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PROCESSING AND PHYSICO-CHEMICAL CHARACTERIZATION OF WHOLE WHEY POWDER

Processamento e caracterização físico-química de soro em pó integral

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ABSTRACT

The purpose of the present study was to produce and to characterize whey powder with high fat content resulting from whey with total content of fat and by lactose crystallization of the concentrated product. Whole whey used was resulting from Coalho type cheese production and vacuum evaporation, lactose crystallization and drying were undertaken at semi-industrial scale. Coalho type cheese is a fresh cheese, obtained by rennet coagulation without addition of starter culture, it has 36% $w \cdot w^{-1}$ to 45.9% $w \cdot w^{-1}$ of moisture and its productions leads to typical sweet whey. The whey powder composition was determined and the lactose crystallization process was described. Isotherms were constructed from equilibrium data established for whey powder and salt solutions with known water activity. The sodium concentration of the samples was determined by flame photometry. The conditions of production applied in this experiment using whole whey lead to the formation of lactose crystals presenting the recommended size and percentage of crystallization for whey powder production. The high fat concentration of the whole whey powder allows its application in the chocolate industry. The proposed method for the determination of Na in dried whey was analytically adequate.

Keywords: spray drying; crystallization; fat; sodium.

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RESUMO

O objetivo do presente estudo foi produzir e caracterizar soro em pó com elevada concentração de gordura, oriundo de soro com teor total de gordura e pela cristalização da lactose do soro concentrado. O soro integral foi obtido da produção de queijo de coalho e a evaporação a vácuo, a cristalização da lactose e a secagem foram realizadas em escala semi-industrial. O queijo de coalho é um queijo fresco obtido por coagulação enzimática sem a adição de fermento lático, possui entre 36% $m.m^{-1}$ e 45,9% $m.m^{-1}$ de umidade e de sua produção acarreta um típico soro doce. A composição do pó foi determinada e a cristalização da lactose foi descrita. Isotermas foram construídas a partir dos dados de equilíbrio entre o soro em pó e soluções salinas com atividade de água conhecida. A concentração de sódio nas amostras foi obtida por fotometria de chama. As condições de produção aplicadas neste experimento, empregando soro integral, produziram cristais de lactose e uma porcentagem de cristalização recomendadas para produção de soro em pó. A elevada concentração de gordura no soro em pó obtido permite a sua aplicação na indústria de chocolates. A metodologia proposta para a determinação de sódio mostrou-se analiticamente adequada.

Palavras-chave: secagem por spray; cristalização; gordura; sódio.

INTRODUCTION

According to Knipschildt et al. (1994) the drying process is one of the best methods for preserving milk products, because this technique provides conversion of milk into milk powder with minimal nutritional loss. Milk drying process involves the formation of amorphous lactose that is highly hygroscopic. According to Hynd (1980), whey powder has the tendency to absorb water from ambient air, causing aggregation of colloid particles of the product during storage and substitution of an amount of amorphous lactose for crystalline lactose as a result of crystallization of the whey concentrate, transforming the whey into a non-hygroscopic product. Fox; Mcsweeney (1998) cited some technological precautions that should be observed when drying whey, such as a low concentration of lactic acid, predominance of pre-crystallized lactose and control of outlet air temperature from the drying chamber. Sorption curves are substantially affected by the presence of amorphous lactose (JOUPPILA; ROOS, 1994). The application of spray drying in whey processing and the development of markets using whey powder as ingredients in foodstuffs for human and animal consumption is of great importance to the dairy and cheese-making industries (MASTERS, 2002). According to Westergaard (2004), centrifugal separation of fat is one stage of whey pretreatment prior to evaporation.

In the industry of fat-enriched dairy powders, the aim of the manufactures is, on the one hand, to accelerate water transfers during drying and, on the other hand, to reduce free fat content of the powders, except for some specific products such as, for example, chocolate where free fat contents has to be increased (SCHUCK, 2007).

In a normal whole milk powder the continuous phase of a particle is the amorphous lactose which forms a very tight membrane protecting the globular fat against extraction. Thus an important condition for achieving high free fat content is to transform a substantial part of lactose into α-lactose-monohydrate. Besides removing part of the

lactose from the protective shell, the crystals formed create a net with craters and channels in between, through which the solvent for free fat determination can penetrate inside the particles. The crystal formation as such exhibits also a positive effect on creation of free fat, affecting the fat globules with sharp edges. Thus crystallization of lactose is an essential part of the process (PÍSECKÝ, 1997).

The purpose of the present study was to produce and to characterize whey powder with high fat content resulting from whey with total content of fat and by lactose crystallization of the concentrated product.

MATERIAL AND METHODS

Technological stages were undertaken at the Laboratories of CândidoTostes Dairy Institute, Juiz de Fora, MG, Brazil, and the sodium determinations were undertaken at Department of Chemistry of Federal University of Juiz de Fora, MG, Brazil.

The stages of the experiment are represented in Figure 1.

Whole whey sample

Whole whey used was resulting from Coalho cheese production. Coalho is a fresh

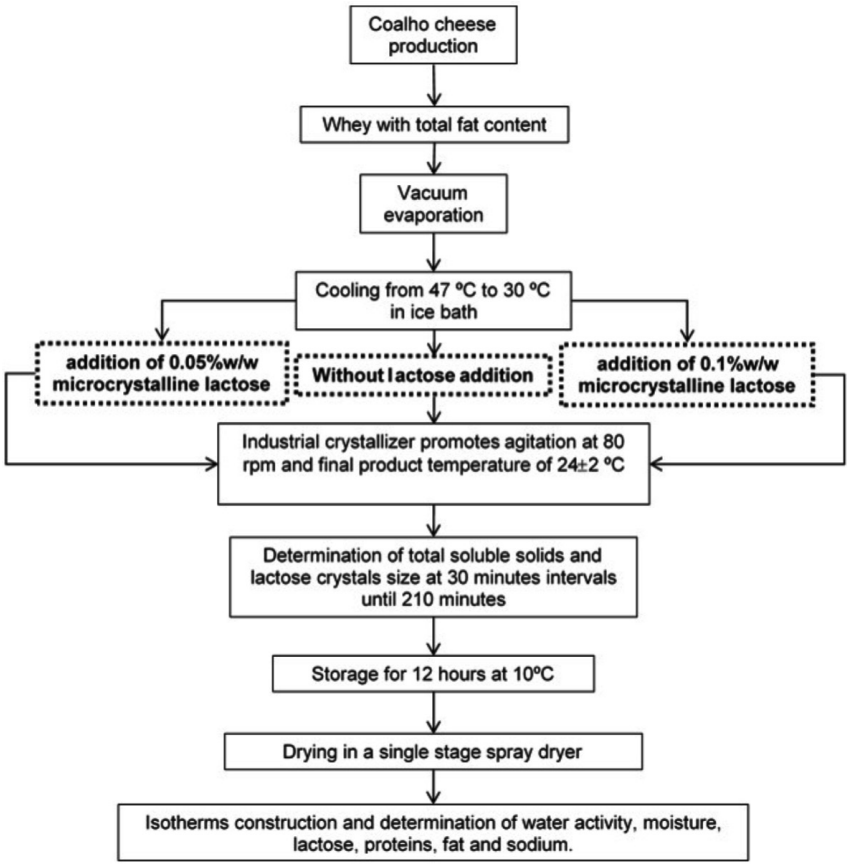

Figure 1 – Stages of the experiment

cheese type, obtained by rennet coagulation without addition of starter culture, it has 36% w·w^{-1} to 45.9% w·w^{-1} of moisture and its productions leads to typical sweet whey. It was applied the cheese technology described by Sobral et al. (2007). The volume of milk for the coalho cheese production was 200 liters for each batch and were performed 3 cheese productions for each concentration of whey (near than 400 liters of whey). As the whole whey did not pass through the centrifugation process it has the total content of fat.

Evaporation, crystallization and spray drying

Vacuum evaporation was performed in a single stage plate evaporator (Junior model, APV, São Bernardo do Campo, Brazil). During the vacuum evaporation the boiling temperature was 47 ºC and the concentration lasted 4 hours. The concentration of soluble solids in the whole concentrated whey (WCW) was standardized to 59 ºBrix ± 1 ºBrix. The attributes for whey vacuum concentration are presented in Table 1.

For the crystallization of the WCW three different treatments were applied: no addition of lactose, addition of 0.05% w·w^{-1} and addition of 0.10% w·w^{-1}. During the experiment three repetitions of each treatment were done resulting in nine productions. The amount of total solids of the WCW was determined by gravimetric methods (AOAC, 2005) and the lactose level by the Cloramina T method (WOLFSCHOON-POMBO; CASAGRANDE, 1982). The concentration of soluble solids (ºBrix) was determined using a digital refractometer (Reichert® model AR200, New York, United States of America). After the evaporation the WCW was transferred to an ice bath leading to a decrease in its temperature from 47 ºC to 30 ºC. The next step was to transfer the WCW to the industrial crystallizer with mechanical agitation. After 120 minutes of crystallization the product was cooled to 24 ºC ± 2 ºC. The total time of crystallization was 210 minutes. To determine the percentage

Table 1 – Attributes for whole whey vacuum concentration (n=9)

Attributes	Treatment 1	Treatment 2	Treatment 3
Pre heating temperature (ºC)	64 ± 2	64 ± 2	64 ± 2
Boiling temperature (ºC)	46 ± 1	46 ± 1	46 ± 1
Steam temperature (ºC)	60.9 ± 0.2	61.8 ± 0.2	60.7 ± 0.4
Temperature difference between the boiling section and heating section (ºC)	14.9 ± 1.2	15.8 ± 1.2	14.7 ± 1.4
Water temperature in the condenser equipment	32 ± 2	31 ± 2	33 ± 1
Steam pressure (MPa)	0.57 ± 0.04	0.60 ± 0.02	0.56 ± 0.03
Total soluble solids in whey (ºBrix)	5.5 ± 0.1	5.7 ± 0.2	5.6 ± 0.1
Total soluble solids in concentrated whey (ºBrix)	59.5 ± 1.0	59.9 ± 0.6	58.5 ± 1.3
Evaporation (minutes)	297 ± 3,7	290 ± 4.2	298 ± 2.8
Whey volume (L)	332 ± 15	355 ± 12	360 ± 6
Concentration factor (yield of the drying process)	10.8 ± 1.0	10.5 ± 0.5	10.0 ± 0.4
Evaporated water (kg)	301.3 ± 19	321 ± 8	324 ± 10
Rate of water evaporation (kg•h^{-1})	60.9	66.4	65.3

of crystallization, equation 1 was used as suggested by Westergaard (2001).

$$\%\ \text{cristalization} = \frac{(B_1 - B_2) \times 9500 \times 100}{L \times TDM \times (95 - B_2)} \quad (1)$$

Where: B_1 = initial ºBrix (time zero); B_2 = final ºBrix (after crystallization);
L = % lactose in the whey solids; TDM= % Total Dry Matter

During the crystallization was determined the percentage of soluble solids and lactose crystal size in intervals of 30 minutes. The average size of the lactose crystals was determined using an optical microscope (Nova®model 107, São Paulo, Brazil) and the image analysis was performed at computer software called MSI Ima Win, employing the technique reported by Martinez et al. (1990).

After crystallization the whey was maintained at 10 ºC for 12 hours before initiating the drying process, carried out in single-stage spray dryer (Minor model, GEA-NIRO, Copenhagen, Denmark). The drying process was performed to obtain a water activity to the value 0,200 on the whey powders (SCHUCK et al., 2008). The inlet temperature of the air was 200 ºC ± 5 ºC and the outlet temperature was 87,5 ºC ± 2,5 ºC.

The isotherms were constructed from equilibrium data established for milk powder and whey and salt solutions with known water activity. The salts and respective water activities (a_w) of the saturated solutions used were: LiCl (0.113), $MgCl_2$ (0.331), K_2CO_3 (0.432), Mg $(NO_3)_2$ (0.544) and NaCl (0.755). Equilibrium was achieved by determining the constant weight after 21 days at average temperature of 25 ºC. Water activity meter equipment (Aqualab Series 3, Jarinú, Brazil) was used to determine water activity. The determinations of protein and fat were done according to Pereira et al. (2000).

The isotherm data was applied to the model proposed by Brunauer et al. (1938), known as the BET model, expressed in equation 2 as:

$$\frac{aw}{m(1-aw)} = \frac{Cb-1}{m1Cb}\,aw + \frac{1}{m1Cb} \quad (2)$$

Where a_w = water activity value of the saline solution, m = mass of water in grams per mass of solids in grams, C_b = energy constant of the BET model which is logarithmically related to the energy difference between water molecules in the pure state and in the monolayer, m_1 = mass of water in gram per mass of solids in gram, in the region of primary sorption. Applying the method of least squares to the experimental data, which states that the values of Y are represented by a_w $(m_1 (1 - aw))^{-1}$ and the values of X are represented by a_w, it was possible to determine the values of m_1 and C_b. The constant C_b is approximately equal to $\Delta(H_1\text{-}H_v)\bullet(R\bullet T)^{-1}$, where R is the ideal gas constant and is equal to 8.314 $J.mol^{-1}.K^{-1}$, T is the temperature in Kelvin and $\Delta(H_1 - H_v)$ is the difference between enthalpy of adsorption in the monolayer and enthalpy of water condensation between water molecules in the pure state and the monolayer at equilibrium temperature T. It was thus possible to determine the value of $\Delta(H_1 - H_v))$ by means of the constant value obtained for C_b.

The MICROCAL 5.0 software (1997) was used for the statistical analysis of the results. Data were analyzed using descriptive statistics, analysis of variance with the Tukey test to compare means "a posteriori" and Student's t-test to compare means of paired data.

Determination of sodium in the samples obtained by flame photometry

The samples were homogenized and masses around 150.0 mg were weighed on an analytical balance.

The sampled portions were then digested, a process which occurs in the

decomposition of organic matter present in the sample. However, in order to establish an efficient procedure for this type of sample, tests were performed with the samples, using mineral acids and a plate heater (IKA brand®, C-MAG HP 10).

The method of digestion was established by testing only with HNO_3 and HNO_3 combinations with other reagents (H_2O_2 or HCl). It was weighed 150.0 mg of whey powder for each different test. The steps of the experiment are shown in Figure 2.

During the process of digestion it was necessary to replace reactants five times for the sample treated only with HNO_3, because of the high concentrate level of organic compounds present in the samples (mainly lactose) and for increase solubilization of salts, (sample 1, whey powder without lactose addition during the crystallization process), six times for the sample treated with H_2O_2 and HNO_3 (sample 2, whey powder with addition of 0.05% $w \bullet w^{-1}$ of lactose during the crystallization process) and nine times for the sample treated with HCl and HNO_3 (sample 3, whey powder with addition of 0.10% $w \bullet w^{-1}$ of lactose during the crystallization process).

At the end of digestion, the digested masses were moved to a 50.0 mL test flask and then filled up with deionized water. The solutions got a slightly yellowish color and no particles.

Subsequently, there were prepared sodium standards for calibration range between 10 and 40 $mg.L^{-1}$ 50.0 mL flasks, as Sodium Chloride PA (Trade Mark Vetec, Lot 09/2006) and HNO_3 2% $v \bullet v^{-1}$. A blank test containing only 2% $v \bullet v^{-1}$ HNO_3 was also prepared for the calibration solutions and all of them were stored in plastic bottles.

The readings of the samples, standards and blanks were performed in a flame photometer (Digimed DM-61) awaiting the stabilization of the analytical signal for Na (analyte) and Li, employed as internal standard at a concentration of 25 $mg.L^{-1}$.

For reliability of results (validation), some parameters of merit were evaluated (accuracy, precision, intermediate precision and linearity) using tests of addition and recovery of analyte (spiked sample) and sample solutions digested according to the procedure described.

RESULTS AND DISCUSSION

The average composition of the whey is shown in Table 2.

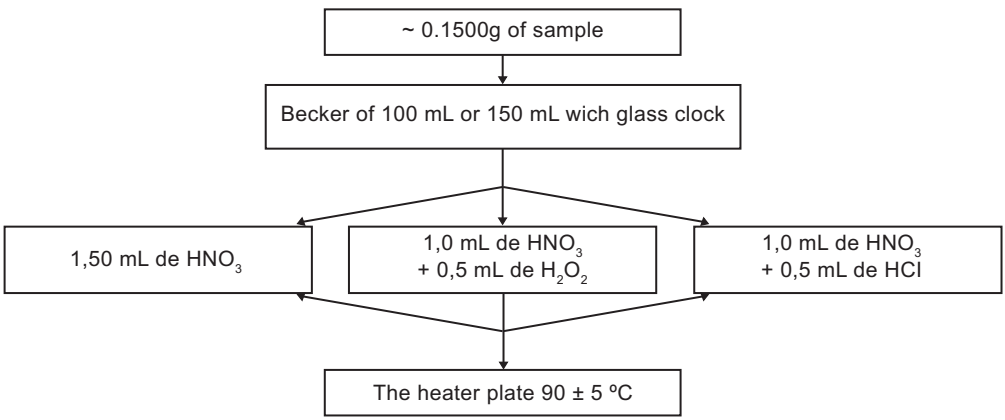

Figure 2 – Stages of digestion procedure

The average composition of the WCW is shown in Table 3.

The values for moisture obtained in treatments 2 and 3 are higher than 4.00% ($w \bullet w^{-1}$) and according to Schuck et al (2004) it leads to problems during powder storage, such as changes in color and caking. The inlet and outlet temperatures of the spray dryer were standardized during the experiment, however the higher values of moisture indicates that these controls were not sufficient. According to Schuck et al. (2008), the outlet temperature is not always the optimum parameter to affect the moisture content of dairy powder and the relativity humidity of the outlet air is the key parameter to optimize the moisture content and water activity of the whey powders.

Ordinary whey powder has 1.0 to 1.5% of fat (USDEC, 2014), however the conditions of vacuum evaporation, crystallization and drying applied in this experiment using whole whey lead to the average concentration of

Table 2 – Composition of the whey (n=3)

Values	Treatment 1	Treatment 2	Treatment 3
Total solid content	7,08	6,82	6,93
Fat content	0,58	0,50	0,63
Lactose content	5,40	5,26	5,21
Protein content	0,79	0,83	0,82
Acidity	0,12	0,13	0,13
pH	6,13	6,07	5,97
% Brix	5,40	5,53	5,77

Where: treatment 1 = whey for production without lactose addition; treatment 2 = whey for production with addition of 0.05% of lactose; treatment 3 = whey for production with addition of 0.10% of lactose.

Table 3 – Composition of the whey powders (n=3)

Attribute	Whey treatment during crystallization		
	1	2	3
Moisture content	3.95 ± 0.14	4.95 ± 0.09	4.67 ± 0.11
Total solid content	96.05 ± 0.14	95.05 ± 0.09	95.33 ± 0.11
Fat content	8.5 ± 0.1	8.1 ± 0.2	8.4 ± 0.1
Lactose content	67.19 ± 1.02	66.9 ± 0.94	66.53 ± 1.12
Protein content	11.17 ± 0.32	10.93 ± 0.22	10.74 ± 0.41
Water activity	0.193 ± 0.008	0.234 ± 0.009	0.200 ± 0.004
Lactose content in the total solids	69.95[a]	70.38[a]	69.79[a]
Percentage of lactose crystallization on the WCW	68	78	79

Where: 1 = without lactose addition; 2 = addition of 0.05% of lactose addition; 3 = addition of 0.10% of lactose addition; a = value calculated by difference.

8.3% ($w \bullet w^{-1}$). In addition the concentrated whole whey presents a range of crystallization from 68% to 79%, and according to Písecký (1997), the crystal formation as such exhibits also a positive effect on creation of free fat, affecting the fat globules with sharp edges. The presence of an elevated fat concentration and lactose crystals leads to the possibility of using the whole whey powder as a good ingredient for the chocolate industry.

To produce a stable whey powder the formation of small lactose crystals is desired in the concentrated whey prior to drying. According to Schuck et al. (2004) the lactose crystal size in concentrated whey has to be smaller than 100 µm to produce a stable powder. In this experiment the average size of the lactose crystals at the end of crystallization was 62.2 µm. The mean of crystal sizes did not differ at 5% of probability, indicating that independently of the quantity of lactose added, the crystals always had the same size. The average size of lactose crystals in sweetened condensed milk is 16 µm (RENHE et al., 2011), the difference of size comparing to concentrated whey can be explained by the high concentration of lactose in this product. According to Renhe et al. (2011), sweetened condensed milk presents near than 13.8% of lactose in the total solids and the concentrated whey present in this work a minimum of 69.79%. The driving force of the crystallization processes is the supersaturation of the solution so it leads to increase crystallization of lactose molecules and then bigger crystals can be formed.

These results indicate that the conditions of concentration and crystallization applied in this experiment using whole whey lead to the formation of lactose crystals presenting the desired size for whey powder production. On the other hand only by the addition of lactose nuclei it was possible to obtain lactose crystallization greater than 70% (68%, 78% and 79% of lactose crystallization for whey powder without lactose addition, by addition of 0.05% and by addition of 0.10% respectively). According to Schuck (2008), Masters (2002) and Písecký (1997), the lactose crystallization higher than 70% leads to a stable whey powder with a low tendency to caking.

The equilibrium data of the whey powders fitted by BET method well as shown by the coefficients of the statistical analysis and values of water in the monolayer, as well as by the difference between enthalpy of adsorption in the monolayer and enthalpy of water condensation, which were in accordance to with Timmerman et al.(2001). All whey powders analyzed presented moisture content above the values for the amount of water in the monolayer, ensuring that the chemical groups were completely surrounded by water, which exerts a protective effect to prevent chemical reactions, particularly oxidation.

From the values predicted by the BET model for water mass in the monolayer and the difference between enthalpy of adsorption in the monolayer and enthalpy of water condensation, the Pearson correlation coefficient was calculated for parametric analysis of correlation between these variables and the chemical composition. The statistically significant correlations are presented in Table 4.

The negative correlation between fat mass and water in the monolayer can be explained by the repulse effect that fat has in water, especially when there is considerable exposure of fatty acids in the whey due to partial or total disruption of the lipoprotein membrane surrounding the fat globule. The surface composition of the whole powders particles is on average of 55% of fat (VIGNOLLES et al., 2007).

The high negative correlation between the moisture content of the powder and the difference between enthalpy of adsorption in the monolayer and enthalpy of water condensation was expected and indicates that the higher the amount of water in the

powder, the smaller is the difference between the enthalpy of adsorption in the monolayer and the enthalpy of water condensation. Possibly lactose content in the dried milk products was positively correlated with the difference between enthalpy of adsorption in the monolayer and enthalpy of water condensation, indicating that water in the monolayer participates in more intense chemical interactions and/or that there is a greater increase in entropy than would be encountered in pure solvent.

Sodium determination in whey samples by flame photometry (method validation and results)

The mass of sample used was based on levels of sodium found in the literature (United States Department of Agriculture (USDA) National Nutrient Database for Standard Reference) and considering experimental conveniences such as glassware and working range (concentration of sodium in solution reading). Therefore, the volume of whey to be weighed and digested was relatively small.

For tests of digestion, the three tests led to obtaining solutions with suitable characteristics for analysis by flame photometry. Furthermore, sodium content found in the three samples which were prepared differently, were very similar, with deviation less than 4.6%. For the sample treated only with HNO_3 the concentration of sodium was 22,3 $mg.Kg^{-1}$, for one treated with H_2O_2 and HNO_3 the result was 23,8 $mg.Kg^{-1}$ and for the sample treated with HCl and HNO_3 the concentration was the 21,8 $mg.Kg^{-1}$.

Thus, it was decided to use only the digestion reaction HNO_3, since it required a smaller number of replacements thus optimizing the process. As for validation tests, the results obtained are shown below.

The addition and recovery test is related to the accuracy of analysis as a whole, since it reflects the amount of given analyte that is recovered in the process in relation to the actual amount present (or added) to the sample before the digestion process. The study of recovery is the "fortification" of the sample and is used by many researchers such as Brito et al., (2003).

Table 4 – Attributes calculated from the equations fitted to the BET model and statistical correlations

Treatments	Mass of water in the monolayer ($g.100^{-1}$ of solids)	$\Delta(H_1 - H_v)$ ($kJ.mol^{-1}$)
1	4.03	8.48
2	4.07	8.04
3	4.53	7.42
Correlations among the variables		
Fat (% w/w)	- 0.866 (p = 0.026)	-
Water (%w/w)	-	- 0.943 (p=0.005)
Lactose (%w/w)	-	0.972 (p=0.001)

Where: $\Delta(H_1 - H_v))$ = difference between the enthalpy of adsorption in the monolayer and the enthalpy of condensation of water between the water molecules in the pure state and in the monolayer; 1 = without lactose addition; 2 = addition of 0.05% of lactose; 3 = addition of 0.10% of lactose.

For this test were analyzed two replicates (m1 and m2) of whey powder without lactose addition containing 25 mg.L^{-1} of Li internal standard. After analysis of these solutions the recovery percentage was calculated using Equation 3.

$$\% \, Recovery = \frac{Na\,SF - Na\,AS}{EF} \, x\, 100 \qquad (3)$$

Where: NaSF = Sodium content on the sample fortified; NaAS = Sodium content in the actual sample; EF = Content expected on the fortification

The average recovery found for sodium content was 106.0 ± 5.6%, indicating that the established method shows good accuracy, with an average error of around 6%.

To ensure that the method does not work only with one sample, this test was applied to the sample 3, resulting in a recovery of 106.5 ± 0.7%.

Regarding the other parameters of merit, satisfactory results were also obtained.

Linearity was assessed from 1 to 40 mg.L^{-1}, in quintuplicate, generating a straight line equation (y = 1.0689x + 0.014) with a correction coefficient of 0.9999.

The precision is estimated based on the reading, also in quintuplicate, a sample solution (whey powder obtained by addition of 0.10% of lactose) and the relative standard deviation was 0.9%, relatively low and less than the limit value of 5.6% recommended by Wood (1999).

Intermediate precision was also evaluated using a single sample solution F9, which was assessed over three months. In this case, the relative standard deviation was 1.4%, and was considered adequate to characterize a small variation, according to research performed by Wood (1999).

Thus, the proposed method for the determination of Na in dried whey was analytically adequate, being then used for the analysis of whole whey powders samples and which had a content of 7.26, 7.62 and 7.03 g kg^{-1} of Na, respectively for whey powders obtained by no addition of lactose, addition of 0.05 % w·w^{-1} and addition of 0.10% w·w^{-1}. These values show that whole whey obtained according to the methods described in this article had a sodium content ranging from 6.3 to 7.8 g kg^{-1}, being a characteristic of both the procedure and materials used.

CONCLUSIONS

The three treatments applied for lactose crystallization lead to lactose crystals presenting the desired size and percentage of crystallization for whey powder production, but the addition of lactose during the crystallization process increases the efficiency and under the conditions of the experiment is the best strategy for lactose crystallization. In addition the high fat concentration of the whole whey powder allows its application in the chocolate industry. The proposed method for the determination of Na in dried whey was analytically adequate and no significant differences were observed among the pretreatments tested.

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