

UNIVERSIDADE ESTADUAL DE MARINGÁ CENTRO DE CIÊNCIAS AGRÁRIAS Programa de Pós-Graduação em Ciência de Alimentos

PROPRIEDADES DE MISTURA DE MUCILAGENS OBTIDAS DA CASCA DO PSYLLIUM (*Plantago psyllium L*) E DA SEMENTE CHIA (*Salvia hispanica L*).

MARIANA MENCONI CHINELLATO

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Dissertação apresentada ao programa de Pós Graduação em Ciências de Alimentos da Universidade Estadual de Maringá, como parte dos requisitos para a obtenção do título de mestre em Ciências de Alimentos.

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"PROPRIEDADES DE MISTURA DE MUCILAGENS OBTIDAS DA CASCA DO PSYLLIUM (*PLANTAGO PSYLLIUM* L.) E DA SEMENTE CHIA (*SALVIA HISPANICA* L.)"

Dissertação apresentada à Universidade Estadual de Maringá, como parte das exigências do Programa de Pósgraduação em Ciência de Alimentos, para obtenção do grau de Mestre em Ciência de Alimentos.

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BIOGRAFIA

Mariana Menconi Chinellato, filha de Célia Regina Menconi Chinellato, nasceu em 14 de janeiro de 1986, na cidade de Limeira, São Paulo.

Graduou-se em janeiro de 2011, como Engenharia de Alimentos pela Universidade Estadual de Maringá-UEM campus sede, com a defesa do trabalho intitulado "Elaboração de banco de dados de julgadores para realização de análises sensoriais", orientada pela professora Ph.D. Miriam Carla B. Ambrosio Ugri.

Na graduação participou de projetos de extensão na área de carnes e polpa de frutas congeladas. Foi membro dirigente no CREA-JR e técnica do laboratório de Tecnologia de Alimentos. Organizou diversos eventos como ENEEALI (Encontro Nacional de Engenharia de Alimentos) no ano de 2010 em Foz do Iguaçu e a SEMANEA (Semana Acadêmica de Engenharia de Alimentos) no ano de 2009 na cidade de Maringá, Paraná.

Tem experiência nas áreas de vegetais atuando principalmente com os seguintes temas: Carboidratos formadores de gel oriundos de fontes vegetais, processamento de alimentos por extrusão, analise sensorial quantitativa, tecnologia de produção de pães e tecnologia de produção de cervejas artesanais.

Dedico

esta obra a todos os cientistas que enxergam o conhecimento como um bem de todos e que compartilham suas experiências para a evolução da Ciências de Alimentos.

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APRESENTAÇÃO

Esta dissertação de mestrado está apresentada na forma de um artigo científico

AUTORES: Mariana Menconi Chinellato e Antonio Roberto Giriboni Monteiro

TÍTULO: Properties of mucilage blends using psyllium husk (*Plantago psyllium L*) and chia seed (*Salvia hispanica L*).

REVISTA: Food and Bioproducts Processing (Qualis B1)

GENERAL ABSTRACT

INTRODUCTION. Hydrocolloids are widely used in food industry as they have ability to retain water, are notable thickeners and gelling, have capacity of synerese controlling and stabilize emulsions. Psyllium husk and chia seed are sources of those hydrocolloids and the physicochemical properties of their mucilage them an emerging ingredients in the manufacture of bread, cakes and desserts as substitute of fat ingredients. Some polysaccharide gels may have weak interactions that, in some cases, are strengthened by the use of mixtures of polysaccharides called mixed gels. An example is the case of dilute solutions of xanthan gum and locust beans, which do not exhibit significant shear stress, but together they do.

AIMS. This thesis aimed to investigate the chia and psyllium mucilage properties when mixed in different proportions and also to check for changes in these features when these mucilage are extracted together.

MATERIAL E METHODS. Psyllium husk (P) and chia seed (C) from the respectively species *Plantago psyllium L* and *Salvia hispanica L*, were obtained in a local market and sent to Cereals Technology laboratory to be storage under room temperature. The mucilage extraction consisted in an aqueous method. Temperature, time and seed/water ratio were settle based in the literature previous studies. Two different methods were used to join the mucilage: blending and combining water extraction in which the raw materials were added and extracted together. Six treatments using mucilage powders of psyllium husk (P) and chia seed (C) were prepared. The blending range was: (T1) 100% P; (T2) 75% P, 25% C; (T3) 50% P, 50% C; (T4) 25% P, 75% C and (T5) 100% C. The combined mucilage was denominated T6. For a complete homogenization, each treatment was dissolved in distilled water, scattered using a mixer, and freeze-dried for 48 hours (-50°C). Factors such as total carbohydrate content, °Brix, pH, oil holding capacity, loss of solubility were used to investigate the mucilage interactions. The mucilage blends profile were also raised as the thermal characteristics, viscosity and the attenuated total reflection (ATR) spectra. In order to compare samples, analysis of variance (ANOVA) followed by the Tukey test allowed the distinction of the treatments to 5% significance level.

RESULTS E DISCUSSION. The aqueous extraction yield for chia, psyllium and combined mucilage were respectively 9.03% of dry chia seed mass, 47.4% of dry psyllium husk mass and 21.98% of dry chia seed and dry psyllium husk mass. The pH, °Brix and oil-holding capacity showed no significant difference between the reconstituided samples. Regarding to carbohydrates content, T5 results were significantly higher than the others. The psyllium mucilage resulted in a solubility of 6.98% (T1) while the chia mucilage was 51.26% (T5). By this fact, psyllium uses most of its mucilage to form the gel chain while chia mucilage may loss the major part of it after centrifugation. Tests conducted to conclude that the combined fraction is consistent with the individual mixed one in a proportion of 32% P and 67% C. The TGA plot of the samples showed thermal effects resembling to that of a natural hydrogel. An early endothermic loss assigned has been reported by the loss of absorbed moisture. Two stages of decomposition were observed for all samples. The first is responsible for the major breakdown of the polymer chain. The second decomposition stage is characterized by a decreasing curve up to the final temperature of 600°C. The char yield average was 38.68%. The mucilage of all treatments were found to have shear thinning properties. Three shear rate points (2.72s⁻¹, 7.82s⁻¹ and 14.96s⁻¹) of the viscosity curve were compared. Once the chia mucilage concentration is raised, the viscosity has a downward trend from T1 to T5. SampleT6 presented unexpected behaviour presenting the highest viscosity value of the blends indicating that

aquous combined extration acts differently than blending process. Similar ATR spectra were obtained for psyllium and chia mucilage on previous studies. In general, the bands in certain regions emerge or become more visible as it adds the different mucilage. Sample T6 did not follow the trend of the mixtures and presented in most regions similar spectra to the psyllium mucilage. Assuming that the molecular structure, among other factors, influences the properties of a polymeric gel, the ATR spectra may explain the T6 viscosity behavior above expectation since it conformation is similar to T1.

CONCLUSION. Blending chia and psyllium mucilage behaves differently than the combined extraction as the viscosity resulted of combined mucilage was higher than expected being the highlight of this work. As the industry seeks to leverage factors, such as thickening, without adding artificial ingredients, combined extraction process may prove to be interesting in this case. The ATR spectra was an important tool to investigate the causes of the increased viscosity by displaying, on the molecular level, the similarities with the highest viscosity samples structures.

Key words: hydrocolloid, TGA, DSC, viscosity, ATR.

RESUMO GERAL

INTRODUÇÃO. Os hidrocoloides são amplamente utilizados na indústria alimentícia uma vez que estes possuem a capacidade de reter água, são notáveis espessantes e geleificantes, têm a capacidade de controlar sinérese e também controlam e estabilizam emulsões. A casca de psyllium e as sementes de chia são fontes desses hidrocoloides e as propriedades físico-químicas de suas mucilagens transformaram estes em emergentes ingredientes para a substituição de gordura na fabricação de pães, bolos e sobremesas. Alguns tipos de gel de polissacarídeo podem ter interações fracas que, em alguns casos, são reforçadas pela utilização de misturas de polissacarídeos chamados géis mistos. Um exemplo é o caso de soluções diluídas de goma de alfarroba e xantana, que não apresentam a tensão de cisalhamento significativa, mas em conjunto o fazem.

OBJETIVOS. Este trabalho propõe uma investigação das propriedades das mucilagens de chia e de psyllium quando misturadas em diferentes proporções e também verifica se há alteração dessas características quando essas mucilagens são extraídas em conjunto.

MATERIAIS E MÉTODOS. Psyllium husk (P) and chia seed (C) das respectivas espécies *Plantago psyllium L* e *Salvia hispanica L*, foram obtidos em um mercado local e enviados ao Laboratório de Tecnologia de Cereais para serem armazenados em condições normais ambientes. A extração das mucilagens consistiu em um método aquoso. Temperatura, tempo e proporção de água/sementes foram fixadas com base em trabalhos anteriores da literatura. Foram utilizados dois métodos diferentes para combinar as mucilagens: mistura e extração combinada onde as matérias primas foram adicionadas em um mesmo sistema de extração. Fatores como o conteúdo total de carboidratos, ^oBrix, pH, capacidade de retenção de óleo e perda por solubilidade foram usados para investigar as interações das mucilagens. Foi também levantado o perfil dessas misturas de mucilagens quanto as características térmicas (TGA e DSC), a viscosidade e o espectro de reflexão total atenuada (ATR). A fim de comparar amostras, análise de variância (ANOVA) seguida pelo teste de Tukey, foi utilizada permitindo distinguir os tratamentos a um nível de significância de 5%.

RESULTADOS E DISCUSSÃO. O rendimento de extração aquosa das mucilagens de chia, psyllium mucilagem combinada foram, respectivamente, 9,03% da massa seca semente de chia, 47,4% da massa seca de casca de psyllium e 21,98% da massa seca das sementes de chia seca mais psyllium. Parâmetros da mucilagem reconstituída com pH, ºBrix e capacidade de retenção de óleo, não apresentaram diferença significativa entre as amostras. No que diz respeito ao teor de carboidratos, T5 apresentou valores significativamente elevados (13,14 g/L) se comparado aos demais (±8,25). A mucilagem de psyllium resultou em uma solubilidade de 6,98% (T1), enquanto a chia foi de 51,26% (T5). Sendo assim, ao que parece o psyllium utiliza a maior parte da sua mucilagem para formar a cadeia gel enquanto a mucilagem de chia pode perder a maior parte desta após a centrifugação. Teste de solubilidade permitiu inferir que a fracção combinada é consistente com uma mistura de mucilagens na proporção de 32% psyllium e 67% chia. O TGA das amostras mostrou efeitos térmicos que se assemelham ao de hidrogéis naturais. Uma perda endotérmica precoce atribui-se à perda de humidade absorvida. Foram observados dois estágios de decomposição para todas as amostras. O primeiro e principal é responsável pela quebra da cadeia de polímero. A segunda etapa de decomposição é caracterizada por uma curva decrescente até a temperatura final de 600°C. A média geral das amostras quanto ao teor de cinzas foi 38,68%. Quanto a viscosidade, todas as amostras apresentaram um comportamento de redução da viscosidade a medida que se aplicou a taxa de cisalhamento. Três pontos da taxa de cisalhamento (2.72 s⁻¹, 7.82 s⁻¹ e 14.96 s⁻¹) foram comparados. Uma vez que a concentração de mucilagens chia é levantada, a viscosidade tem uma tendência de queda de T1 para T5. T6 apresentou um comportamento inesperado apresentando valor mais elevado do que as outras misturas. A extração aquosa quando combinada age de forma diferente que o processo de mistura individual. Espectros similares aos da literatura foram obtidos com a técnica de ATR para as mucilagens de psyllium e chia. Em geral, as bandas em certas regiões emergem ou tornar-se mais visíveis uma vez que se adiciona a mucilagem diferente. A amostra T6 não seguiu a tendência das misturas e apresentou em grande parte das regiões, espectros semelhantes ao da mucilagem do psyllium. Partindo do princípio que as estruturas moleculares, entre outros fatores, influenciam as propriedades de um gel polimérico, o ATR pode explicar o comportamento acima do esperado da viscosidade de T6 uma vez que este é bem semelhante conformação de T1 (amostra de maior viscosidade).

CONCLUSÃO. As propriedades das misturas das mucilagens de chia e psyllium se comportam de forma diferente dependendo da forma como são misturadas. Na extração combinada a viscosidade resultou em um valor maior do que o esperado se destacando das demais misturas. À medida que a indústria procura potencializar fatores, tais como o espessamento, sem fazer adição de ingredientes artificiais, o processo de extração combinada pode revelar-se interessante neste caso. A análise dos espectros do ATR foi uma ferramenta importante para investigar as causas da viscosidade elevada da amostra combinada, exibindo, em nível molecular, as semelhanças estruturais com a molécula de psyllium.

Palavras chaves: hidrocoloides, TGA, DSC, viscosidade, ATR.

Properties of mucilage blends using psyllium husk (*Plantago psyllium L*) and chia seed (*Salvia hispanica L*).

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ABSTRACT

This paper used psyllium (P) and chia (C) mucilage blends obtained through mixing or combined extraction to investigate the interactions properties of these potential polysaccharide ingredients, important sources to the food industry as thickeners and stabilizers. We found the values of 9.03% for extraction yield, 47.4% and 21.98% for chia, psyllium, and combined mucilage. The samples were prepared as following percentages: (T1) 100% P; (T2) 75% P, 25% C; (T3) 50% P, 50% C; (T4) 25% P, 75% C and (T5) 100% C and the combined extraction mucilage was T6. Factors such as pH, °Brix, and oil-holding capacity revealed no significant difference between the samples; higher carbohydrates values were indicated for the content of chia seed (13.14 g/L). Solubility losses ranged from seven to 51.25 percent without the occurrence of an interaction effect. The thermal effects was similar to natural hydrogels and the chia mucilage was more stable during the major breakdown stage of decomposition. The corroboration of the interaction property occurred through the viscosity factor. The viscosity of the combined sample had higher values than the blended samples and the attenuated total reflection (ATR) spectra indicated more molecule conformation similarities with the psyllium than chia.

Highlights: Psyllium husk aqueous extraction presented higher crude yield compared to chia seed extraction although both results were higher than currently literature studies; \blacktriangleright Mass balance was used to predict the combined extraction ratio; \blacktriangleright Combined extraction produced mucilage with higher viscosity than expected showing this parameter as dependent on the extraction technique; \blacktriangleright The ATR spectra produced important evidences of the mucilage similarities pointing an explanation to the unexpected results.

Key words: hydrocolloid, TGA, DSC, viscosity, ATR.

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1. Introduction

The food industry has largely employed hydrocolloids for their ability to retain water and representing remarkable thickeners and gelling in addition to the capacity to synerese controlling and stabilize emulsions (Phillips & Williams, 2000; Muñoz, Cobos, Diaz, & Aguilera, 2012).

Psyllium (eg. *Plantago psyllium L*) contains soluble fibers that are functional hydrocolloids and it is found in the plant seed of the *Plantago* genus with over 200 species distributed in temperate regions worldwide, such as India and Iran. (Guo, Cui, Wang, Goff, & Smith, 2009; Rahaie, Gharibzahedi, Razavi, & Jafari, 2012). Considered a low-cost polysaccharide source of great technological potential, it is renewable and presents biodegradability characteristics and hydro affinity that allow its application as hydrogel (Thakur & Thakur, 2014). The polysaccharides found in psyllium are constituted of xylose (74.6 %), arabinose (22.6 %), and traces of other sugars. With approximately 35% of non-reducing terminal residues, psyllium polysaccharides are highly branched constituted of β (1 \rightarrow 4) bonds with D-xylopyranosyl residues in the main chain (Fischer, et al., 2004).

Soaking chia seed (eg. *Salvia hispanica L*) from annual herbaceous plant belonging to the *Lamiaceae* family into water makes it exude its hard adhered mucilage (Lin, Daniel, & Whistler, 1994; Capitani, Spotorno, Nolasco, & Tomas, 2012). This mucilage have physicochemical properties responsible for its emerging as an ingredient for the production of bread, cakes, and desserts substituting fat ingredients (Borneo, Aguirre, & Leon, 2010; Capitani, Spotorno, Nolasco, & Tomas, 2012). Lin et al. (1994) describe the constitution of the fibrous content of the chia mucilage as high molecular weight polysaccharides (from 0.8 to 2.0 x 10^{-6} Da) with basic units tetrasaccharides residues, 4- O-methyl - α D-glucopyranosyl, branches with β -D- xylopyranosyl residues in the main chain, and units of ($1 \rightarrow 4$) - β -D- xylopyranosyl - ($1 \rightarrow 4$) - α -D- glucopyranosyl ($1 \rightarrow 4$) - β -D- xylopyranosyl.

Polysaccharides (gums, hydrocolloids) promote the viscosity of liquid solutions modifying or controlling their flow properties and texture; for semisolid products, they act on the deformation properties (BeMiller & Huber, 2010). The polysaccharide gel may have weak interactions, in some cases reinforced with the use of polysaccharides mixtures, called mixed gels, such as in dilute solutions of xanthan gum and locust beans, which only present significant shear stress when combined (Walstra & Vliet, 2010).

This paper is an investigation of the interaction between two emerging thickening ingredients, psyllium and chia mucilage, by applying a combined water extraction as well as the blending of different mucilage proportions.

2. Materials and methods

2.1. Materials

Psyllium husk (P) and chia seed (C) from the respectively species *Plantago psyllium L* and *Salvia hispanica L*, were obtained in a local market and sent to Cereals Technology laboratory. The husk and seed were separated stored in polyethylene bag under normal conditions of brightness and room temperature until the extraction processing. All solvents were anal

2.2. Mucilage extraction

Chia mucilage extraction was carried out based on Muñoz et al. (2012) by inserting samples of whole seeds in a beaker with distilled water 1:40 at 80°C. After a two-hour constant stirring using a screw propeller stirrer, the aqueous suspension extracted was filtered in cloth, spread on a drying tray and air-forced dried at the temperature of 50°C during 20 hours. The mucilage was subjected to hermetic storage and refrigeration (\pm 5°C) until the analysis. Ratio of 1:100 was established for the psyllium mucilage extraction based on Ahmadi et al. (2012), using distilled water at 80°C during one-hour stirring and subjected the aqueous suspension to a double filtering in cloth with the same drying and storage conditions employed for the chia mucilage. The third extraction consisted of a combination of the abovementioned methods. It was proceeded with the addition of 50g chia seed into 2L becker with distilled water at 80°C and after one- hour stirring, 20g of psyllium husk; the extraction continued for another hour and the aqueous suspension was subjected to double filtered in cloth. The drying and storage conditions remained were the same employed to the previous samples. Yield was measured by dividing the weight of the mucilage obtained through the initial raw material weight and multiplied by 100. Equation (1) was used to estimate the combined mucilage yield , where m_p and m_c are the weight of psyllium and chia, y_p and y_c are the yield of the isolated extraction previously carried out.

Combined mucilage (%) =
$$\frac{m_p * y_p + m_c * y_c}{m_p + m_c}$$
 (1)

2.3. Experimental plan

This experiment was conducted with six treatments using mucilage powders of psyllium husk (P) and chia seed (C). The mixing range was: (T1) 100% P; (T2) 75% P, 25% C; (T3) 50% P, 50% C; (T4) 25% P, 75% C and (T5) 100% C. The combined mucilage was denominated T6. For a complete homogenization, each treatment was dissolved in distilled water, scattered using a mixer, and freeze-dried for 48 hours.

2.4. Reconstituted mucilage solutions

The reconstituted of frozen dried mucilage was based on the Leon-Martinez (2011) method, with modifications. A magnetic stirrer (Fisatom 7BD) was used to scatter the mucilage (room temperature, 90 min) and distilled water to prepare the 1:100 (mucilage:water rate) solutions.

2.5. Total carbohydrates content, brix and pH

The content of total carbohydrate was assessed through the phenol-sulfuric method described by Dubois et al. (1956). A refractometer Briobrix was used to determine the Brix (°Brix). The pH of the reconstituted mucilage solution was determined using potentiometric measurement at 25 °C.

2.6. Oil-holding capacity (OHC)

Oil-holding capacity was assessed based on Capitani et al. (2012) by subjecting an aliquot of 10mL of one percent reconstituted mucilage to a two-minute homogenization using a Fisatom 7BD magnetic stirrer at 5000 rpm. An oil-in-water emulsion was prepared by adding 10 mL of soybean oil under a constant three minutes stirring. The emulsion was subjected to a 30-minute centrifugation at 455xg for; subsequently, the measure of the supernatant oil volume was proceeded. Oil-holding capacity is expressed as mL oil held per mL sample.

2.7. Water solubility index (WSI)

Samples were dissolved in 30 mL of distilled water (1:100), scattered it using a magnetic stirrer (Fisatom 7BD) for five minutes at 60°C, and centrifuged at 455xg for 30 minutes (20°C). The supernatant was subjected to a six-hour drying process at 100°C and the final weight was divided by the initial weight with the result multiplied by 100 indicating the value for the water solubility index.

2.8. Thermal characteristics

TGA (Thermogravimetric analysis) and DSC (Differential Scanning Calorimetry) was performed according to Iqbal et al. (2011) using a simultaneous thermal analyzer NETZSCH STA 409 PG/PC, under nitrogen atmosphere at a flow rate of 50 cm³ min⁻¹ with 10 °C min–1 heating rate at ambient temperature range (25°C) to 600 °C using platinum crucible. TGA peak was found through differentiate TGA curve

2.9. Viscosity

A controlled stress rheometer (Brookfield DV-III, USA) with a concentric cylinder geometry (SC4-27) was used for the viscosity of the freeze dried reconstituted mucilage. The constant temperature of 25°C was maintained using a water bath (Brookfield TC-502, USA).

2.10. Attenuated total reflection (ATR) spectra

ATR was employed to observe the structural modification of the samples. The spectra of the freeze dried mucilage powder were obtained using an infrared Fourier transform spectrometer (model Vertex 70v, Bruker, Germany) with platinum ATR diamond f/ vaccum. The spectral range was 400–4000 cm⁻¹ with 128 scans, resolution of 4 cm⁻¹, aperture setting 6mm and acner velocity of 10Hz.

2.11. Statistical analysis

We carried out the statistical Analysis of Variance (ANOVA) followed by the Tukey test to distinguish the treatments using on RStudio software (Illinois/NCSA) with a five-percent significance level. Extraction yield, total carbohydrates content, brix, pH, oil-holding capacity, water solubility index and viscosity were carried out in triplicate.

3. Results and Discussion

3.1. Aqueous extraction

The crude yield was approximately 9.03% for dry chia seed mass, 47.4% dry psyllium husk mass, and 21.98% dry chia seed as well as dry psyllium husk mass.

The mucilage of chia seed has a strong attachment to the coat after water extraction making the separation an important factor in the yield process. Timilsenaa, Adhikarib, & Kasapisa (2016) obtained 5.6% yield by conducting an lyophilization 1:20 (seed:water ratio) of soaked seeds with swollen mucilage and mechanical separation grounding into powder passing through 200 μ m sieves. Muñoz et al. (2012) obtained 6.97% with 1:40 (seed:water ratio), 8.0 pH controled extraction, tenhour drying at 50°C, and mucilage separation from the seed rubbing the dried sample over a 40mesh screen. Studies have successfully approached the yield factors (temperature, pH, water:seed ratio) with this particular paper focusing on the separation method. The 9.03% yield was achieved through the separation using cloth and a higher seed:water ratio (1:40). The highest ratio benefited the separation through a low thickness aqueous suspension. By separating the seed before the drying procedure, we believe that extraction yield was higher considering that the procedure was conducted with the soaked seed. Felisberto et al. (2015) achieved 7.86 g /100 g of chia seeds by separating the (1:40) aqueous solution with a brush depulper and a vaccum filtering before the drying stage.

Despite the many resports on psyllium yield, only a few studies, such as Ahmadi et al. (2012), indicated 28% of filtrate dry matter for psyllium husk using hot water extraction. The method with more common application involves alkaline extractions followed by acid neutralization. No previous reports in literature are registered for the psyllium husk aqueous extraction combined the chia seed; however, equation 1 estimated a yield of 19.99%, which is numerically similar to the experimental result (21.98%). This study does not reveal any evidence of sinergic yield interaction between the

raw materials during aquous extration using the studied conditions, although further experimental design studies would allow further information by using controled factors such as pH.

The pH of the reconstituted mucilage ranged from 6.33 (T1) to 6.83 (T4) and the brix from 0.75 (T1, T2, and T3) to 1.0 (T6). Oil-holding capacity ranged from 0.895 (T1) to 1.66 mL_{oil}/mL_{sample} (T3). No significant difference between the samples was observed regarding those parameters. However the carbohydrate content of T5 (13.14 g/L) was significantly different from others where the values ranged between 7.37 (T1) and 8.68 g/L (T4).

The solubilization loss was measured using the WSI (Water Solubility Index) (Figure 1). The psyllium mucilage resulted in an insolubility of 6.98% (T1) while the chia mucilage resulted in 51.26% (T5). By separating in two phases, gel and supernatant, psyllium uses most of its mucilage to form the gel chain while chia mucilage may loss the major part of it after centrifugation. The results of the blends indicated the expected behavior and the individual WSI revealed no evidence of a synergic effect of the blendings.

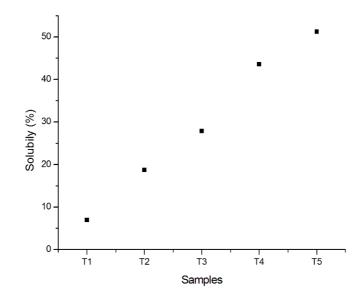


Fig. 1: Comparison of mucilage samples for the water solubility property.

Considering that the WSI chart have a linear range ($R^2 = 0.9913$) between the blends, this study investigates whether the combined extration has the same behavior of the blended ones using the same mucilage proportion. In order to have it revealed, we used mass balance (Figure 2) to predict the amount of chia and psyllium mucilage on the combined mucilage. Accordingly, a sample containing a mixture of 32% C and 67% P was prepared for a triplicate test. The obtained WSI was 23.18%, close to the 22.63% found for the combined mucilage leading to the conclusion that the combined fraction is consistent with the individual mix at a proportion of 32% P and 67% C (between T2 and T3).

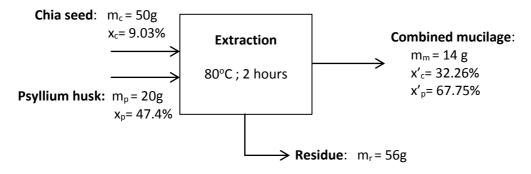


Fig.2: Mass balance of combined extraction where m_p and m_c are the weight of psyllium husk and chia seed; x_p and x_c are the percentage of mucilage of raw material; m_r is the residual weight; m_m is the mucilage weight, and x'_p and x'_c are the final fraction of each mucilage.

This results suggest that it is possible that the combined mucilage extraction have no interference on the solubilization of the individual mucilages. It is important and convinient when planning and developing a new product to consider an expected behaviour for the mixtures.

3.2. TGA and DSC

The TGA plot of the samples revealed thermal effects similar to natural hydrogels reported in a few earlier studies (Iqbal, Akbar, Saghir, Karim, & Koschella, 2011; Timilsena, Adhikari, & Kasapis, 2016). The observation of an endothermic loss had been previously reported due to the loss of absorbed moisture at a range of 50-100°C. A possible explanation for the low temperature of moisture loss is the use of platinum crucible substituting hermetical sealing.

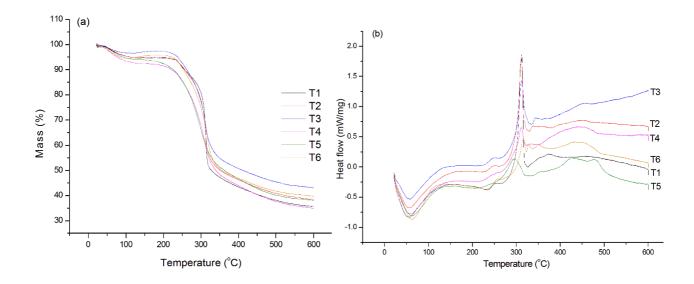


Fig. 3: (a) TGA and (b) DSC curves of mucilage samples.

The average representative weight loss was 4.94% despite the significant difference between T3 and T4 (2.33% and 7.29%, respectively) (Table 1). Two stages of decomposition were observed for all of the samples. Generally, at stage one, the initial decomposition temperature (IDT) ranged from 142 to 211°C and at stage two, the final decomposition temperature (FDT) ranged from 329 to 350°C. Stage one resulted in a 45.63% weight loss in average, with an exothermic enthalpy alteration. This stage is responsible for the major breakdown of the polymer chain forming reasonably high molecular mass volatiles. Studies have reported an IDT of 220-280°C and a FDT of 310-375°C for

chia, psyllium, and natural polysaccharides mucilage (Timilsena, Adhikari, & Kasapis, 2016; Iqbal, Akbar, Saghir, Karim, & Koschella, 2011; Iqbal, Akbar, Hussain, Saghir, & Sherd, 2011). Chia mucilage results indicate better stability with the lowest weight loss of 41.33% and T6 with the major breakdown occurring at the highest temperature. The TGA peak previously observed in T4 was followed by the highest dehydration value. This study suggests that the amount of water in the polysaccharide chain could be responsible for the weakness of the main structure for exposing more boundaries compared to the others. The second decomposition stage was characterized by a decreasing curve up to the final temperature of 600°C and a mean final weight of 10.75%. The char yield average was 38.68%.

Sample	Dehydration (%)	Stage 1 (°C)	TGA Peak* (°C)	Weight loss (%)	Stage 2 (°C)	Weight loss (%)	Char yield (%)
T2	4.78	207-330	312.67	46.44	330-600	9.83	38.95
T3	2.33	206-330	310.54	44.27	330-600	9.60	43.80
T4	7.29	165-330	302.04	44.55	330-600	12.89	35.27
T5	5.29	142-340	313.20	41.33	340-600	14.65	38.73
T6	5.14	211-350	315.17	47.66	350-600	8.00	39.20

Table 1. TGA results of the mucilage samples.

*TGA peak was obtained by differentiate TGA curve.

Despite of the fact that psyllium and chia mucilage have thermal characteristics of hydrogels, it was possible to differentiate these materials by the degradation behavior on stage 1 and 2. Chia mucilage required more specific heat to keep the breakdown after stage 1 by the exothermic peaks observed. In addition, chia had the lowest weight loss (41.33%) at stage 1 followed by 14.65% loss over 340°C showing that this mucilage have higher thermal stability compared to psyllium mucilage.

3.3. Viscosity

As a characteristic of non-Newtonian fluids, viscosity presented variations according to the shear rate applied (Fig 4). All of the treatments had mucilage indicating shear thinning properties, attributed to the presence of high molecular weight materials; therefore, the apparent viscosity indicated a pseudoplastic behavior (F.M. León-Martínez, 2010).

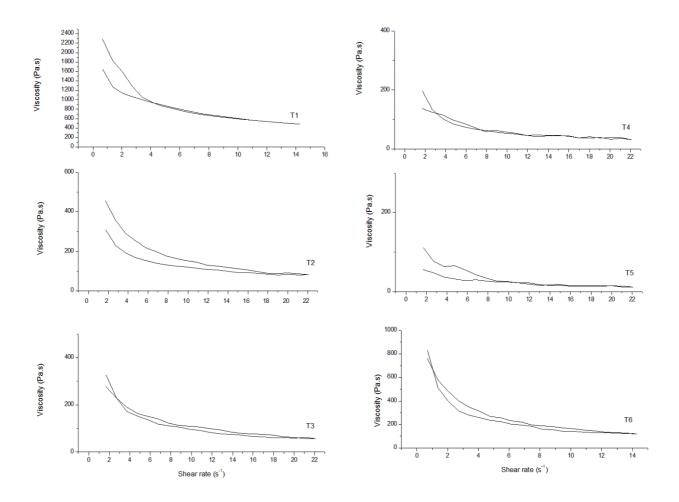


Fig. 4: Viscosity curves of mucilage samples.

The figure 5 exhibits and compare three shear rate points $(2.72s^{-1}, 7.82s^{-1} \text{ and } 14.96s^{-1})$ of the viscosity curve. At 2.72 s⁻¹, the viscosity has a downward trend once the chia mucilage concentration is raised (T1= 285.86, T2= 360.27, T3= 228.47, T4= 123.02, T5= 46.87 Pa.s). This fact is also exhibited to 7.82 s⁻¹ and 14.96 s⁻¹ shear rates, which may be regarded as an advantage since embodying of psyllium into food eventually have restrictions regarding their gelling power. Beyond that, T6 presented unexpected behaviour revealing the highest value of the blends (401.28 Pa.s). According to mass balance, the expected results were meant to be between T2 and T3. This is an indication that the aquous combined extration acts differently than a simple mucilage mixing.

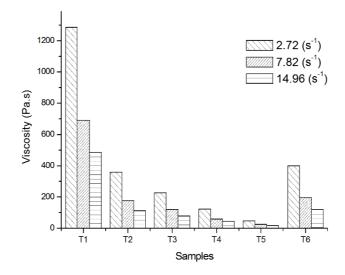


Fig. 5: Viscosity comparison of mucilage samples of three different shear rates.

3.4. ATR

Figure 6 illustrates the ATR spectra of the samples. Previous studies had obtained similar spectra for psyllium and chia mucilage. By comparing the samples, we observe that the similar broad bands ranged between 3358 to 3307 cm⁻¹, representing the hydroxyl (-OH) stretching of the gross carbohydrates; 2928 to 2886 cm⁻¹, the C-H stretching of the aromatic rings and the methyl group (CH₃); 1724 to 1726 cm⁻¹, the C=H stretching vibration of carboxylic acid; 1598 and 1420 cm⁻¹, and

the symmetric stretching of carboxyl group (-COO⁻) ion present in uronic acids. The bands at 1750 cm⁻¹ and 1155 cm⁻¹ may represent the bending vibration of C=O and C-O-C of the pyranose ring. At 1038 cm⁻¹ can be related to C-O-C stretching of 1→4 glycosidic bonds and C-O-H bending, considered a characteristic of polysaccharide compounds. The band at 844 cm⁻¹ may represent the β-nanomeric C-H deformation and glycosidic linkages attributed to glucopyranose and xylopyranose units (Cerqueira, et al. (2011), Togrul & Arslan (2003), Timilsenaa, Adhikarib, & Kasapisa (2016)). The difference between the samples occurred at the 700 range, representing cis C-H out-of-plane bends, at 1250 cm⁻¹ (C-O- stretching), 1377 cm⁻¹ (-CH₃ symmetric bend), 821 cm⁻¹ (C-O out-of-plane bend) and 1653 cm⁻¹ (>N-H of secondary amine) (Timilsena Y. W., 2016; Coates, 2000; Tipson, 1968).

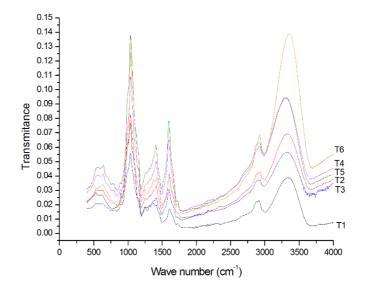
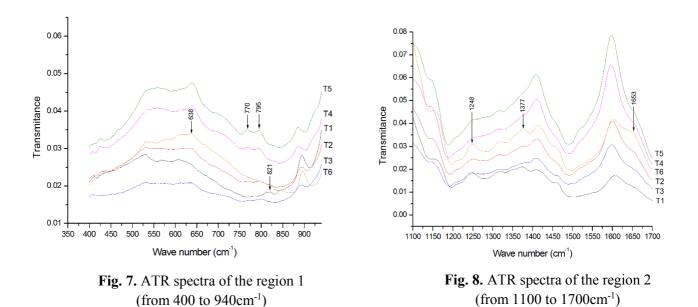


Fig. 6. ATR spectra of the mucilage samples

In general, sample bands in certain regions emerge or become more visible with the addition of the different mucilage. For example, the bands of 770 cm⁻¹ and 795 cm⁻¹, where T5 and T4 present peaks, T3 and T2 have a subtle increase and T1 revealed no evidence of a peak. Sample T6 did not present the same tendency of the mixtures having indicated a similar behavior to the psyllium mucilage (T1) in some regions. In addition to not having the above-mentioned peak (770 cm⁻¹ and

795 cm⁻¹), the similarity between samples T1 and T6 appear in peaks at 1248 and 1377 cm⁻¹. The remaining samples (T2 and T3) revealed only one increase soften with the increases in the chia mucilage concentration (T4 and T5). Differences between T1 and T6 molecules were found in bands 638 cm⁻¹, 821 cm⁻¹ and 1653 cm⁻¹. Assuming that the molecular structure, among other factors, influences the properties of a polymeric gel, the ATR spectra may explain the T6 viscosity behavior above expectation since its conformation is similar to T1 (highest viscosity values).



4. Conclusion

It is important for the industry to understand the interactions among ingredients in order to achieve optimum production or even develop a new product. This study revealed that mixing chia and psyllium mucilage results in a different behaviour when compared to the combined extraction since the viscosity results were higher than expected. For this reason, the combined mucilage is the highlight of the work. As the industry seeks to leverage factors, such as thickening and the absence of artificial ingredients addition, combined extraction processes may constitute an interesting alternative. The ATR spectra was an important instrument to investigate the causes of the increased viscosity for revealing on a molecular level the similarities of the highest viscosity samples structures.

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