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RESEARCH ARTICLE

Ultrasound-assisted extraction of *Psyllium mucilage*: Evaluation of functional and technological properties

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ABSTRACT

The objective of this work was to optimize the extraction of psyllium mucilage and to evaluate its functional and technological properties, aiming for the first time the use in ice cream. A factorial design was carried out to test different extraction conditions. Were evaluated the water solubility (WS) and oil absorption capacity. Mucilage (gel and dried) was applied in ice cream, and submitted to overrun, melting, color and sensory analysis. Time and temperature were significant parameters, being the best conditions at 60ºC for 2 hours. Samples presented WS between 200 and 1400 g/kg, increasing proportionally to temperature. Ice cream with gel presented higher value and started the melting process faster than the mucilage one. There was a significant difference between samples in flavor, color and texture, and the purchase intention of most tasters indicated "maybe would buy", and both ice cream (with gel and with mucilage) presented hight acceptance index. Psyllium presented good technological properties, that are very desirable in food products. It was possible to find the best condition for psyllium mucilage extraction and an ice cream application obtaining a product with good acceptance (82%).

Keywords: Functional propertie; Extraction; Sensory analysis; Ice cream

INTRODUCTION

Demand for functional foods has increased due to a number of factors, such as consumers trying to prevent diseases, aging populations, rising health costs, scientific evidence that diets can affect occurrence and progression of diseases and changes in food regulation. Concomitantly, we can observe a growth in development of foods and products with functional activities (Singh, 2019).

Functional food is classified as all foods or components thereof that have high nutritional value inherent to their chemical composition, which provide in addition to nutritional function, a potentially beneficial role to improve, maintain and strengthen consumers health (Bultosa, 2016). An example of these foods is Psyllium, that is currently attracting interest (Figuero & Staffolo, 2019).

The genus *Plantago* comes from Western Asia, and has more than 200 species, of which *Plantago ovata* and *Plantago psyllium* are the most commercially cultivated in the world. The peel is the main product obtained from seed milling, which yields about 10 to 25% the dried seed weight. The main characteristic is its high content of soluble and insoluble fibers, being that the soluble fibers in presence of water can form a transparent and mucilaginous gel (Huerta et al., 2016). This gel has excellent water-holding properties, acting as an emulsifier and stabilizer of emulsions, being useful in ice cream formulations. According Maestrello et al., (2017) due to these properties, its application in industry is quite interesting and can replace in whole or in part the emulsifier and fat, giving greater nutritional and functional quality to the products.

Psyllium has been used in the development of new products, and enrichment or substitution of ingredients in food preparation, due to its emulsifying, gelling and thickening capacity. In addition, it can be used *in natura* providing physiological benefits, acting significantly on

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water retention in the fecal matter and normalizing the rate of the digesta passage, and the formation of feces in the colon (Kaur et al., 2017).

Another plus is that psyllium does not have gluten in its composition, making it safe for celiacs. Also, it can be used in the production of beverages, desserts, dairy products, biscuits, pasta and bread.

However, to improve the use of psyllium, it is of great importance to study its properties in relation to food production technology, being that functional property is a specific technological property (water absorption capacity, fat absorption capacity) that influences the physical appearance and behavior of a food product in acharacteristic way. It depends not only on the protein, lipid and carbohydrate contents of a food, but also on the interaction of these and other components in the food matrix (Porte et al., 2011).

It is necessary to improve physical-chemical, functional, sensory and biological properties of psyllium, to promote its use in food. Due to its extremely strong water absorption ability, it is challenging to disperse psyllium in water or aqueous solutions, even with vigorous stirring and heating, factors that reduce its applicability (Wärnberg et al., 2009).

One method that can be used to disperse of psyllium mucilage ins solutions is the ultrasonic bath. Ultrasound energy is an efficient way to improve the performance of many different applications in the food industry, such as organic and organic compound extractions, homogenization and other applications (Nascentes et al., 2001). The use of ultrasound or sonication to break the cell membranes has the advantage of greatly reducing extraction time and increasing extraction yield. The use of ultrasound application disrupts the cell wall structure and accelerates diffusion through membranes; thus, the cell lyses and therefore contributes to the release of cellular contents (Falleh et al., 2012).

According to Souza et al (2020) the aqueous extraction (by using hot water) is the most common method used to obtain psyllium polysaccharide, but Nie et al. (2017) reported a different way that soon after crude polysaccharide is precipitated with ethanol and centrifuged.

The choice of the product was because ice cream is a product sensorialy enjoyed and consumed by people of all ages. However, it is composed of fat, emulsifiers and sugar. As today's consumers search for healthier food products with functional appeal, low caloric density, a high fiber content and the sensory qualities of traditional products, there is an opening in the market for the development of new formulations of ice cream to comply with consumer demand (Campos et al., 2016). In this paper was proposed an investigation of the use a natural ingredient, the psyllium mucilage and gel, as a substitute for the emulsifier/stabilizer.

Thus, the objective of this work was to optimize the extraction using the ultrasonic bath and to evaluate functional and technological properties of psyllium mucilage, aiming later application in ice cream (in gel or mucilage).

MATERIALS AND METHODS

Materials

Psyllium (*Plantago ovata*) and in natura components used in this research were purchased from the same lot in the local commerce of the city of Maringa, Parana, Brazil. The reagents used were of analytical grade.

Experimental design for mucilage extraction

First of all, a factorial design was carried out with 8 experiments and 3 repetitions at the central point to test different extractiobn conditions using the ultrasonic bath application (40 Hz), considering constant temperature (25°C) and as variables, the psyllium ratio parameters: water (1:60, 1:80, 1:100), time (1, 2, 3 hours) and pH (2, 6, 10), being considered as response the yield and emulsion stability.

Mucilage obtention and analysis

Samples were prepared in equivalent ratio of psyllium: water (pH being adjusted when necessary with hydrochloric acid and/or 0.1 M sodium hydroxide). Subsequently, it was placed in an ultrasonic bath for a fixed time under constant stirring at 500 rpm.

After that, the gel was filtered in two steps, the first to remove larger particles using a cotton cloth and then the gel was filtered with organza fabric to remove smaller particles. The gel was transferred to silicone trays and carried to a forced air circulation oven at 50 °C for 24 hours until the mixture was completely dried (Campos et al., 2016).

The separated mucilage was then weighed on analytical scale and this data used for the calculation of extraction yield. Emulsion stability analysis was performed for each sample obtained. All assays were performed in triplicate.

Yield and emulsion stability (ES)

The yield was calculated as a function of the inlet mass of psyllium. For the emulsion stability test, the methodology described by Chau et al. (1997) was used. The emulsified layer was measured and the emulsion stability was calculated according to Equation 1. All tests were performed in triplicate.

$$EE(\%) = 100 \times \frac{V_{EAF}}{V_{SAI}} \quad (1)$$

Where VEAF is the volume of the final heated emulsified layer (mL) and VSAI is the volume of the initial heated suspension (mL).

Instrumental color

Color was analyzed using the Konica Minolta CR-410 portable colorimeter. The system used was CIEL*a*b*, in which the L * coordinates were measured, representing the luminosity, a* which indicates the shades red (+)/green (-) and b* yellow (+)/blue (-). The analysis was performed in triplicate.

Mucilage and gel analysis – Water solubity and Oil absorption

The following analysis were also performed with the mucilage gel not dried, obtained in the mucilage step. Thus, it waschosen to named gel, the wet mucilage, and mucilage, its dry version.

Water solubility was based on the methodology of Betancour-Ancona et al., (2004). 40 mL of a solution (1:100) was prepared in a centrifuge tube and kept stirring at 25, 30, 50, 60 and 80 °C bath for 30 min. The suspension was centrifuged at 4000 rpm for 15 minutes and the supernatant decanted. A 10 mL aliquot of the supernatant was air-dried in forced circulation oven at 120 °C for 4 hours until constant weight was reached. The percentage of solubility was calculated by equation 2 and then a graph was constructed.

$$Solubility\left(\frac{g}{Kg}\right) = \frac{dry\ weight\ at\ 120°C \times 400}{weight\ of\ the\ sample} \quad (2)$$

The oil absorption capacity was based on the methodology of Capintani et al., (2012), in which a 10 mL aliquot of 1:100 mucilage solution was homogenized for 2 minutes at 4000 rpm. 10 mL of soybean oil was added and stirring was continued for 3 minutes; The emulsion formed was centrifuged for 30 minutes at 2000 rpm, then the volume of the supernatant (oil) was measured. Absorption capacity of oil was expressed in mL of absorbed oil per mL sample.

Mucilage and gel application in ice cream: overrun, melting and sensory analysis

Both mucilage and gel, were added to ice cream, the extraction was performed according to Antigo et al., (2017), 80 °C for 1.2 hours and ratio 1:100 (seed: water m/m). The basic formulation of the ice cream was composed of 72% UHT milk, 4% milk powder, 6% pasteurized cream, 15% crystal sugar and 1% flavoring (white chocolate flavor). Thus, two samples were produced, one with gel and the other with mucilage, mucilage was added to both formulations at 2% (defined in preliminary tests), as a substitute of stabilizer and emulsifier. For each formulation, the ingredients were weighed separately, added into a 2 L beaker and homogenized by mechanical overhead stirrer for 2 min, then taken to a discontinuous ice cream producer (Sorvemaq), for 20 minutes at -18 °C until freezing, according to the methodology proposed by Vacondio et al. (2013). After manufacture ice creams were packed in 2L plastic containers and stored at -18 °C.

The overrun analysis was performed with both gel and mucilage ice cream, the initial and final volumes were measured, being the initial volume for the ice cream base (V_i) and volume of the final product (V_f), the overrun was calculated using equation 3.

$$overrun = (V_f - V_i) \times 100 \quad (3)$$

The melting test was performed according to the methodology described by Granger et al. (2005). The data obtained was plotted on a graph where melted ice cream weight is a function of time.

Sensory analysis was performed shortly after the ice cream manufacturing at the State University of Maringa. Acceptance test used a nine points hedonic scale, where 1- Was dislike extremely, and 9- Like extremely; and the pucherse intention (1-3) and the acceptance index (IA=average divided by the maximum grade*100). The attributes evaluated were color, texture and flavor, using a team of 100 untrained tasters and potential consumers. Samples of 20 g were presented in white disposable plastic containers encoded with random three-digit numbers. The use of mucilage in ice cream was approved by the UEM Ethics Committee under number: 36660514.5.0000.0104.

Statistical

By analyses of the fatorial desing was applied Statistic 7.0. Others result obtained was submitted to analysis of variance (ANOVA) and compared by Tukey test with significance level of 5% using Sisvar 7.0 (Ferreira, 2014).

RESULTS AND DISCUSSION

Psyllium Mucilage Extraction

In the design (Table 1) it can be seen that only time was significant ($p < 0.05$), an increase in time results in higher yield and emulsion stability (ES), as shown in the Pareto diagram (Figs. 1 and 2). Time and pH were significant for yield, while ratio and time, as well as ratio and pH, were significant for emulsion stability. Regarding color, there was no significant difference ($p < 0.05$) between samples. The following equation was obtained for yield: Y = 3.16

Table 1. Conditions and results of mucilage extraction for yeld and EE (%), and color (L, a*, b*)

Run	Ratio (w/w)	Time (h)	pH	Yield (%)	EE (%)	L	a*	b*
1	1:60	1	2	39.93	41.30	83.25	-2.91	11.24
2	1:100	1	2	42.56	37.45	82.24	-2.65	10.67
3	1:60	3	2	47.63	42.05	80.49	-2.80	10.36
4	1:100	3	2	33.40	38.10	80.49	-2.36	9.85
5	1:60	1	10	32.77	35.16	80.49	-2.36	9.85
6	1:100	1	10	25.60	42.43	79.86	-2.20	12.00
7	1:60	3	10	35.77	42.09	79.86	-1.99	13.10
8	1:100	3	10	47.97	42.46	80.33	-2.20	12.00
9	1:80	2	6	46.50	43.58	81.32	-2.75	10.09
10	1:80	2	6	43.00	39.88	82.23	-2.88	9.79
11	1:80	2	6	38.33	42.01	82.23	-3.21	10.09

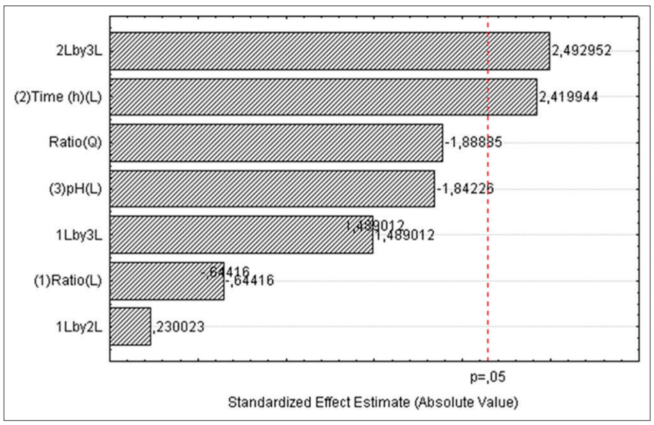

Fig 1. Pareto diagram for yeld (%), time variation, pH and ratio seed: water.

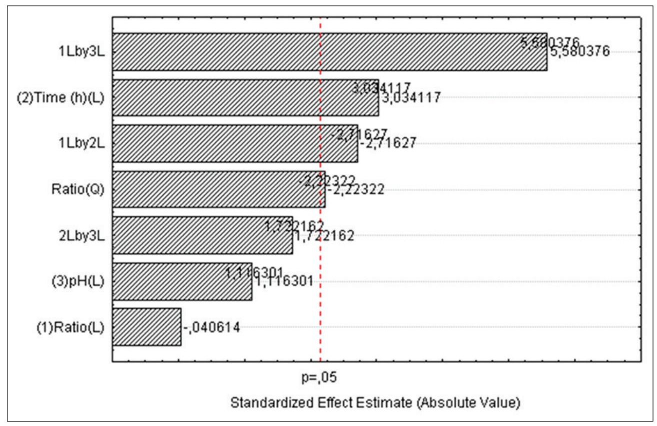

Fig 2. Pareto diagram for EE (%), time variation, pH and ratio (seed: water).

$X_2 + 3.26 X_2 X_3$. And the following equation for emulsion stability: $Y= 1.04 X_2 – 0.93 X_1 X_2+ 0.91 X_1 X_3$.

Finally, only the results of the ultrasonic bath were analyzed in the central area of the experiment, being that, pH 6.0 for 2 hours, and 1:80 ratio (seed: water). The yield obtained was 42% and emulsion stability 38%, values very close to those obtained in similar conditions with the use of ultrasonic bath (tests 9, 10 and 11, Table 1).

Antigo et al. (2017) evaluated the extraction of psyllium mucilage without ultrasonic bath at 80 °C for 1.2 hours and seed: water ratio of 1: 100 with stirring at 1000 rpm, the authors obtained yield of 65.0%, higher results than the present study.

The yield values obtained in the design ranged from 25.60 to 47.97%, values below that obtained in literature (Antigo et al., 2017). Probably, these lower values are due to temperature as mentioned above and also the second filtration step used in this work, because organza performs finer filtration, removing smaller particles from the previous filtering, this step was not used by the authors.

Mucilage extraction yield may also vary depending on various parameters such as the seeds botanical origin and genotype, the pH, the extraction time, the seed solids to solvent ratio, and among others. In some cases, seed soaking in water does not allow adequate to associate mucilage. The dissociation of the inner adherent layer can be achieved only by targeted physical, chemical or enzymatic treatmen (Soukoulis et al., 2018).

The values obtained for emulsion stability in the design ranged from 35.16 to 43.58%, differently from values above 90.00% found by Martins et al., (2016) when studying chia flour. As they are different seeds, this discrepancy may also be due to one being gel and the other being flour.

Water solubility and oil absorption capacity

It can be seen from Fig. 3 that with increasing temperature there was an increase in water solubility for both samples. The mucilage showed a slightly higher water solubility than the gel.

Due to this high ability to solubilize in water, mucilage and gel have a potential industrial application, since the higher solubility mucilages are considered to have better quality (Mhinzi & Mrosso, 1995). Therefore, they can be used in the preparation of various products such as bakery, as this is one of the properties needed to obtain a longer soft and moist product, making this property important (Souza et al., 2008).

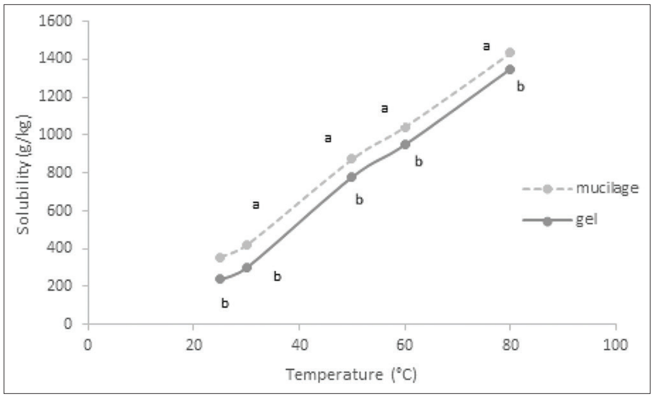

Fig 3. Solubility of psyllium gel and mucilage as a function of temperature.

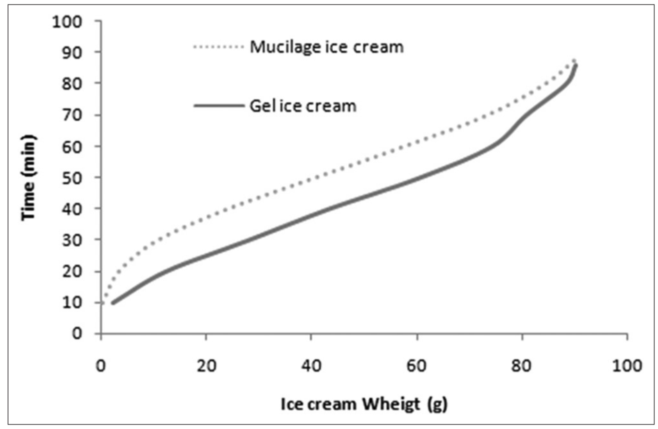

Fig 4. Ice cream melting process.

Regarding the oil absorption capacity, the same happened, the mucilage (8,125%) presented a higher capacity than the gel (7,5%), however the values are not significantly different ($p < 0{,}05$). These results may have occurred because the gel did not go through the drying step, even though the mucilage was reconstituted to perform the analyzes. But they are great values for this ability to absorb oil, which makes mucilage and gel efficient to use as ingredients in viscous products such as soups, processed cheeses and pastas and as meat extenders (Porte et al., 2011).

Application in ice cream

Regarding the incorporation of air (overrun), it was observed that the gel presented a value of 80% while the mucilage 60%. Maestrello et al., (2017) produced ice cream with chia mucilage (*Salvia hispanica* L.) and obtained yield values between 25 and 55%, considering addition of mucilage from 0.6 to 1.8%, values lower than those obtained in the present work. It is known that ice cream with a lot of air has a foam consistency and ice cream with little air is very heavy. According to Souza et al., (2008) in a discontinuous producer machine, the air is incorporated by stirring inside the syrup at atmospheric pressure, obtaining 50 to 100% overrun, so the overrun found here is within the standard for a discontinuous ice cream producer. Fig. 4 shows melting versus time for ice cream with mucilage and gel.

Ice cream with gel started the melting process faster than the mucilage ice cream (Fig. 4). Both ice creams melted close to 90 minutes, but gel ice cream melted first and not steadily, this may have occurred because gel has a higher amout of free water, forming ice crystals that tend to melt faster, not forming a three-dimensional network with fat during the aeration and freezing process. Since it is the interaction of fat with air bubbles and ice crystals responsible for the melting speed (Passos, et al., 2016). This may also have occurred because the overrun result is higher, which means that gel ice cream has a higher amount of air.

Granger et al., (2005) report that fat interferes with ice cream melting because fat globules that surround air bubbles stabilize the gas phase, consequently increasing the levels of fat aggregation, improving the resistance of the ice cream to melting. Also, Damodaran (2007) emphasizes that the addition of hydrocolloids slows down the rate of ice crystals formation because it increases the viscosity of the liquid phase, forming a three-dimensional network, which decreases water mobility. Thus, it is noted that both formulations with addition of psyllium mucilage showed longer melting time, therefore mucilage may represent a good alternative to substitute fat.

Table 2 presents the results of ice cream color parameters. Due to the ice cream being white chocolate flavor, the formulations presented light tonality, the luminosity ranged from 84.78 to 87.26 close to maximum luminosity (100). There was a significant difference ($p < 0.05$) for the parameters a *, which indicates the shades red (+)/green (-), and b *, which represents yellow (+)/blue (-), the sample with gel was more luminous and had more green and yellow.

All analyzed sensorial parameters, for both ice cream samples, obtained scores from 6 to 8, corresponding to the terms of the hedonic scale "like slightly", " like moderately" and "like very much", these results show general product acceptance. We concluded this by comparing our results with others found in literature, as example, Maestrello et al. (2017) evaluating an ice cream with chia mucilage presented scores 6.5 and 6.9 (using the same formulation and scale), for 1.2 and 1.8 % of mucilage addiction, respectively. Campos et al., (2016) obtained scores between 7.01 (1%) and 6.87 (2%) of chia mucilage addiction.

It is important to highlight that psyllium mucilage presented nutritional benefits by comparing with other mucilages (as example: Opuntia ficus, chia, and okra), and also according Souza et al (2020) presented antioxidant compounds that are very desirable in food products.

Table 2. Instrumental color results of ice cream

Sample	L	a*	b*
Mucilage icre cream	84.78[a]±0.02	-6.25[a]±0.08	20.34[a]±0.08
Gel ice cream	87.26[a]±0.05	-6.90[b]±0.03	20.89[b]±0.07

Averages with equal letters, in the same colunm, do not differ significantly at the 5% level of significance.

Table 3. Sensory results

Sample	Gel ice cream	Mucilage ice cream
Color	7.59[a]	7.25[b]
Smell	7.21[a]	6.87[b]
Flavor	7.89[a]	7.66[a]
Texture	6.79[b]	7.44[a]
Global Aceptance	7.45[a]	7.45[a]
Pucherse intention	2.52[a]	2.39[a]
Acceptance index (%)	82.78	82.78

Averages with equal letters, in the same line, do not differ significantly at the 5% level of significance.

Table 3 shows that there was a significant difference ($p < 0.05$) between the samples in smell, color and texture. Both formulations used equal ingredients, only the type of mucilage was substituted, but gel ice cream flavor obtained a higher score, pleasing more tasters. The significant difference ($p < 0.05$) in color occurs because mucilage is harder to solubilize in ice cream solution, causing dark spots to apper, which may have resulted the lower score. The difference in texture was significant, with the mucilage sample obtaining higher scores, which may have occurred because gel ice cream contains more water, which leads to crystallization and faster melting, compromising texture. There was no significant difference in taste and overall appearance ($p < 0.05$). The intention of purchase for both ice creams was close, "maybe would buy or would not buy".

CONCLUSION

In view of the results obtained, we can conclude that the best conditions for the extraction of psyllium mucilage use the ultrasonic bath, which contributes to process optimization. The potential of psyllium as a functional food, is due to its technological and functional properties, thus, it can be applied in numerous food products, adding nutritional value, and contributing properties, such as emulsification.

Psyllium is an alternative for the food industry, due to its high yield and viable source, and it can generate desirable texture and sensorial characteristics that satisfy consumers requirements, highlighting the high acceptance index (82 %) in the ice cream obtained in this research. However, there are few studies on psyllium mucilage, so further research is needed to discover other peculiarities of this seed.

Conflict of interest

Authors declare that they have no conflict of interest.

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