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CHARACTERIZATION OF MIXTURES OF NATURAL POLYMERIC MATERIAL FOR ENCAPSULATION, BY SPRAY DRYING

CARACTERIZACIÓN DE MEZCLAS DE MATERIALES POLIMÉRICOS NATURALES PARA ENCAPSULACIÓN, MEDIANTE SECADO POR ASPERSIÓN

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Abstract: Background: The characterization of the encapsulating materials to be used in the encapsulation is very important to obtain adequate results and maintain the characteristics of the materials to be encapsulated when subjected to spray drying. Objectives: The objective of this research was to characterize a mixture of natural polymers as an encapsulating material to be used in spray-drying equipment, © Vibrasec brand. Methods: The experimental part was carried out in the facilities of the Engineering Laboratory, University of Córdoba, Montería, Colombia. The physicochemical characterization of the encapsulating material (native cassava starch, oatmeal and sodium alginate) was carried out: granulometric analysis, glass temperature, suspension analysis: pH, acidity, moisture content, apparent density and apparent viscosity, was evaluated. the gelatinization temperature at different concentrations (3.5 and 7%) and temperatures (27, 60 and 90 °C) in order to determine the appropriate temperatures and solid concentrations of the encapsulating material in mixture to prepare the suspensions that meet the drying equipment restrictions (700 mPas) © Vibrasec. Results: The results indicated that sodium alginate should be worked at a maximum of 2% solids concentration and a temperature of 85 °C, while oatmeal (7%) and native cassava starch (10%) should be worked at a temperature of 60°C. In the preparation of the solutions of the mixtures of the encapsulating material, it was established that the maximum concentration of the mixtures should be 12% to obtain a good homogenization of the mixtures and a viscosity (700 mPas) within the restriction of use of the equipment. drying © Vibrasec. Conclusions: It was possible to characterize the encapsulating material made up of oatmeal, sodium alginate and native cassava starch, establishing the appropriate conditions to be subjected to spray drying in the Marca © Vibrasec equipment.

Keywords: sodium alginate, native cassava starch, oatmeal, encapsulating material, spray drying

Resumen: Antecedentes: Las actuales tendencias están orientadas al uso de polímeros naturales como material encapsulante en la búsqueda de nuevas alternativas que mantengan la viabilidad de microorganismos a encapsular mediante el secado por aspersión. La caracterización de los materiales encapsulantes a emplear es muy importante para obtener resultados adecuados. Objetivos: El objetivo de esta investigación fue caracterizar una mezcla de polímeros naturales como material encapsulante de una mezcla de cultivos probióticos mediante uso de un equipo de secado por aspersión, marca© Vibrasec. Métodos: La parte experimental se realizó en las Instalaciones del Laboratorio de Ingenierías, Universidad de Córdoba, Montería Colombia. Se realizó la caracterización fisicoquímica del material encapsulante (almidón de yuca nativo, harina de avena y alginato de sodio): análisis granulométrico, temperatura vítrea, análisis a las suspensiones: pH, acidez, contenido de humedad, densidad aparente y viscosidad aparente, se evaluó la temperatura de gelatinización a diferentes concentraciones (3 ,5 y 7 %) y temperaturas (27, 60 y 90 °C) con el fin de determinar las temperaturas y concentraciones de sólidos adecuados del material encapsulante en mezcla para preparar las suspensiones que cumplan las restricciones del equipo de secado (700 mPas) © Vibrasec. Resultados: Los resultados indicaron que el alginato de sodio debe trabajarse máximo al 2 % de concentración de sólidos y una temperatura de 85 °C, mientras que la harina de avena (7 %) y el almidón de yuca nativo (10 %) deben trabajarse a una temperatura de 60 °C. En la preparación de las disoluciones de las mezclas del material encapsulante se estableció que la máxima concentración de las mezclas debía ser del 12 % para obtener una buena homogenización de las mezclas y una viscosidad (700 mPas) dentro de la restricción de uso del equipo de secado © Vibrasec. Conclusiones: Se logró caracterizar el material encapsulante conformado por harina de avena, alginato de sodio y almidón de yuca nativo estableciendo las condiciones adecuadas para ser sometidos al secado por aspersión en el equipo Marca © Vibrasec.

Palabras claves: alginato de sodio, almidon de yuca nativo, harina de avena, material encapsulante, secado por aspersión.

1. INTRODUCTION

Encapsulation is a technique where liquid droplets, solid particles, or gases are covered with a porous polymer film. This membrane, barrier, or film is isually made of components that form a polymer network (Fuchs et al., 2006. The structure created by the encapsulating agent around the encapsulated substance (core) is called a wall; this wall protects the core from deterioration and helps release it under desired conditions (Fuchs et al., 2006, Rodríguez, et al., 2016; Gandomi, et al., 2016; Nunes, et al., 2017).

Spray drying is the most common method of encapsulating food ingredients, as it is more cost-effective compared to other techniques (Arslan, et al., 2015; Eckert, C. et al., 2017), and due to equipment availability and the food stability of the final product (Gul, 2017; Ceja-Medina et al., 2020); This technique can be applied to water-soluble materials, fish oils (DHA y EPA), natural pigments, probiotic cell concentrates, and powdered milk (Ceja-Medina, et at., 2021; Sun, et al., 2023). In spray drying, the selection of encapsulating agents or coating materials is critical, as they can affect the the properties of the emulsion before drying, particle size, and flow properties, as well as the retention of volatile compounds during the process and the shelf life of the powder after drying.

An important factor is the encapsulating agents used, among which we find types, such as lipids: milk fat, lecithins, waxes, stearic acid, monoglycerides, diglycerides, and hydrogenated oils; these are excellent film-formers capable of coating individual particles, providing uniform encapsulation. Carbohydrates are widely used, primarily in spray drying techniques, as encapsulation support. This group includes starches, maltodextrins, and gums. Currently, research is being conducted on the use of polymeric microencapsulation matrices derived from polysaccharides and their possible combinations to ensure the proper preservation of the encapsulating layer and the active material to be encapsulated (Nie, 2013; Esquivel-González, et al., 2015; Fritzen-Freire, et al., 2021; Madsen, et al., 2022; Mikkel et al., 2022).

The protective material must possess certain properties that depend on the chemical characteristics of the encapsulated material, its application, storage conditions, and the process, as outlined by (Nag, 2011; Moumita, et al., 2017; Homayouni-Rad et al., 2021 he first step in encapsulation is selecting an appropriate encapsulation matrix (Rios-Aguirre, y Gil-Garzon, 2021; Tamtürk, et al., 2023). Research on this topic focuses on the search and application of new coating materials and novel encapsulation techniques (Ávila-Reyes, 2014; Ceja-Medina, et al., 2021; Barros, et al., 2022).

In this research, three materials were evaluated, chosen for their encapsulating and prebiotic properties, and subjected to spray drying using a Vibrasec© brand machine. Native cassava starch (*Manihot sculenta Crantz)* and sodium alginate have been studied for their ability to be used as wall materials for encapsulation, with a wide particle size distribution (Aristizábal, y Sánchez, 2007; Lupo-Pasin et al., 2012; Etchepare, et al., 2016). In the encapsulation of probiotics, these materials are used in moderate concentrations; however, they exhibit low resistance to gastric acids. This characteristic can be counteracted by modifying the gel structure, using mixed polymer systems for matrix formation, or coating the macroparticles (Lupo-Pasin et al., 2012; El-Sayed, et al., 2017; Tao, T., et al., 2019; Triviño, 2019).

Oat flour contains starches, β - α Glucans and small amounts of proteins, making it a prebiotic material with the potential to form strong, resistant, and flexible films to improve the properties of sodium alginate and native cassava starch (Ta, et al., 2021). The beta-glucan in oats may lead to an FDA-approved health claim (Yonekura, et al., 2014; Yuan, et al., 2023 17). Several studies have published some physicochemical and rheological characteristics of the main components of oats, with notable work conducted by (Berski, et al., 2011; Zamora-Vega, et al., 2012; Zamudio-Flores, et al., 2015; Flores-Peña, et al., 2013).

Research on this topic focuses on the search for and application of new coating materials to be subjected to spray drying. Therefore, this study aimed to evaluate the physicochemical characteristics (solid content and gelatinization temperature) of a mixture of natural polymeric materials with prebiotic properties to encapsulate a mixture of microorganisms using the spray drying technique, in order to determine the appropriate concentration while meeting the viscosity restrictions of the Vibrasec© equipment.

2. MATERIALS Y METHODS

The experimental work was conducted at the Nanotechnology Laboratory, Food Technology Center at the University of Pamplona, the Applied Engineering Laboratory, and the Bioprocesses and Fermentations Laboratory at the Berastegui campus of the University of Córdoba, Colombia.

The encapsulating agents used were: Native cassava starch (INNOVAYUCA), sodium alginate (SQ 942), and ground oat flour.

2.1. Characterization of encapsulating material and solutions.

The physicochemical evaluation of the encapsulating material and the solutions was carried out as described below:

Table 1: Analysis conducted on encapsulating materials and solutions

Evaluated Parameters	Method Used
Particle size analysis	AOAC. 965.22, (1995)
Moisture content (%)	Gravimetric method by volatilization
Bulk density (g/mL)	Goula, A. M., and Adamopoulos, K. G. (2012)
Glass transition temperature	Paredes-López, O., et al., (1994).
Apparent viscosity of solutions	Brookfield viscometer Nielsen (2010)
Apparent density of solutions	A.O.A.C. 962.37, (1995)
pH	Potentiometric method AOAC 981.12 (2012)
Acidity as lactic acid (% m/m) min	Titration with NaOH AOAC. (942.15, 2005)

Once the solid concentration and glass transition temperature (Tg) of the three encapsulating materials were established, tests were conducted to determine the maximum solid concentration to be used for each material individually and with the mixture of the materials in suspension: heating tests at different solid concentrations against the apparent viscosity to meet the viscosity requirements for the use of the Vibrasec© equipment, technique employed by (Nielsen, 2010; Aristizábal, y Sánchez, 2007).

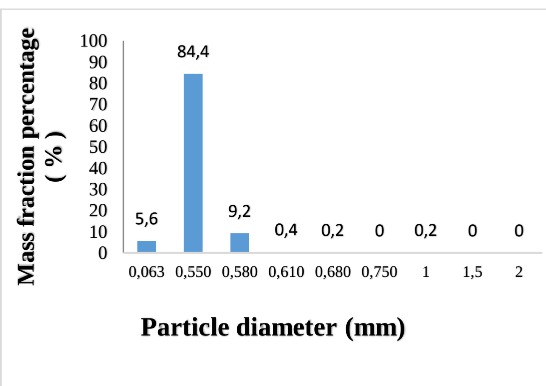
2.2 Statistical evaluation of the data

All measurements for the statistical analyses were performed in triplicate. To statistically evaluate each of the experiments conducted, significant differences between treatments were determined by performing an ANOVA analysis with a 95% confidence interval and a Tukey multiple comparisons analysis of the average values.

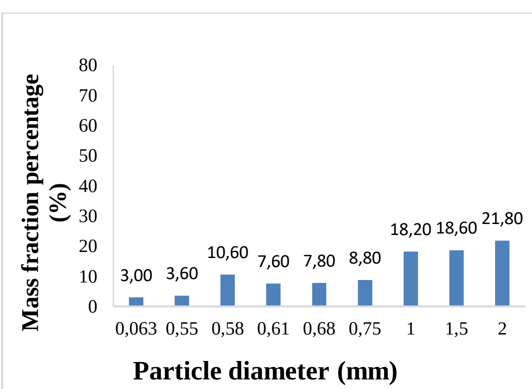
3. RESULTS

3.1. Particle size analysis of oat flour and native cassava starch

The particle size analysis conducted on the encapsulating materials: oat flour and native cassava starch, allowed for the determination of the particle size distribution, (Flores-Peña,, et al., 2013; Aristizábal, y Sánchez, 2007).



(a)



(b)

Fig. 1. Particle diameter distribution of (a) oat flour and (b) native cassava starch.

Figure 1 shows the particle diameter distribution of oat flour, with 84.40% having a particle size of 0.55 mm and an average diameter of 0.53 mm, corresponding to a finely ground product (49, 66). The particle size and surface layer of oat flour, due to its high content of dietary fiber, influence the physicochemical properties of the required encapsulating matrix, promoting dissolution and homogenization with the other materials to be mixed. Similarly, the particle size analysis of native cassava starch (Manihot sculenta Crantz) is presented, showing a non-homogeneous particle size distribution, where 58.6% fell within the range of 1 to 2 mm and an average interval of 0.54 mm (41.4%), resulting in an average particle diameter of 1.14 mm for cassava starch, which corresponds to a very fine powder (Flores-Peña, et al., 2013; CODEX STAN 176. 1989; De Araujo, et al., 2016).

The results of the particle size analysis of the encapsulating materials show differences in particle size between oat flour and native cassava starch (*Manihot sculenta Crantz)*, From fine to very fine, an aspect that was considered for the mixing process, establishing that the preparation of the encapsulating matrix should be performed in a wet manner (dissolution) to prevent the phenomenon of disaggregation, which would hinder mixing. This is in line with what was stated by (Esquivel-González, et al., 2015) who established that a greater number of pores allow for increased particle hydration, and an available surface facilitates the binding of cations; hence the importance of fine granulation in the encapsulating materials to be used.

3.2. Physicochemical indicators of the encapsulating materials

Table 2 presents the results of the analyzed indicators:

Table 2: Physicochemical indicators of the encapsulating materials

Encapsulating Material N°	Bulk Density (g/mL)	Moisture Content (%)	pH Value ---
Oat flour	1.082 (0.01)	14.67 (0.01)	7.0 (0.01)
Native cassava starch	1.530 (0.03)	12.53 (0.04)	6.5 (0.03)
Sodium alginate	0.990 (0.01)	12.27 (0.07)	7.0 (0.02)

Reported average values correspond to 3 determinations.

As shown in Table 2, the encapsulating materials exhibited pH, bulk density, and moisture content within the ranges of the standards (García-Ceja, and López-Malo, 2012; Madsen, et al., 2022) making them suitable for forming capsules of different sizes due to the molecular interactions of the various polymeric materials to be combined (Barbosa-Cánovas, 2005; Lupo-Pasin, et al., 2012; Arslan, S., Erbas, M., Tontul, I., Y Topuz, 2015).

3.3. Differential scanning calorimetry (DSC) of the encapsulating material

Table 3 presents the glass transition temperature (Tg) values of the encapsulating material.

Table 3: Glass transition temperatures (Tg) of the encapsulating materials.

Encapsulating Material	Glass Transition Temperature, Tg (°C)
Sodium alginate	85
Native cassava starch	63
Oat flour	62

The calorimetry (DSC) results allowed for the evaluation of whether the encapsulating materials to be used exhibited incompatibility within the formulations of both the individual components and the mixture. As shown in Table 3, the glass transition temperature of the three materials is in the range of 62 - 85 °C, which favors obtaining suitable conditions for the solutions when making the different mixtures (Perdomo, et al., 2009; Ceja-Medina, et al., 2020). This result indicates that the feeding temperature of the encapsulating materials should be maintained at temperatures below 63 °C for native cassava starch (*Manihot sculenta Crantz)* and oat flour could be at 60 °C to facilitate its solubilization without gelatinizing (Richardson, et al., 2000), meanwhile, sodium alginate should be processed at a temperature below 85 °C, so they should be prepared separately before mixing.

3.4. Apparent viscosity of the encapsulating materials

One of the restrictions of the drying equipment (© Vibrasec) for its use was that the viscosity of the encapsulating materials should not exceed 700 mPa.s. Figure 2 graphically presents the behavior of the apparent viscosity of the three encapsulating materials at the three evaluated concentrations.

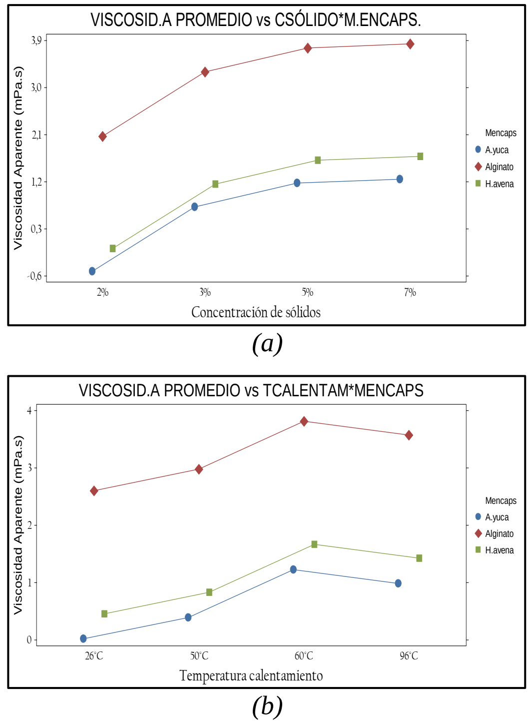


Fig. 2. Behavior of the apparent viscosity of the three encapsulating materials (a) versus heating temperature and (b) solid concentration.

The statistical analysis with p-values < 0.05 indicates that the apparent viscosity varies according to the encapsulating material, heating temperature, and solid concentration evaluated (see Figure 2).

Table 4 presents the results of the Tukey multiple comparisons analysis of the average values.

Table 4: Summaryof the Tukey multiple comparisons analysis of the average apparent viscosity values

Statistical technique: ANOVA					
Multiple comparisons test: Tukey					
Significance level: α =5%					
Statistical software: Statistix versión 10					
Variables	**Factors**	**Factor Level**	**Mean**	**P-Value**	**Interpretation**
APPARENT VISCOSITY	**Encapsulating material**	Alginate	3,239a	0.000	There are differences
		Oat flour	1,092b		
		Cassava starch	0,654c		
	Solid concentration (%)	7	2,2546a	0.001	There are differences
		5	2,1796a		
		3	1,7218b		
		2	0,4923c		
	Heating temperature (°C)	60	2,2327a	0.000	There are
		50	1,9927ab		
		96	1,3978b		

	26	1,0251b	differences

The highest average apparent viscosity was observed with sodium alginate (Nielsen, 2010; Holkem, et al., 2016), followed by native cassava starch and oat flour, with the latter showing the lowest viscosity at the evaluated temperatures and concentrations. Similarly, the average apparent viscosity varies across the three materials depending on the solid concentration. At 7% and 5% concentrations, a statistically similar viscosity is produced between oat flour and native cassava starch (Karimi, et al., 2023) (see Table 4).

On the other hand, the apparent viscosity of the three materials varies according to the heating temperature. It was observed that oat flour and cassava starch exhibited similar viscosities at temperatures between 50 and 60 °C, while sodium alginate showed the highest viscosity values as the temperature and solid concentration increased. Additionally, the highest average viscosity was observed at 60 °C (De Araujo, et al., 2016).

Table 5 shows the results of the viscosity of sodium alginate solutions at temperatures of 60 and 96 °C, considering the water absorption capacity at different solid concentrations of 2% and 3% (w/v), given that the maximum reported value for its use as an encapsulating material for probiotics has been 5% (Rios-Aguirre, et al., 2021).

Table 5: Apparent viscosity of sodium alginate at different solid concentrations and temperatures.

Encapsulating agent	**Apparent viscosity (mPa.s)**			
	Solid concentration/ Temperature (°C)			
	2 / 60	**2 / 96**	**3 / 60**	**3 /96**
Sodium alginate	404.6 (0.01)	1,040.6 (0.01)	4,296.6 (0.01)	7,490.0 (0.01)

Reported average values correspond to 3 determinations. p-value <0.05

The results in Table 5 show that for sodium alginate, the apparent viscosity increased from 404.6 to 7,490 mPa.s in the temperature range of 60 to 96 ± 1 °C, exceeding the maximum allowable value (700 mPa.s) for the use of the drying equipment. Therefore, it was determined that the sodium alginate solution should be prepared at a solid concentration not greater than 2% with constant stirring to facilitate its solubility.

Results corroborating those presented by (Perdomo, et al., 2009; Wan et al., 2010; Mikkel, et al., 2022) indicate that the viscosity of alginate solutions depends on the concentration. From 2% onwards, the viscosity of the solution increases significantly and exhibits non-Newtonian behavior, meaning its viscosity is not constant.

Table 6 presents the results of the apparent viscosity of native cassava starch and oat flour solutions at concentrations of 3%, 5%, and 7% at a temperature of 60 °C.

Table 6: Evaluation of the apparent viscosity of native cassava starch and oat flour at different solid concentrations at 60 °C

Material	**Apparent viscosity (mPa.s)**		
	Solid concentration (%)		
	3	**5**	**7**
Native cassava starch	189.2 (0.07)	356.7 (0.01)	653.7 (0.01)
Oat flour	6.8 (0.00)	10.1 (0.00)	34.5 (0.00)

Determinations were performed in triplicate, reporting the average value. p-value < 0.05

As shown in Table 6, the viscosity of native cassava starch increases with increasing concentration and does not exceed the maximum required viscosity value (700 mPa.s); thus, native cassava starch can be used at a maximum concentration of 7% at a temperature of 60 °C. A gradual increase in the apparent viscosity was also observed as the concentration of oat flour in the dispersion increased, with average viscosity values ranging from 6.8 to 34.5 mPa.s, which are below the value set as a restriction. Therefore, oat flour can be used at concentrations above 7% at a temperature of 60 °C.

These results coincide with those reported by Dikeman and Fahey (2006), Lazaridou and Biliaderis (2007), Regand et al. (2011), Rodríguez-Restrepo et al. (2017), and Zamudio-Flores et al. (2021), who indicate that the increases in apparent viscosity values among the dispersions are directly associated with the higher fiber content in the greater concentrations due to the presence of β-glucans in oat flour.

Table 7 shows the apparent viscosity of the suspensions of encapsulating material mixtures at the defined working temperature (60 °C) at different solid concentrations.

Table 7: Apparent Viscosity of Suspensions of Encapsulating Material Mixtures

Concentration of solids	Sodium Alginate	Native Cassava Starch	Oat flour	Apparent Viscosity
%	%	%	%	mPa.s
16	1	7	8	796 (0.3)
14	1	7	6	693 (0.4)
12	1	5	6	495 (0.6)
10	1	5	4	253 (0.4)

Determinations were performed in triplicate, and the average value is reported. p-value < 0.05.

As observed in Table 7, for mixture solid values of 16%, the viscosity was above 700 mPa.s, not meeting the restrictions required by the drying equipment. Meanwhile, the mixtures with solid concentrations in the range of 10 to 14% had viscosity values lower than the declared working restriction.

Table 8 shows the behavior of the encapsulating material mixtures subjected to spray drying in the Vibrasec equipment.

Table 8: Behavior of the Mixture of Materials in Suspension at Different Solid Concentrations in the Vibrasec Equipment

Solid concentration (%)	Inlet Air Temperature (°C)	Atomization Speed (min^{-1})	Behavior in the Chamber	Moisture Content (%)
20	200	35000	Saturation	---
16	200	35.000	Saturation	9.23
14	200	35.000	Saturation	8.68
12	200	35.000	Normal	4.58
10	200	35.000	Normal	4.32

Based on these results, the mixtures of the encapsulating material were subjected to the spray drying process at a feed temperature of 60 °C and inlet air temperatures of 200 °C. As observed in Table 8, solid concentrations of 14% and 16% exhibited saturation in the chamber and a moisture content between 8.68% and 9.23%. These results align with the findings of Rios-Aguirre et al. (2021), Salinas et al. (2021), and Tamtürk et al. (2023), who indicate that the inlet air temperature is directly proportional to the drying rate of the microcapsules and the final water content. Higher adsorbed moisture leads to greater cohesive forces between particles, resulting in increased contact points among them, which can indirectly affect the dispersion rate of the encapsulating materials, as reflected in the saturation of the drying chamber. In contrast, solid concentrations of 10% and 12% showed normal behavior in the chamber, with moisture content between 4.58% and 4.32%.

This result allowed for the selection of the 12% solid concentration as the most suitable mixture of the encapsulating material in suspension to be used in spray drying under the evaluated conditions of the Vibrasec equipment, discarding the 10% concentration due to its considered low solid content (Soto et al., 2023; Tamtürk et al., 2023).

4. CONCLUSIONS.

It is concluded that oat flour, native cassava starch, and sodium alginate possess physicochemical properties that favor their use as encapsulating materials, with the selection of a 12% solid concentration as the most suitable mixture of encapsulating material in suspension to be used in spray drying.

CONTRIBUTIONS OF THE AUTHORS

All authors contributed equally to the development and writing of this research.

DECLARATION OF COMPETING INTERESTS

The authors declare that they have no conflicts of interest.

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