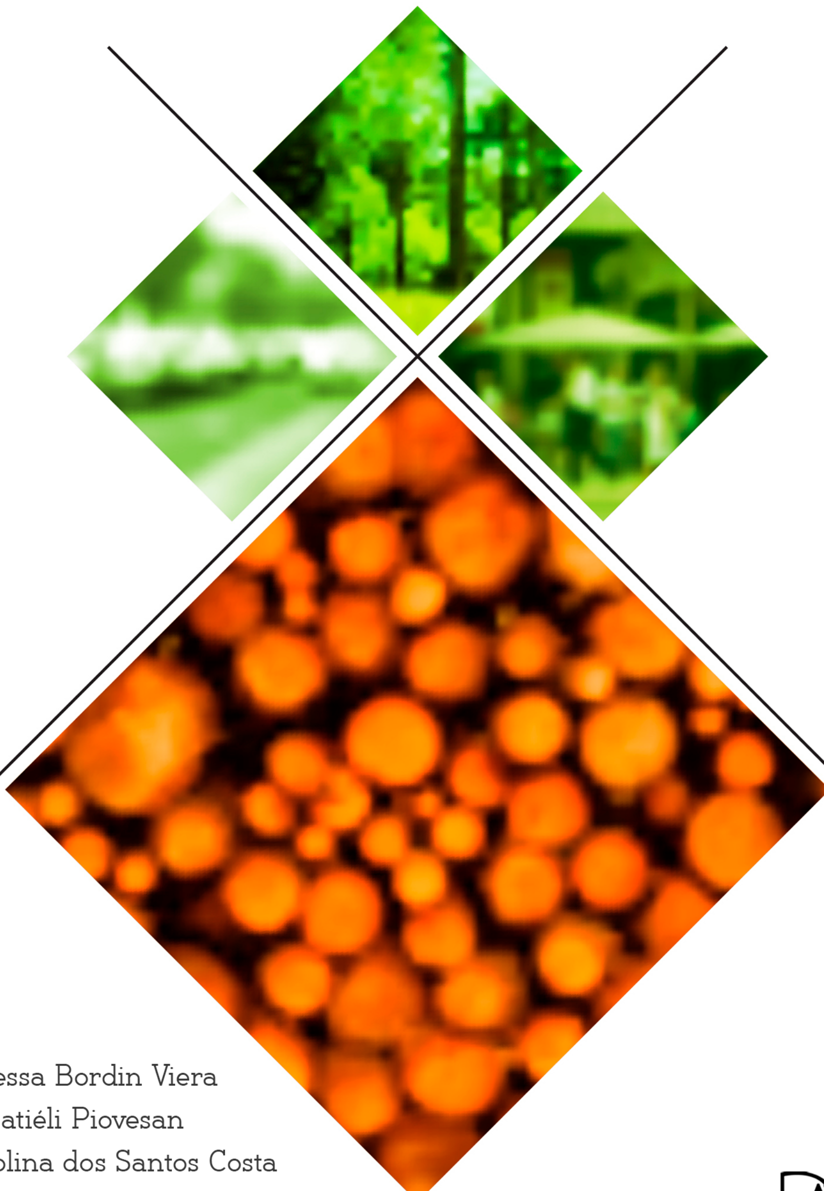

Vanessa Bordin Viera
Natiéli Piovesan
Ana Carolina dos Santos Costa
(Organizadoras)

Atena Editora
Ano 2020

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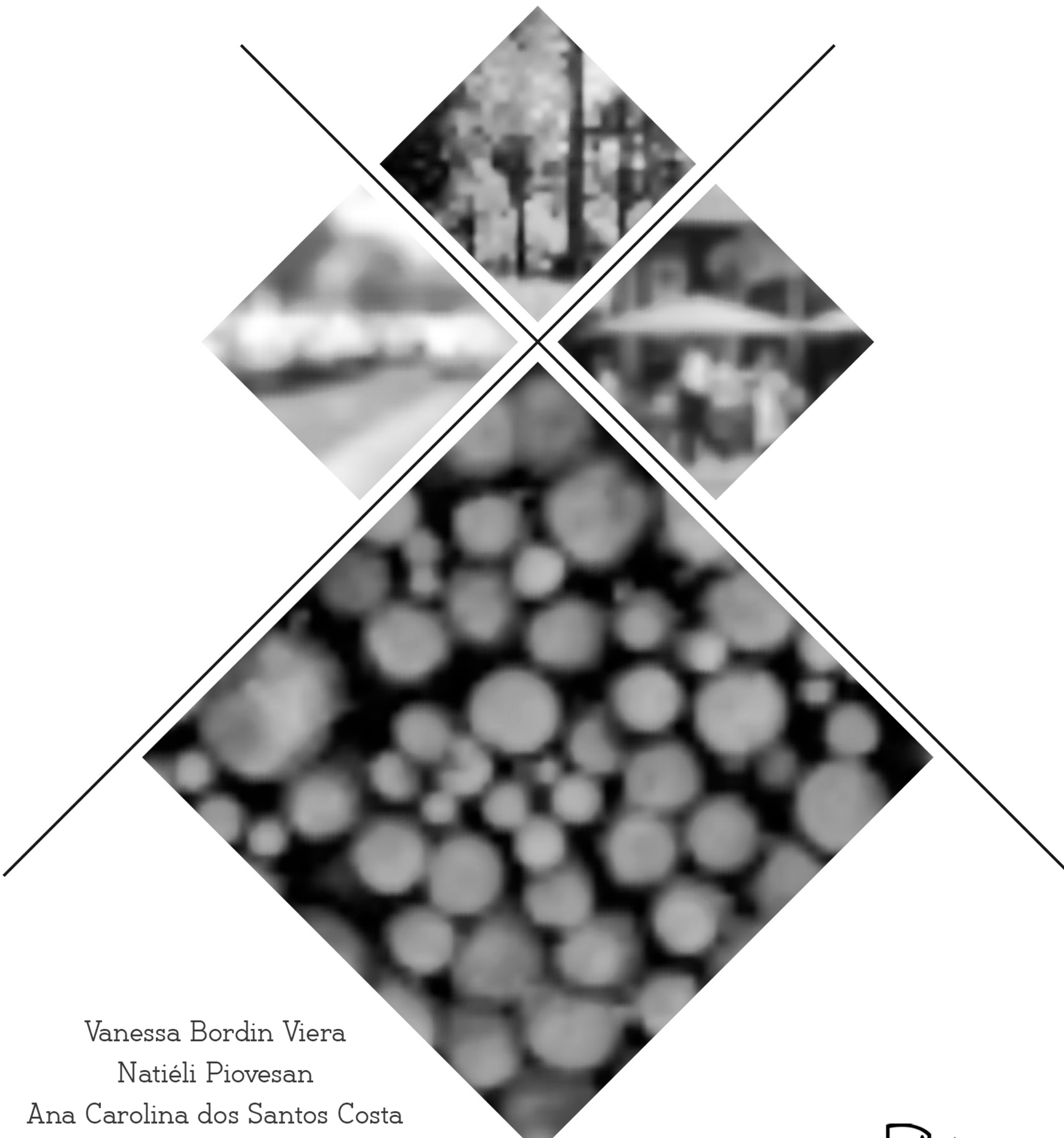

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Investigação científica no campo da engenharia e da tecnologia de alimentos

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APRESENTAÇÃO

O *e-book* “Investigação Científica no Campo da Engenharia e da Tecnologia de Alimentos” está recheado com 22 artigos científicos com uma vasta temática, como desenvolvimento de novos produtos, análise sensorial de alimentos, análises microbiológicas, modelagem matemática na secagem de alimentos, validação de métodos, entre outros. Os artigos são atuais e trazem assuntos relevantes da área de Engenharia e Ciência e Tecnologia de Alimentos, contribuindo para a ampliação do conhecimento dos leitores na área.

Convidamos os leitores para conhecer e se atualizar através da leitura desse e-book. Por fim, desejamos a todos uma excelente leitura!

Vanessa Bordin Viera

Natiéli Piovesan

Ana Carolina dos Santos Costa

SUMÁRIO

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CAPÍTULO 20

THERMOGRAVIMETRIC DETERMINATION OF MOISTURE IN GLUCOSE AND CANE SYRUPS USING FIBERGLASS PAPER

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ABSTRACT: Food high in sugar content such as sugar cane and glucose syrups is used almost all over the world. In this research, a method was developed to determine the water amount in cane and glucose syrups by TG / DTA, and also by a heating rate optimization study and use of a fiber-glass paper in alumina crucible using experimental planning. The method was verified using accurate and precise estimates. Results of the optimization indicated that the utilization of the lower heating rates (5 °C min-1) and the use of a glass-fiber paper in alumina crucible improve the efficiency of sample dehydration. In addition to good precision and accuracy calculations, the procedure does not demand reagents or toxic solvents which are required in other methods of determination of moisture content, dispenses sample treatment and allows the identification of the punctual elimination temperature of the water, an advantage that is remarkable when compared to other thermal degradation processes of volatiles.

KEYWORDS: Development, Water Content, Foods Rich in Sugar, TG/DTA.

DETERMINAÇÃO TERMOGRAVIMÉTRICA DE UMIDADE EM XAROPES DE GLICOSE E MELADO DE CANA DE AÇÚCAR USANDO PAPEL DE FIBRA DE VIDRO

RESUMO: Alimentos com elevado teor de açúcar, como o melado de cana-de-açúcar e xaropes de glicose, são praticamente usados em todas as partes do mundo. Nesta pesquisa, foi desenvolvido um método para a determinação da quantidade de água nesses xaropes por TG / DTA, através de um planejamento experimental variando a taxa de aquecimento e a utilização de papel de fibra de vidro em cadinho de alumina. O método foi validado por meio das estimativas

de exatidão e precisão. Os resultados da otimização indicaram que a utilização das menores taxas de aquecimento (5 ° C min^{-1}) e o uso de papel de fibra de vidro no cadinho de alumina melhoram a eficiência da desidratação da amostra. Além da boa exatidão e precisão do método, o procedimento proposto não exige uso de reagentes tóxicos que normalmente são usados em outros métodos de determinação de umidade, dispensa o tratamento da amostra e ainda permite a identificação da temperatura de eliminação pontual da água, uma vantagem que é notável quando comparado a outros processos de degradação térmica de voláteis.

PALAVRAS-CHAVE: Desenvolvimento, Teor de água, Alimentos ricos em açúcar, TG / DTA.

1 I INTRODUCTION

Water is one of the main constituents of food products. The amount in food widely varies, such as, for instance: brown sugar (1.3 %), honey (17.1 %), milk (89.3 %), yogurt (85.1 %) and ice cream (61.0 %). The ease of removing water depends on how it exists in the food product (free water, adsorbed water, and water of hydration)(Nielsen, 2010). Food high in sugars present strong interactions with water due to the polarity of the substances present. To separate the water, higher temperatures are necessary and may cause decomposition of the food (Heinze and Isengard, 2001). The water volume in a portion of food is the main factor contributing to its deterioration by microorganisms and changes through chemical and enzymatic reactions (Passos *et al*., 2013). Knowing that the water quantity in food rich in sugars such as honey, cane, and other syrups, is of fundamental importance in conservation, storage, maintenance of the quality and marketing of these processed products (Celestino, 2010); drying processes play an essential role in food technology due to increased requirements on the marketed product quality (Heinze and Isengard, 2001; Isengard, 2001; Vlachos and Karapantsios, 2000; De Caro *et al*., 2002; Mathlouthi, 2001; Knochel *et al,* 2001; Isengard and Heinze, 2003). The technique used to avaliated the water amount in food will be debated as well the way water is present in it. To better control and optimize the drying methods, several analytical methods have been developed to evaluate the moisture content in food with greater reliability. Heinze and Isengard (2001) determined of the amount of water in sugar syrups by drying with halogen. Vlachos and Karapantsios (2000), quantificated water in starch-based products using conductance techniques; Isengard and Heinze (2003) used the Karl Fischer technique to evaluate total and surface water in sugars. The determination of moisture in cane and glucose syrups is usually carried out in a vacuum oven at 70 °C (Nogueira *et al*., 2009). This method present the disadvantage of determines the total water content in the food, i.e. water together with other volatile substances which are present in cane and syrups and may evaporate along with the water, decomposing or partially oxidizing the sample during heating. One of the techniques that allow the observation of mass loss during homogeneous heating of a sample is thermogravimetry (TG). The differential thermal analysis (DTA) is generally used together with TG to obtain analyte information through temperature change to identify phase transitions without mass variation (Haines, 2002). TG / DTA has been used as a precise method in the detection of moisture content in various food products (Acquistucci *et al*., 1991; Ducat *et al*., 2015; Silva *et al*, 2008 ; Tomassetti *et al*., 1989). It has been rarely used, however, for foods with high sugar content (Silva *et al*., 2008; Felsner, 2001); since they show intense bubbling and spattering effects during heating hindering the full output of the water molecules (Felsner *et al*., 2004). The crusting layers in the sugar burning also prevent the efficient removal of water from these foods. With that, the objective of this work was to optimize a thermogravimetric system to evaluate the amount of water in the cane and syrups in a dynamic air atmosphere, using a fiberglass paper to eliminate the unwanted effects during the drying mentioned above. The optimization was performed by using factorial design and a method validation by applying statistical analysis.

2 I MATERIALS AND METHODS

2.1 Samples

In this study, were used, twelve samples, six cane syrups (from Brazil and Canada) designated samples C1 to C6, and six glucose syrups (from Brazil and Canada) designated samples G1 to G6. The samples came from different manufacturers and lots and maintained in its original packaging until the analysis and stored in a cool dry place after opening them.

2.2 Experimental methods

2.2.1 Thermogravimetry (TG/DTA)

For determination of the moisture contents in molasses and syrup samples the simultaneous TG / DTA was used (Seiko Exstar 6000). Synthetic air (50.0 mL / min-1) was also used to obtain a constant dynamic atmosphere during the temperature rise (ambient to 200 °C). Alumina crucible (50 µL) was used for the samples (3.0 mg) as a reference for performing the procedure. Two factors (heating rate and a fiberglass paper) and two levels (5 and 10 °C min-1, without and with fiberglass paper) of the factorial design were applied for water contents analysis in cane and glucose syrups samples by thermogravimetry.

2.2.2 Karl Fischer titration

The results obtained with the proposed method in determining the water contents using TG / DTA were compared with Karl Fischer's volumetric titration (reference Method 014 / IV; (IAL, 2008). Used titrant equipment of the brand Quimis (Q349-1), Karl Fischer reagent without pyridine (Biotec, Brazil) and anhydrous methanol (JT Baker, USA).

2.2.3 Physicochemical Analyses

The physicochemical analyses were performed on the samples: cane and glucose syrups (reducing sugars, pH, HMF (5- (hydroxymethyl) 2-furfural), Brix and acidity) according to the methods indicated for Physicochemical Methods for Food Analysis (IAL, 2008). The conductometric ashes and enzymic browning (absorbance measurement at 420 nm) were performed according to the method indicated by ICUMSA (2007). The final drying temperature (Tfd) of the samples was determined by the thermogravimetric drying curve.

2.2.4 Optimization study

A 2^2 factorial design was applied to evaluate the variables rates of heating and use of fiberglass paper in the alumina crucible on the moisture contents of the samples of cane and glucose syrups. The experimental design was done in a random way and on duplicates. Equation 1 presents the calculation of factor effects.

$$E_f = (R_+) - (R_-) \qquad \text{Equation (1)}$$

where (R_+) and (R_-) are the means at the (+) and (-) levels of the factors.

Statistical calculations (at 95% confidence level) were applied to the results obtained in the experimental design. All the statistical data analyses were carried out using the software Minitab for Windows version 16.2.2 (MINITAB, 2010).

2.2.5 Validation Studies

This study was validated using the precision and accuracy parameters according to AOAC (Wernimont, 1995; INMETRO, 2018). These parameters were used to compare the results of the thermogravimetric method developed with the Karl Fischer values (reference method). To assess the accuracy of thermogravimetry, a paired t test was applied for the mean differences in water content obtained by analytical methods and an appropriate F test to calculate confidence intervals for the ratio between the variations and weighted standard deviations (Sp) (Miller and Miller, 2002; Wernimont, 1995; INMETRO, 2018). All statistical analyses were performed at a 95% confidence level by using the software Minitab for Windows version 16.2.2 (MINITAB, 2010) .

2.2.6 Principal Component Analysis of Physicochemical Characteristics in Samples

The principal component analysis (PCA) was applied to experimental data, to check the possible similarities and differences between the analyzed samples and the correlation between variables. In interpreting the data obtained from the physicochemical analysis of cane and glucose syrups, the variables used in the statistical analysis were the reducing sugars, ash conductimetric, water content, acidity, HMF, T*fd*, ° Brix, pH and of browning degree.

3 I RESULTS AND DISCUSSION

3.1 Physicochemical parameters

The principal component analysis was applied to the experimental data obtained in the determination of the physicochemical parameters of sugar cane and glucose syrups to check for possible similarities and/or differences between them, as well as the correlation between the variables. To explain the differences found between the samples, the levels of reducing sugars, organic acids (expressed as acid), Brix, pH, ash, Tfd, browning degree, and HMF were determined (Table 1).

Samples	Brix	pH	Ash (g/100 g)	Reducing Sugar (g/100 g)	Degree Browning (nm)	Acidity (meq g^{-1})	Tfd ^oC	HMF (mg kg^{-1})
C1	76.32	4.60	1.49	66.61	1.420	52.4	147	536.4
C2	79.98	4.06	0.49	33.56	0.203	41.2	130	115.7
C3	82.83	3.65	0.42	54.44	0.323	60.4	120	893.1
C4	82.33	5.28	0.95	33.33	0.955	35.7	135	109.2
C5	82.68	4.30	1.36	45.67	0.920	80.1	125	593.3
C6	79.57	5.34	2.34	40.64	3.125	61.5	128	20.90
G1	82.88	4.35	0.44	60.74	0.194	3.8	137	1655.9
G2	79.30	4.37	0.32	59.45	0.210	5.3	147	1086.9
G3	84.68	2.65	0.55	64.72	0.140	45.0	138	1,627.9
G4	79.32	5.00	0.47	40.32	0.090	1.4	140	50.3
G5	67.38	5.89	0.20	0.00	0.060	2.8	135	0.0
G6	77.82	3.72	0.39	37.87	0.002	2.9	130	30.7

Table 1 Data on the chemical composition of cane and glucose syrups samples expressed as mean values.

Figure 1 shows the weights of the 1st (43.15 %) and 2nd (30.79 %) about the main components variables, totalizing 73.94 % of the total variability of the sample. It can be observed that the moisture, TFS, pH, and browning degree variables are inversely correlated with the other variables by Factor 1. HMF and reducing sugars variables were positively correlated with each other (Factor 1): the higher the reducing sugar contents the greater their contribution to the formation of HMF. The negative correlation between pH and HMF parameters is due to the favoring of HMF formation in acidic media by sucrose degradation. Moisture also showed a negative correlation with the HMF, because when are lower the moisture contents of cane and glucose syrups, the higher will be the content of

HMF contaminant in foods. There was a positive correlation by Factor 2 of the PCA between the variables moisture, HMF, TSF and reducing sugars in the negative axis. These variables were separated into two distinct groups: samples of cane (C1 to C6) are positioned in the positive axis of PCA2, while samples glucose syrups (G1 to G6) were located in the negative axis in the shaft. It can be seen that all cane syrup samples showed lower moisture content, and higher ash content, browning degree, acidity, and pH.

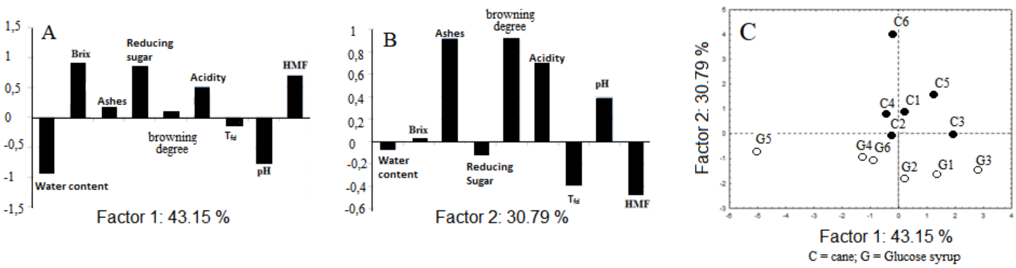

Figure 1 Weights of the 1st principal component (A), 2nd (B) and separation of samples (C) defined by principal components analysis.

3.2 Optimization Study of Thermogravimetric Method

To optimize the thermogravimetric method was applied to an experimental design 2^2 to investigate the influence of the heating rate and a glass fiber paper in the water contents of cane and glucose syrups. The sixteen TG/DTA curves showed a similar thermal drying profile for the water removal step. The results of the factorial design and the calculated effects are shown in Table 2.

Test	Heating rate °C.min^{-1}	Glass fiber paper	Water contents averages (g/100g) ± SD for Cane	Water contents averages (g/100g) ± SD for Glucose
1	5	without	21.78 ± 0.13	14.00 ± 0.42
2	10	without	16.30 ± 0.42	4.44 ± 0.14
3	5	with	20.57 ± 0.09	15.25 ± 0.04
4	10	with	22.68 ± 0.05	16.08 ± 0.23
Effects			Estimate ± SD Cane	Estimate ± SD Glucose
Global mean			20.34 ± 0.04	12.46 ± 0.04
Main effects: Heating rate (1) Glass fiber paper (2)			-1.67 ± 0.08 2.57 ± 0.08	-4.39 ± 0.08 6.43 ± 0.08
Effects of second order interactions : (1) x (2)			3.78 ± 0.08	5.22 ± 0.08

Table 2 Results, effects and standard errors calculated for the water contents for the cane and glucose syrups by thermogravimetry by factorial design 22.

From the analysis of *t*-test at a 95 % confidence level, the factorial design variables (heating rate and fiberglass paper) were considered significant for the two investigated foods, and an important effect of interaction between these was observed (Table 2). This means that the variables heating rate and fiberglass paper cannot be optimized independently. Change of heating rate level from 5 °C min-1 to 10 °C min-1 resulted in an average decrease of water contents of approximately 1.67 % for cane and 4.39 % for glucose syrup (Table 2). A very rapid heating rate results in an uneven distribution of temperatures over the sample, which results in the elimination of incomplete moisture. This is a consequence of the resulting contraction of the release of water. A possible cause of the contraction is the case hardening (formation of a hard and almost impermeable film on the surface of the product), particularly common in foods with a high concentration of dissolved sugars and other solutes such as cane and glucose syrups. Solutes move from center to surface of the product with the water; the surface water evaporates and the solute is deposited, closing the pores. The combination of the contraction with the closing of the pores prevents the migration of most of the moisture remaining inside, resulting in non-uniform drying and a decrease in drying rates. They may also be coupled with dehydration, browning reactions, particularly the Maillard reaction and caramelization and loss of volatiles. An existing problem in the separation of moisture in foods rich in sugars is the decomposition of existing constituents and the release of water. For example, carbohydrates decompose to 100 ° C forming carbon and water. This water generated in the decomposition of carbohydrates is not the moisture we want to measure (Nielsen, 2010). Due to these results, it is suggested that heating rates lower are most appropriate for the removal of water in these foods, minimizing the chances of introducing systematic errors in this type of determination. Usually, fiberglass paper is already being used in other drying methods, such as infrared radiation drying, and microwave radiation (Heinze and Isengard, 2001; Knochel *et al.*, 2001). The application of glass fiber paper in thermogravimetric experiments in this work resulted in an increase in 2.57 % water contents for the cane and 6.43 % for the glucose syrup. This indicates that the glass fiber paper improves heat transfer during thermogravimetric drying because it allows a uniform distribution of the sample by reducing their capillary effect case hardening, thereby facilitating the elimination of moisture (Figure 2). The effect of the glass fiber paper (Table 2) explains the higher values of moisture contents observed in the samples of cane and glucose syrups. Samples rich in sugar, such as cane and glucose syrups tend to form a crust when heated (Figure 2) which hinders the elimination of water. The glass fiber paper acts as a sponge, promoting the uniform distribution of the sample in the capillary; this allows the surface to breathe and prevents crusting, which may interfere with the analysis (Nielsen, 1994).

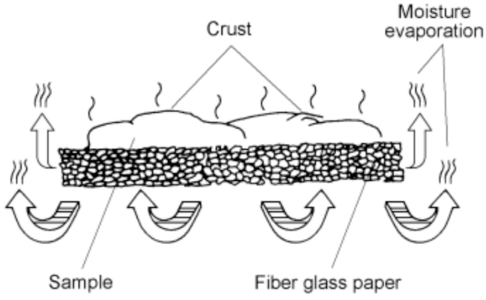

Figure 2 Diagram of fiberglass paper action in the drying of rich sugar samples

Felsner et al., 2004

3.3 Thermogravimetric Profile Drying

On the TG curves of cane and glucose syrups between 25 °C and 600 °C, there are four points of inflection that indicate changes in the speed of mass loss during thermogravimetric drying. The drying profile of the samples was a characteristic of the foods with high sugar contents as illustrated in Figures 3 (a) and (c). The first step of mass loss relates to the elimination of water. The second, third and fourth stages involve the thermal decomposition of other components, carbonization, and oxidation of organic matter, respectively. To check only water loss from the samples was plotted a graph of water contents versus temperature (Figures 3 (b) and (d)).

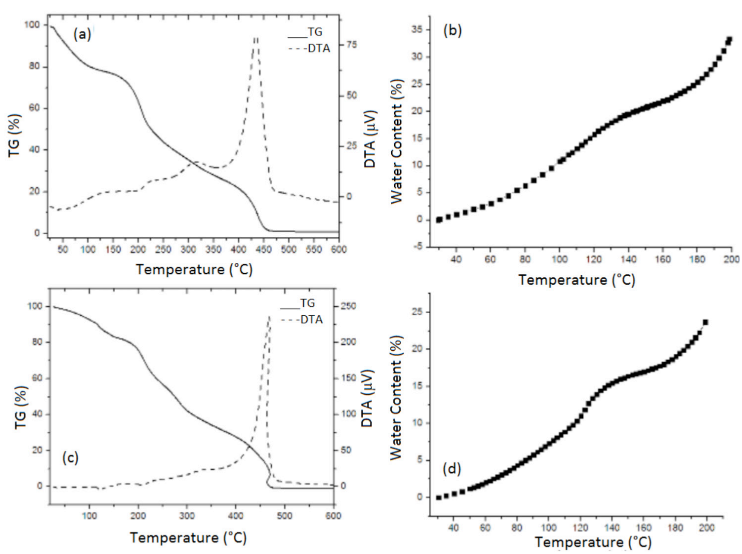

Figure 3 (a) TG (solid line)/DTA (dotted line) curves from cane (a) and glucose syrups samples (c) analyzed in the optimization study and drying thermogravimetric curves obtained from cane (b) and glucose syrups samples (d) in optimized conditions.

In Figures 3 (b) and (d), three weight losses were detected, suggesting that the water present in these matrices is linked in different ways. The mass loss between 25 °C and 110 °C is attributed to free water present on the surface of the matrix, easily removable. The mass loss between 110 °C and 150 °C indicates water tightly bound to the food matrix which depends on a higher temperature to be removed. Between 150oC and 170 °C is the loss of water mono-layer. Above 170 °C, rapid mass loss is attributed to the volatile degradation of sugar products present in this matrix, like fructose and glucose. The sigmoid behavior observed is very similar to the desorption isotherms for foods high in sugars (Mathlouthi, 2001; Ross *et al.*,2013; Kumaresan and Moorthy Babu,1997; Lee *et al.*, 2011a; Lee *et al.*, 2011b; Simkovic *et al.*, 2003; Quintas *et al.*, 2007; Eggleston *et al.*, 1996; Orsi, 1973). The water content of the other samples analyzed by thermogravimetry during the validation study was determined in the same way and have similar thermal behavior. However, the final drying temperature (Tfd) showed differences between evaluated samples (120-147 °C). It is known that the presence of impurities of various kinds and in varying amounts may accelerate the degradation of sugars and interfere in the water analysis (Ross *et al.*, 2013; Lee *et al.*, 2011a; Eggleston *et al.*, 1996).

3.4 Validation studies

The validation of the analytical methodology was performed by measurement of moisture values and comparison of data of the thermogravimetry and Karl Fischer titration (Table 3). The accuracy of the thermogravimetric method was evaluated using a paired *t*-test at a 95% confidence level, for the differences between the means of the moisture contents determined by the two analytical methods: thermogravimetric analysis and Karl Fischer titration. The results of this statistical test (t = - 0.66, p = 0.523; for cane and t = - 0.40; p = 0.693 for the glucose syrup) suggest that there are no significant differences in water contents obtained by the two analytical methods for these matrices. To assess the accuracy of the method was applied an *F*-test that considers the ratio between the variances of the thermogravimetric method and those of Karl Fischer titration. We calculated the confidence intervals for the ratio between the variance and standard deviations at a 95 % confidence level. The analysis of the F-tests (Fobs = 1.28; p = 0.685 for the cane and Fobs = 1.03 and p = 0.958 to glucose syrup) suggest that thermogravimetry and Karl Fischer methods have similar accuracy for the determination of water contents in sugar cane and glucose syrups. Those results are confirmed by the calculation of confidence intervals for the ratio of the variances (0.370 to 4.461 for cane and from 0.297 to 3.589 for glucose syrup), which have limits below 1.0. This indicates that there are no significant differences between the precision of the optimized thermogravimetric methods and that of the Karl Fischer titration. Thermogravimetric calculations confirm that the technique is optimized and validated for determining accurately water in cane and glucose syrups.

Samples	Water contents (g/100g) ± SD**	
	Karl Fischer*	TG/DTA
C1*	20.52±0.08	20.57±0.09
C2*	18.60±0.04	18.58±0.08
C3*	14.01±0.06	14.03±0.06
C4*	17.41±0.02	17.35 ±0.08
C5*	16.73±0.06	17.71±0.06
C6*	18.83±0.09	18.78±0.10
G1	15.21±0.08	15.25±0.04
G2	19.46± 0.03	19.47± 0.03
G3	15.22±0.05	15.17±0.02
G4	20.59± 0.03	20.52± 0.08
G5	32.24±0.07	32.20±0.07
G6	21.84±0.06	21.83±0.08

** published by Ducat et al., 2018*

*** Standard deviations were calculated using duplicate measurements from water contents (Karl Fischer and TG/DTA methods).*

Table 3 Water contents averages and standard deviations for cane (C) and glucose syrups (G) samples obtained by analytical methods.

4 | CONCLUSIONS

Analysis of the thermogravimetric profile of drying suggests that the cane and glucose syrups have complex thermal behavior. The results of optimization studies performed by the factorial design suggest that a heating rate of 5 ° C min-1and the use of fiberglass paper are best fitted to thermogravimetric drying of the samples. Those conditions provide better heat transfer in the sample during the drying process by minimizing the influence of degradation reactions in removing moisture. This study proved that depending on the sugar content in the samples, each requires a different drying temperature. Chemical composition of sugar-rich samples (molasses, honey, syrups) interferes with the drying process by indicating that fixed temperature drying methods are not suitable. The validation study showed that thermogravimetry has adequate accuracy and precision for the determination of the physical-chemical parameter in samples since significant random and systematic errors were not observed. The use of thermal analysis for determining the water content in sugar cane and glucose syrups can be advantageous over other methods, such as vacuum drying oven, infrared radiation drying or by Karl Fischer titration. This thermal analysis technique does not use expensive reagents or toxic solvents and further does not make use of any sample preparation, and it is also faster than drying in a vacuum oven. Besides, it allows the identification of the temperature in which water is eliminated and it obtains information on the stability and profile degradation which may contribute to the optimization of cane from the sugar production process and glucose syrups.

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