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SHORT COMMUNICATION

Evaluation of the calcium yield in the microencapsulation process

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Abstract

The aim of this study is to enrich gluten-free products in calcium, from a natural source. The calcium was micro-encapsulated at low pH 4,17 (ascorbic acid 20% was used) by spray-drying or lyophilization, and the % of calcium was determinated by ICP-MS. Batches of gluten-free(GF) cookies were made with the obtained powders, and the % of calcium was again determinated by ICP-MS, and some differences were found. We conclude that the best way to microencapsulate calcium is by lyophilization. The differences of calcium % in the GF products could be due to the different particle size of the powders.

Key words: gluten-free, celiac disease, microencapsulation, spray-drying, lyophilization, calcium, ICP-MS.

Introduction

Celiac disease (CD) is an autoimmune disorder, in which the gluten acts like an allergen, causing inflammation of the small intestine and leading to numerous abdominal as well as nongastrointestinal symptoms (DONALD *& al.* [1]), more specifically is the allergy to gliadin and prolamin peptides present in wheat, rye and barley (STEFAN *et al.* [9]), and it usually manifests with intestinal symptoms such as diarrhoea and malabsorbtion (EDWARD [3]).

The word „celiac", comes from the Greek *'Koiliakos'* which means - 'suffering of the bowels' (JANE *et al.* [5]).

It has been demonstrated by large screening studies that the CD prevalence is higher than previously thought, almost 1% of Europe and USA population is affected (DUBE *et al.* [2]; HOLTMEIER & CASPARY [4]).

A very important aspect in the gluten-free diet is to provide products with an improved content in minerals and vitamins that are necessary to celiac suffering people for a normal life. There is a recognized/demonstrated link between CD, calcium absorption and osteoporosis (KEMPPAINEN et al. [8]), so between CD and calcium absorption, that is why it is necessary to develop calcium-enriched gluten free products. Most people get their calcium needs from dairy products (L'ABBE [6]), but often in the initial steps of the CD it is recommended to avoid all the dairy products. Many times, CD is associated with different milk allergies. In the present work/study, our aim is to obtain calcium-enriched GF cookies using a casein-free source of calcium. There are also a lot of experimental evidences that a gluten- and casein-free diet can ameliorate the symptoms associated with autism spectrum conditions (WHITELEY *et al.* [7]).

Materials and methods

A casein-free source of calcium produced from calcareous marine algae (*Lithothamnion sp.*) (aq) with 36,87% Ca (soluble in week acids, odourless, neutral taste and appereance of white powder) bought from HSH Chemie S.R.L. (Roumania) was used.

Different technologies of enriching gluten-free cookies in Ca were tested:

1. aq powder (P1) was directly added to the cookies dough;
2. aq powder was first microencapsulated by spray-drying (P2, P3, P4);
3. aq powder was first microencapsulated by lyophilization (P5);
4. aq was first solubilized in acids and spray-dried (P6).

Aq was microencapsulated in a mixture of maltodextrin, modified starch and β-cyclodextrin by spray-drying on a Bϋchi Mini Spray Dryer B-290, at a temperature of 180^o-C, pump 10-15%. All the samples were spray-dried at pH=4,17, at this pH the aq powder was completly dissolved. The acid pH was achieved using ascorbic acid ($C_6H_8O_6$ 20% Scharlau, Chemie S.A.). Different weights of aq powder were used: P2 (10g Aq), P3 (5g Aq), P4 (3g Aq).

For the microencapsulation using the lyophilization technique the same mixture of maltodextrine, modified starch and β-cyclodextrin was used at the same pH (achieved with ascorbic acid 20%) on a Christ liophylizator at approximmately -54^{o}C and pressure 0,048 mbar. 5g of aq were used (P5).

A direct spray-drying using the same parameters as for P2, P3, P4, without the mixture of maltodextrine, modified starch and β-cyclodextrin was used for P6, at the same pH, 5g of aq powder were used.

The % of Ca was determined for all the calcium enriched powders (Figure 1), using an ICP-MS Agilent 7500ce Octopole Reaction System, for the sample pretreatment (digestion) a microwave oven Ethos One, Milestone was used, at 121^{o}C for 1h 33 minutes. All the samples were digested with hydrogen peroxide (H_2O_2 30% Merck, Germany) and nitric acid (HNO_3 69% Panreac, Canada).

The powder samples were further used to make batches of gluten-free cookies, using the same recipe for each batch, each GF batch had approximately 395g. The different weights of the powder samples were added and mixed with the rest of ingredients so every batch will have the same amount of Ca (aprox. 500 mg) as shown in the table (d. GF cookies/ g used). The cookies were cooked for 20 minutes at 200^{o}C. The raw materials (rice, sugar, margarine, eggs) were bought from a local supermarket (Spain). The % of Ca was determined once again by ICP-MS for the GF cookies obtained (Figure 2).

Results and discussions

Comparing all the microencapsulation techniques, it can be seen that the most effective method for the Ca yield is the one by lyophilization P5, and the most uneffective is P6 (Table 1). Althought all the GF batches should have had the same content of calcium, it seems that there are some small differences due to process loses, or an uneven distribution of the calcium in the GF dough.

Table 1. a. aq used for microencapsulation b. microencapsulated powder
c. Ca loss in ME powder d. g of powder used in the GF cookies

No.	a. aq used for microencapsulation			b. microencapsulated powder		c. Ca loss in ME powder		d. GF cookies
	Batch	g of aq	g Ca	Obtained powder	g Ca	g Ca	% Ca	g used
1.	P1(aq)	-	-	-	-	-	-	1,35
2.	P2	10	3,69	73,70g	1,71	1,98	54	21,55
3.	P3	5	1,84	60,70g	0,93	0,91	50	32,68
4.	P4	3	1,11	52,30g	0,65	0,46	41	40,00
5.	P5	5	1,84	106,00g	1,69	0,15	**8**	31,45
6.	P6	5	1,84	9,70g	0,63	1,21	**66**	7,72

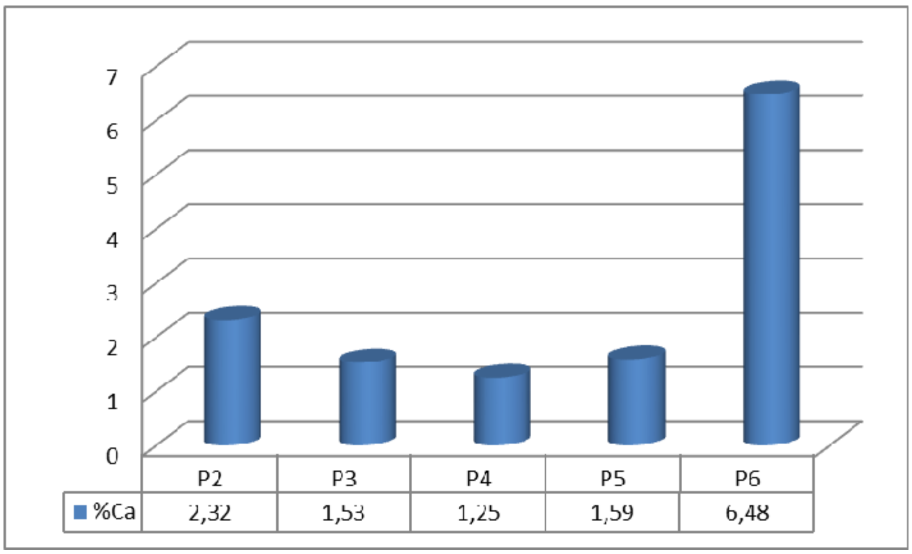

Figure 1. % of Ca determined by ICP-MS

The samples of gluten-free cookies obtained usig microencapsulated calcium, had a higher content of Ca as shown in the figure 2.

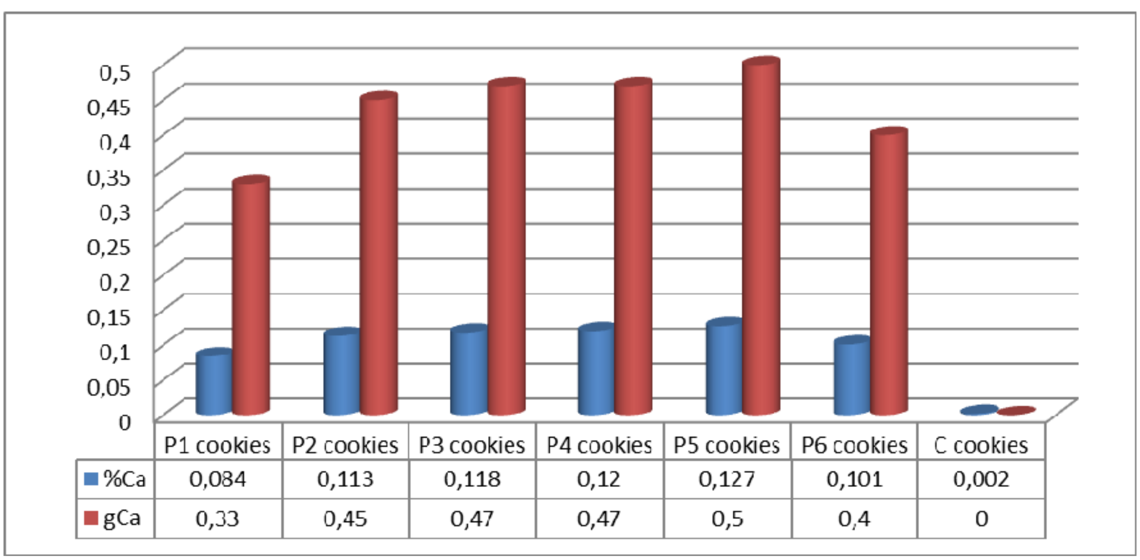

Figure 2. % of Ca and g of Ca reported to the dry matter in the GF cookies

Conclusions

These work was made to microencapsulate and solubilize calcium in a mixture of maltodextrin, modified starch and β-cyclodextrin, and to be further used in different food recepies.

Regarding the Ca solubilization and microencapsulation, it is clear that the best way to microencapsulate calcium, with the smallest losses, is by lyophilization, only 8% of the calcium was lost; for the spray-drying microencapsulating technique the highest % of calcium reported to the dry matter was in P1 (3g of aq). The direct spray-drying technique(P6) was the most unaffective(Table 1).

We can conclude that for this study all the enrichment methods have similar results in the gluten free cookies (P2, P3, P4, P5 and P6). The differences between the GF cookies batches could have been caused by the uneven distribution in the dough. It is easier to incorporate the microencapsulated calcium in all kinds of food and beverages due to its solubility.

Further experiments will be made to study if the microencapsulation affects, and in what way, the calcium bioavailability.

Acknowledgements

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