

# Development of microparticles from biopolymer obtained from potato, carrot and chayote peel waste to be use in cosmetic formulations

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## Abstract

**Background:** Starch is present in discarded vegetable peel and it can be used as material for microparticles production. The purpose of this study was to develop microparticles of biopolymer obtained from peeled potato, carrot and chayote waste to be used as actives carrier in cosmetics.

**Methods:** Potato, carrot and chayote peels were donated from the university canteen. They were weighed, immersed in sodium metabisulfite solution (0.2%; w:v) for 24h, and after they were ground in a blender. The material obtained was filtered and starch was separated by decantation and dried. The starch was subjected to characterization assay. The microparticles were produced using 0.1g of potent starch (mixture of three plant species; 1:1:1) in 10 ml of water at 80°C magnetic stirring. The mixture was subjected to stirring with ethanolic solution (1:2; v:v) for 4h and the precipitate was centrifuged. The microparticle solution had its physical and chemical characteristics evaluated. **Results:** Starch obtained from potato, carrot and chayote waste peels showed white, orange and green colors, respectively, and a yield of 3.23%, 0.62% and 1.17%. Microparticles presented a slight orange color, exhibited zeta potential value of  $-27.96 \pm 1.0$  mV, the average particle size of 4494.37nm, PDI index of 0.2 and in the scanning electron microscopy analysis presented spherical shape. Fourier transfer infrared spectroscopy analysis showed peaks similar to literature. **Conclusion:** The peels waste present starch and have capacity to be used as material to develop microparticles using waste contributing to dispose of waste and reduction of environmental impact.

**Keywords:** biopolymer, cosmetics, microparticles, starch, vegetal peels, waste.

## 1. INTRODUCTION

Biopolymers or natural biopolymers are formed by long monomers chain of the same kind or a combination of different monomers [1]. These polymers are produced from alive organisms, that can be classified in three groups: polysaccharides, proteins, and nucleic acids [2,3].

In accordance to the same authors, the biopolymers contain many favourable characteristics in comparison to synthetic polymers used as material of pills with a prolonged liberation of actives, such as, biocompatibility and biodegradability and for having abundant renewable sources [3].

Besides, the association of the degradability process of these materials and their renewable sources promotes a lesser waste amount and a lesser environment impact, and these materials are easily degraded during the industrial processing.

Therefore, the natural polymers present a low toxicity, high stability, flexibility for chemical changes, low cost and they are biodegraded, justifying their attractive and vast application as drug carriers [4].

Micro and nanotechnology is a knowledge field with a big scientific and economic impact, which represents an important highlight in the health area, considering the use of implants and the system of drugs control liberation [5].

Then, microtechnology contains the particles manipulation of nanometric size from 1 to 1000 nm, creating the microparticles, which are normally created by synthetic materials such as copolymers of methacrylic acid, acrylic esters or methacrylic [6, 7], and they can be in the shape of microcapsules, which are a reservoir system (capsules) [8]; or microspheres (encapsulated), which are formed by a polymeric matrix [9], involved by a membrane, usually polymeric, isolating the nucleus from the external outside [8].

The microparticles obtained from natural material can be constituted of biodegradable polymers from plant material (rubber, cellulose, and wood), animals (leather), and alive organisms (proteins and nucleic acids) [10].

Starch is an example of natural polymer or biopolymer, classified as a reserve polysaccharide present in vegetables, such as potato, chayote, carrot, and manioc, in carbohydrate reserve form [11].

It is constituted of two macromolecules: amyloses, made of a linear chain, water insoluble; amylopectin, made of ramification chains. Both structures present D-glucopyran monomers [12].

However, starch can provide a formation of a viscous solution when exposed to hot water, becoming adequate to the production process of gel formulation, polymeric pellicle, and microparticles [11].

The particles of the polymeric wall load and protect active components, such as vitamins and polyphenols, for example, avoid a possible instability originated by light and/or heat oxidation, and unexpected reactions from the outside in contact with substances, thus providing a protection to the active, increasing its efficacy, promoting the controlled and gradual liberation of them in the organism, when desired, the expressive decrease of the toxicity by reducing the plasmatic peaks of maximum concentration [13,14].

As seen, the spreading of active substances in microparticles have been an important alternative to increase the efficiency and security of actives. In addition, the biopolymers use becomes interesting, because it presents a lot of favourable characteristics in comparison to synthetic polymers such as high biocompatibility and biodegradability and for having abundant renewable sources [3].

Furthermore, the association of the degradation process of these materials and their renewable sources promotes a lesser waste amount and lesser environmental impact, being easily degraded during the industrial processing [15].

Then, the biopolymers use obtained from waste material, such as food peels, becomes the present work a promise for the development of new formulations or material for the confection of microparticles, impacting positively in the discard of waste, biocompatibility and biodegradability of compounds contained in formulations of topic use.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Plant material containing starch, such as potato peels (*Solanum tuberosum*), chayote peels (*Sechium edule*) and carrots peels (*Daucus carota*). For the production and characterization of the biopolymers, nanoparticles and formulation were used Sodium Metabisulfite, Glycerin, Absolute Ethyl Alcohol, Water, Octyl Stearate, Cetereth-20, Cetearyl Alcohol, Diazolidinyl urea, Potassium Hydroxide, Potassium iodide, Acetic acid, Sodium hydroxide and Sodium chloride. The glassware used were beakers, spatulas, pipettors, pipettes, volumetric flasks, thermometer, heating source, pycnometer, baguette, beaker, Buchner funnel, filter paper, kitassat, ring, clamp, plastic spatula, metal beaker and watch glass. The equipment used were: magnetic stirrer (IKA RCT basic, Brasil), analytical balance (Quimis, Q500B210C, Brazil), microprocessor centrifuge (Quimis, Q222TM, Diadema- SP, Brazil), air circulating oven (7Lab, SEDI-C 40L, Brazil), refrigerator (Electrolux, Duplex DC49A – 462 L, Brazil), vacuum pump (Edwards, Bomba de vácuo de diafragma D-LAB, Tamboré Brazil), blender (Walita Philips, ProBlend 4 Philips RI2004/91, Brazil, 2004), dynamic light scattering equipment (Nano ZS, Malvern Instruments Ltd., Malvern, UK), infrared spectrophotometer (Femtd, 800XI, São Paulo- SP, Brazil), peagometer (Marte, MB-11, Brazil), viscosimeter (Quimis, Q860M21, Diadema Brazil), and scanning microscope (Tescan, Vega 3 SBH, Shanghai, China).

### 2.2 Methods

#### 2.2.1 Starch extraction (adapted from Vericimo, 2010)

The vegetable peels were selected, washed with running water, dried, and weighed in order to calculate the yield obtained from the process. The peels were immersed in an aqueous solution of Sodium Metabisulfite (0.2%) for 24 hours and, after this period, the material obtained was filtered and grinded with distilled water in a blender (Walita Philips RI2004/91, Brazil, 2004). The obtained mass was vacuum filtered (Edwards, D-LAB diaphragm vacuum pump, Tamboré, Brazil) and the starch was separated from the water by decantation and dried in an air circulation heater (7Lab, SEDI-C 40L, Brazil) at 60°C for 20 hours [18].

#### 2.2.2 Process yield

To calculate the starch process yield, there was determined a relation between the peels initial weight and the final starch weight obtained. The starch extraction yield was calculated by the following equation [19]:

$$Yield (\%) = 100 \times \frac{IPM}{FSM}$$

*IPM= Initial Peels Mass*

*FSM= Final Starch Mass extracted from the peels.*

## **2.2.3 Characterization of starch**

### **2.2.3.1 Determination of amylose content by spectrophotometry**

To determine the amylose content, 100 mg of the starch sample were added in a 100 mL volumetric flask and 1 mL of 95% (v/v) ethyl alcohol and 9 mL of 1 mol/L NaOH solution were added. After 12 hours, the starch gelatinization was observed. After this period, the volume of the volumetric flask will be completed with distilled water. An amount of 5 mL of the solution was transferred to another 100 mL volumetric flask and 1 mL of 1 mol/L acetic acid and 2 mL of potassium iodide solution was added (0.2 g of I<sub>2</sub> and 2g of KI for 100 mL of solution). The presence of starch was verified by the formation of a blue colored product and the optical density was identified by spectrophotometer (Femtd, 800XI, São Paulo- SP, Brazil) in the visible region at 620nm [19,20]. The amylose content was calculated from the calibration curve using standard amylose.

### **2.2.3.2 Analysis by Fourier transfer infrared spectroscopy (FTIR)**

The spectra obtained from the starch sample from Fourier transfer infrared spectroscopy spectrometric analysis were recorded on FTIR spectrophotometer (Femtd, 800XI, São Paulo- SP, Brazil). The samples were submitted to KBr addition, pressed into pellets, and then analyzed in a range of 4000-500 cm<sup>-1</sup> [22].

### **2.2.3.3 Scanning electron microscopy (SEM)**

The microstructure of starch was observed using an electron microscope scanning (Tescan, Vega 3 SBH, Shanghai, China). Starch granules was its morphologies photographed. Digital image analysis was used to determine the average dimension of the particles [22].

### **2.2.3.4 pH analysis**

To determine the pH value, 0.6 g (2%) of starch was dispersed in 30 ml of pure water and the solution stirred for 30 min. After the stirring the pH was immediately measured in a calibrated pHmeter (Marte, MB-11, Brazil) at room temperature (20°C) [23].

### **2.2.3.5 Viscosity analysis**

The viscosity of aqueous starch dispersions was verified using a rotation viscometer (Quimis, Q860M21, Diadema Brazil) he aqueous starch dispersions at concentrations ranging from 0,5 to 3% (w/v) were then heated until the starch gelled and analyzed at room temperature. The viscosity values were expressed in Pa.s [24].

## **2.2.4 Preparation of microparticles containing starch extracted from vegetable peels**

To develop the microparticles, a dispersion of 0.1 g of starch obtained from vegetable peels was prepared in 10 ml of pure water obtained. The sample was stirred in a magnetic stirrer (IKA RCT basic, Brasil) at 80°C until complete gelatinization of the starch. The dispersion was then co-precipitated in ethanol in a 1: 2 ratios under magnetic stirring. Thereafter, the sample was kept for 4h at room temperature under continuous mechanical stirring. The precipitate obtained was centrifuged (Quimis, Q222TM, Diadema- SP, Brazil) at 3000 rpm for 15 minutes. The starch samples will be stored at 4 °C for further analysis [22, 25].

## **2.2.5 Characterization of microparticle**

### **2.2.5.1 Determination of particle size, zeta potential and polydispersity index**

Particle size, polydispersity index, and zeta potential were analyzed by dynamic light scattering (DLS) (Nano ZS, Malvern Instruments Ltd., Malvern, UK). The light intensity was transformed into diffusion factor and the average of 10 measurements were obtained in triplicate and at room temperature [26, 27, 28].

### 2.2.5.2 Scanning electron microscopy (SEM)

The microstructure of microparticle was observed using an electron microscope scanning (Tescan, Vega 3 SBH, Shanghai, China). Starch granules had its morphologies photographed. Digital image analysis was used to determine the average dimension of the particles [22].

### 2.2.5.3 Analysis by Fourier transform infrared region spectroscopic (FTIR)

The spectra obtained from the microparticle sample from the Fourier transform infrared region spectroscopic analysis were recorded in an FTIR spectrophotometer (Femtd, 800XI, São Paulo- SP, Brazil). The samples were subjected to KBr addition, pressed into pellets, and then analyzed in a range of 4000-500 cm<sup>-1</sup> [22].

### 2.2.6 Development O/W emulsion containing starch from vegetable peels

An oil-in-water (O/W) emulsion (Table 1) was developed using the universal method for preparing emulsions. The starch obtained from vegetable peels was added to the formulation in aqueous phase at a concentration of 5%. However, the aqueous phase was transferred to a beaker and heated to 75°C. The oily phase was separated into another beaker and heated to 60°C. Subsequently, the aqueous phase was transferred to the oil phase and stirred manually until the emulsion production and until it cooled.

Table 1: Composition of oil-in-water emulsion.

Ingredients (INCI Name)	Concentration (%)
Cetearyl Alcohol	10.0
Ceteareth-20	6.0
Octyl Stearate	3.0
Glycerin	2.0
Diazolidinyl Urea	1.5
Aqua	87.5

### 2.2.7 Statistical Analysis

All assays were performed in triplicate. Statistical analysis was performed using the ANOVA test ( $p < 0.05$ ) for independent variables and the Origin version 8 software of Windows.

## 3. RESULTS

### 3.1 Starch extraction

Power starch obtained from potato, carrot and chayote waste peels presented white, orange and green colors, respectively, according Fig.1.

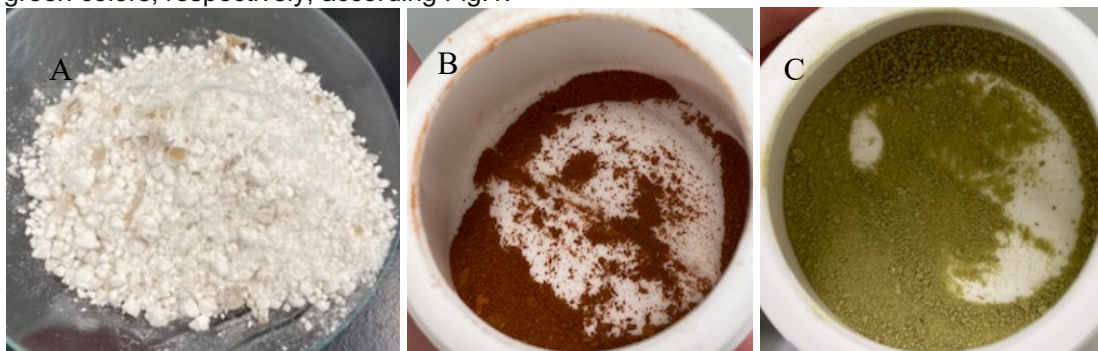


Figure 1: A- Starch potato; B- Starch chayote; C- Starch carrots.

In addition, the starch extracted from potato, chayote, and carrot peels showed productivity yield of 3.23%, 1.17%, and 0.63%, respectively.

### 3.2 Characterization of starch

#### 3.2.1 Determination of amylose content by spectrophotometry

The amylose concentration expressed in sample, constituted by the starch from three vegetable, was of  $20.02 \pm 0.094 \mu\text{g} \cdot \text{mL}^{-1}$ , and the percent amylose of 4.99%. The data was determined for amylose ( $y = 0.0221x - 0.0044$ ;  $R^2 = 0.998$ ; Fig.2) analytical curves [31].

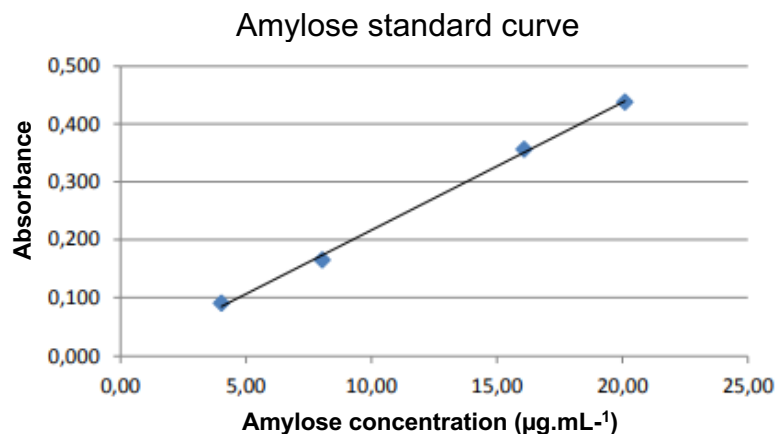


Figure 2- Amylose absorbance standard curve [32].

#### 3.2.2 Analysis by Fourier transfer infrared spectroscopy (FTIR)

Fig. 3 shows the infrared spectrum of starch extracted from potato, carrot, and chayote peels, and the similarity of spectra can be observed, confirming the starch presence according to identification of functional groups, such as OH, C-H, C-O-C and C-O and C=O described in Table 2.

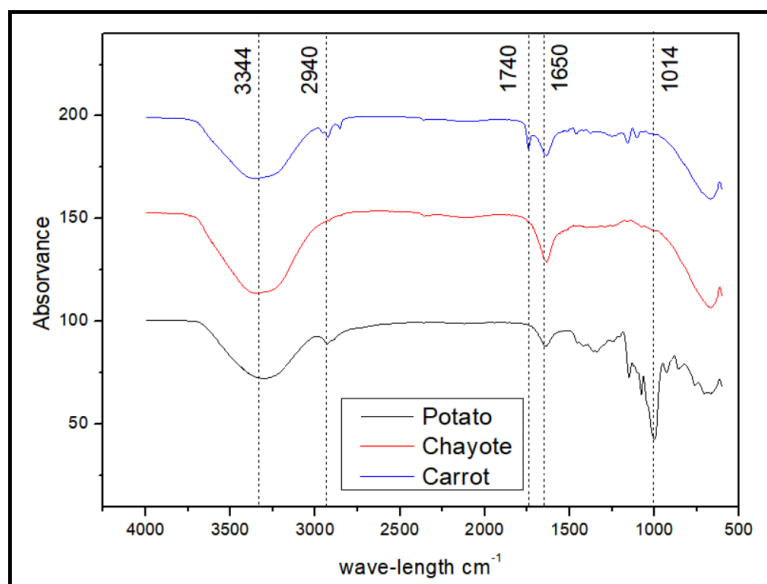


Figure 3 - Infrared spectrum of starch extracted from the peels.

Table 2: Identification of the functional groups at their respective wavelengths

Functional groups	Wavelength $\text{cm}^{-1}$
OH	3000- 3600
C-H	2928
C-O-C/ C-O	900-1160
C=O	1740

### 3.2.3 Scanning electron microscopy (SEM)

In scanning electron microscopy analysis, samples were analyzed individually, to identify the presence, shape and size of starch extracted from different peels.

The chayote microscopy (Fig.4A and 4B) showed lower concentration and size of starch grains compared to the potato and rounded shape. The potato peel sample (Fig.4C and 4D), expressed the highest concentration of grain starch in SEM assay, different sizes, shapes and a greater agglomeration of grains. The carrot (Fig.4E and Fig.4F) expressed low concentration of starch grains, being difficult to observe to grains shape and size.

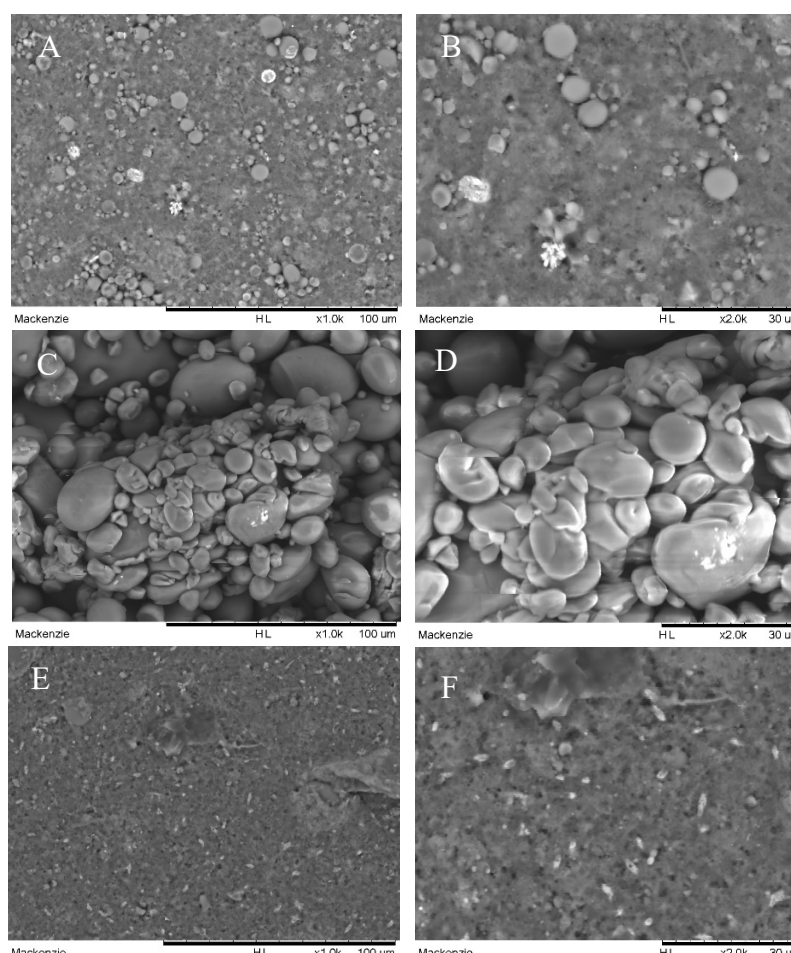


Figure 4- Microscopy of carrot chayote starch, (A) 1.0kx and (B) 2.0kx; potato starch (C) 1.0kx and (D) 2.0kx; and carrot starch (E) 1.0kx and (F) 2.0kx, respectively.

### 3.2.4 Determination of pH and viscosity values

Dispersion of starch sample containing from vegetable peels (1:1:1) presented pH value of 5.48. For viscosity analysis, five starch dispersions were analyzed (Table 3). It was observed a gradual increase of viscosity when starch concentration was increased (Fig. 5).

Table 3: Table of starch concentration in the sample and the viscosity of the samples.

Concentration (g)	Viscosity (Pa.s)
0.25	0.005
0.5	0.008
1.0	0.021
1.5	0.060
2.0	0.097

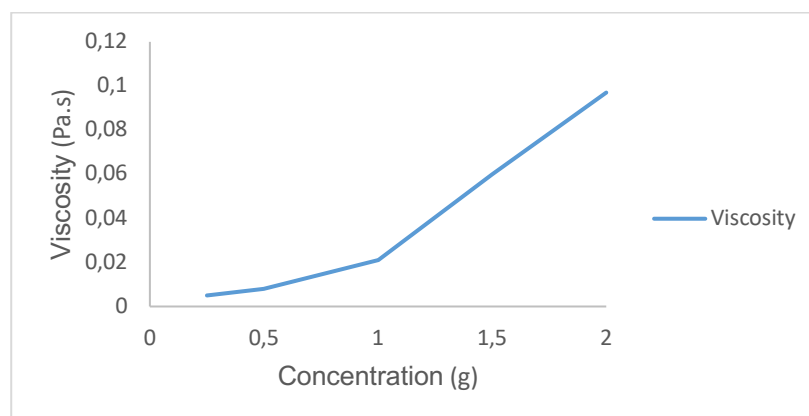


Figure 5: Viscosity determination of different concentration of starch sample.

### 3.2.5 Preparation and characterization of microparticles containing starch extracted from vegetable peels

Microparticles showed an orange coloration, zeta potential value of  $-27.96 \pm 1.0$  mV, particle size of 4494.37nm, micrometric morphology, spherical shape and PDI index of 0.2.

FTIR analysis shows the presence peak recorded in the  $3660\text{--}3300\text{ cm}^{-1}$  range attributed to hydrogen-bonded O-H (-OH group of glycoside unit) stretching vibration. This data was found in spectrum of all vegetal species. Peaks for C-O stretching vibration were found at around  $1000\text{ cm}^{-1}$  (axials deformation for C-O-C systems and C-O ligations in alcohol) in all peels samples and in microparticles sample, however, in more concentration in potato sample. Peak for C=O bending vibration at  $1622\text{ cm}^{-1}$  were also found in microparticles and specially in carrot and chayote peels samples.

### 3.2.6 Development O/W emulsion containing starch from vegetable peels

One O/W emulsion presented creamy aspect, lightly orange color and characteristic odor (Fig.6). The pH value found was of 5.76 and density of  $0.97\text{ g.ml}^{-1}$  value.



Figure 6: An O/W emulsion containing starch from potato, chayote and carrot peels.

## 4. DISCUSSION

Starch is a biopolymer widely present in vegetables [11], and it can be possible find in peels, a waste kind possibly discarded. According Nitta and Numata [3], biopolymers contain many favorable characteristics such as biocompatibility and biodegradability and presents abundant renewable source. In addition, the degradation process of these materials and their renewed sources promote a lower waste amount and lower environment impact [39].

Therefore, these characteristics become the biopolymers application attractive in cosmetic formulations [38].

In this study, starch from potato, carrot and chayote peels were donated by one of the cafeterias located on Mackenzie Presbyterian Mackenzie, being one material from waste discarded by University. The starch obtained by waste peels showed white colors for the potato sample, orange, due to the beta-carotene present in carrots, and green, due to the chlorophyll present in chayote.

The extracted process also showed a productivity yield data similar to that of Usa et al [30], in which a productivity output of 4.9% was determined for potato starch.



The starch extracted from potato, carrot and chayote peels presented an amylose concentration value similar to found by Bandeira et al. [31], in which the result was of  $10.923 \pm 0.191 \mu\text{g.mL}^{-1}$ . On the other hand, the amylose percentage presented a lower result, when compared to the same author showing data of  $21.846 \pm 0.384\%$ , in the soluble starch sample. This percentage difference is due to the starch used in sample, because the sample analyzed in this study contained three equal proportions of starch from different peels, in which, it can be concluded that each vegetable peel presents different concentrations of starch and, consequently, of amylose.

In relation to analysis by Fourier transfer infrared spectroscopy (FTIR), main absorption bands of starch structure were observed OH groups reference to glycosidic unit, axial deformation of C-H bonds present on the ring and axial deformations of C-O-C system and of C-O bonds present in alcohols. New absorption at  $1740 \text{ cm}^{-1}$  characteristic of vibrational stretching of ester carbonyl ( $\text{C}=\text{O}$ ) was observed, which may be due to the process of recrystallization of starch and its esterification that occurs at temperatures above  $80^\circ\text{C}$  from its processing [29].

Samples were evaluated individually in order to observe of starch presence, size and shape in each sample from vegetable peels. The SEM analysis of samples showed starch granules grouped, due to gelatinization phenomenon and irregular sized as observed by Szymońska and collaborators [33]. In addition, starch from potato peel sample presented higher concentration according to data found in yield process and amylose concentration assays.

The pH values were performed in this paper and it was similar results found by Silva et al [32], in which was observed an average of 5.48 pH value for the native starch analyzed. Data determined that starch can be easily used in topical formulations. Viscosity analysis presented also desirable data for cosmetic formulation as thickener agent.

For microparticles development, magnetic agitation was used, and it was obtained microparticles with orange coloration, due to the beta-carotene presence in starch extracted from carrot peel. This characteristic can offer antioxidant activity for microcapsules, protecting ingredients possibly loaded. Microcapsules exhibited zeta potential value of  $-27.96 \pm 1.0 \text{ mV}$ , similar to describe by Gurrola and collaborators [34] when used ultrasonic technical for development starch particles.

Particles showed micrometric morphology, spherical shape, functional groups found in starch standard and PDI index of 0.2 as desirable. The average particle size was  $4494.37 \text{ nm}$ . Studies performed by Bai and collaborators [35] and Chan and collaborators [36] described that the use of ultrasound method to develop starch micro or nanoparticles can contributed for the production of particles with smaller sizes.

Starch is a type of option of materials commonly used such as biopolymer in cosmetic formulas such as thickening agents in topical formulations, influencing the rheological behavior, physical stability, and sensorial of formulas occasioned mainly due to the viscosity and spreadability increase [37].

Then, an emulsion was developed containing low concentration of fatty material, becoming low oily sensation and appropriate for topical application. Formulation presented lightly orange color due to beta- carotene presence from carrot peel. This data can offer natural coloration and antioxidant activity to emulsion. Density, consistency and pH values obtained were also considered desirable for one formula for topical use.

## **5. CONCLUSION**

Starch was obtained from vegetable peels waste, presented ability to production of micro and/or nanoparticles and to increase the viscosity in cosmetic formulations, being used as a thickener. In addition, beta-carotene presents in carrot peel offered antioxidant property to microcapsules and topical formulation. Therefore, the peel waste, besides presenting promising results, are also one important alternative to be used in cosmetic products, due to their low cost, contribution to waste elimination and reduced environmental impact.

## **6. ACKNOWLEDGMENTS**

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## **7. CONFLICT OF INTEREST STATEMENT**

None.

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