INFRARED SPECTRA OF THE TARO MUCILAGE OBTAINED THROUGH DIFFERENT EXTRACTION TECHNIQUES

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ABSTRACT: The taro (*Colocasia esculenta*) mucilage can be used as a natural additive in food. However, in literature, there is no reference to a standard extraction technique. Therefore, it is important to study the mucilage obtained through different techniques in order to evaluate which extraction method is capable of obtaining the mucilage with the best technological properties and the least amount of impurities. The objective of this work was to apply the FTIR (Fourier-transform infrared spectroscopy) in the characterization of taro mucilage obtained through three different extraction techniques. The taro rhizomes acquired led to mucilage extraction through the high temperature method (80 °C) (HT), high temperature extraction (80 °C) and precipitated with ethanol (HTE), and extraction at room temperature and precipitated with ethanol (RTE). The powder mucilage obtained was analyzed through infrared (FTIR). The main bandwidth obtained in every mucilage was hidroxy (-OH). Only HT has a bandwidth characteristic to the methyl group. Taking into consideration the spectra analyzed, the presence of carbohydrates and proteins in every mucilage.

KEYWORDS: *Colocasia esculenta, FTIR*, mucilage from the taro rhizome.

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1. INTRODUCTION

The mucilage capacity to improve the functional and rheological properties of food, as a natural additive, triggered the interest for the exploration of this hydrocolloid which can come from various vegetal sources. Some examples of vegetables which are sources of mucilage are yam (*Dioscorea* sp.), taro (*Colocasia esculenta*), Barbados gooseberry (*Pereskia aculeata*), and *X. americana* seeds (Contado et al., 2009; Tavares et al., 2011, Andrade et al., 2021, Andrade et al., 2020, Andrade et al., 2015, Nagata et al., 2014; Arafa & Badr; 2023; Lima Júnior et al., 2013; Bazezew et al., 2022). The taro rhizome mucilage, which is the focus of this work, can be applied in the food industry as an emulsifier, as a thickenes, and as a stabilizer, as well as in the partial replacement of hydrogenated fat in the baking industry (Contado et al., 2009; Tavares et al., 2011; Nagata, Andrade, & Pereira, 2014, Andrade et al., 2021; Arafa & Badr; 2023). It also holds an appeal as a natural product.

The taro mucilage is mainly made of carbohydrates (galactose, arabinose, and glucose) and proteins (Andrade et al., 2021; Andrade et al., 2020; Andrade et al., 2015). With the use of specific extraction techniques, there can also be the presence of starch, which is a polysaccharide that is considered an impurity and may decrease the emulsifying action of the mucilage (Andrade et al., 2020).

According to Andrade et al. (2020), the extraction technique may change the technological properties of the mucilage obtained, its chemical composition, and its physical structure. Thus, it is important to study the mucilage obtained through different techniques in order to find the technique that results in a product with better technological characteristics and, when possible, better performance and low energy and reagent spending for the extraction.

The FTIR spectroscopy can be used for the identification and characterization of organic, inorganic, and polymeric compounds. Basically, what is measured in this analysis is the energy fraction transmitted or absorbed in relation to the incident energy in a particular wavelength or wavenumber (Smith, 1979). The use of this analysis method brings a series of advantages. The reduction in analysis time, the substantial decrease of sample amounts, and the increase in capacity to identify or characterize complex structures stand out (LOPES; FASCIO, 2004).

In the mid-infrared region, which is the range used for the analysis in this work, there can be found a huge number of research and applications taking into consideration that it present a huge amount of information used for the functional characterization of organic compounds (Skoog et al. 2002).

The spectroscopy in the infrared region is extremely useful for the characterization of the extracellular polysaccharide coming from bacteria. Such chemical compound is also present in the mucilage. Therefore, FTIR becomes an interesting technique to be applied in the study of taro mucilage obtained through different extraction techniques (Osiro et al., 2000; Forato et al., 2010; Andrade et al., 2015, Tavares et al., 2011; Andrade et al., 2020) since it is able to detect differences in the chemical composition.

The objective of this work was to apply the FTIR technique, which is an important tool for the food analysis field, in the characterization of taro mucilage obtained through three different extraction techniques.

2. MATERIAL AND METHODS

2.1 Different extraction techniques of mucilage coming from taro rhizomes

The taro rhizomes (*Colocasia esculenta*) were acquired in the fruit and vegetable retail trade in the city of Lavras, Minas Gerais, Brazil. They were washed in running water, peeled, and, once again, washed in running water. Later, they were used for different mucilage extraction techniques.

2.1.1 Taro mucilage extracted at room temperature and precipitated with ethanol (RTE)

Portions of 300 g of taro rhizome were crushed in an industrial blender (Lucre, Catanduva, Brasil) for five minutes, and, in the end, all portions were reunited and homogenized.

The crushed taro mucilage was manually extracted through filtration in a polyester mesh (40 cm x 40 cm), in accordance with Contado et al. (2009). After the filtration process, the mucilage was precipitated with ethyl alcohol 99.5% in a proportion of three alcohol volumes for each volume of watery mixture. The precipitated was separated and dried in a greenhouse with air circulation at 40 °C until it reached constant mass. Then, in was kept at a desiccator with silica until the analysis was carried out (Andrade et al., 2020).

2.1.2 Taro mucilage extracted at high temperature (HT)

The methodologies of Sharma, Bharadwaj, and Gupta (2008), Deveswaran, Bharath, Furtado, and Basavaraj (2009), and El-Mahdy and El-Sebaiy (1984) were followed, with changes by Andrade et al. (2020). The peeled rhizomes were soaked in water for 30 minutes, Later, they were left for almost three hours at 80 °C in a double boiler. After cooling, the mucilage was extracted using a polyester mesh (40 cm x 40 cm).

The filtered mucilage was freeze-dried for almost 75 hours in a Liobras (L101) device. After freeze-drying, the material in the form of flocks was macerated in a mortar with a pestle, homogenized, and kept in a desiccator with silica until the analysis was performed.

2.1.3 Taro mucilage extracted at high temperature and precipitated with ethanol (HTE)

The aforementioned methodology (2.1.2) was followed. However, after the filtration in the polyester mesh, ethyl alcohol 99.5% was added in the proportion of three volumes for each volume of watery mixture for the precipitation of the mucilage.

The precipitation was separated and dried in a greenhouse with air circulation at 40 °C until it reached constant mass. Later, it was kept in a desiccator with silica until the analysis was performed.

2.2 Infrared spectrum with attenuated total reflectance (ATR-FTIR)

The ATR-FTIR spectra of the mucilage obtained were collected through as spectrometer (IRAffinity-1) equipped with an attenuated total reflectance (ATR)

device with a zinc selenide (ZnSe) crystal. The spectra were acquired with 64 scan and resolution of 4 cm^{-1} in the between 4.400 and 600 cm^{-1}.

3. RESULTS AND DISCUSSION

The works of Andrade et al. (2020) and Andrade et al. (2021) studies various extraction techniques of taro mucilage varying temperature, ethanol use as a precipitant of the mucilage, and drying techniques. According to the first work, the mucilage extracted at low temperature (4 °C) and precipitated with ethanol has excellent emulsion activity and stability. It can, therefore, be used in the food industry as a natural additive. The mucilage extracted at low temperature is considered to be pure due to the absence of starch, which is one of the major contaminants present in the mucilage when using some extraction techniques.

For Andrade et al. (2020), the mucilage extracted at high temperature (with or without the use of ethanol) and the one extracted at room temperature and precipitated with ethanol have considerable emulsion stability (91.29%, 66.98%, and 69.05%). Besides, the mucilage extracted at high temperature and precipitated with ethanol has high emulsifying activity (76.39%). Thus, it is important to study them in the search of further details. One of the options is to perform the infrared analysis which provided information regarding functional groups present in the products.

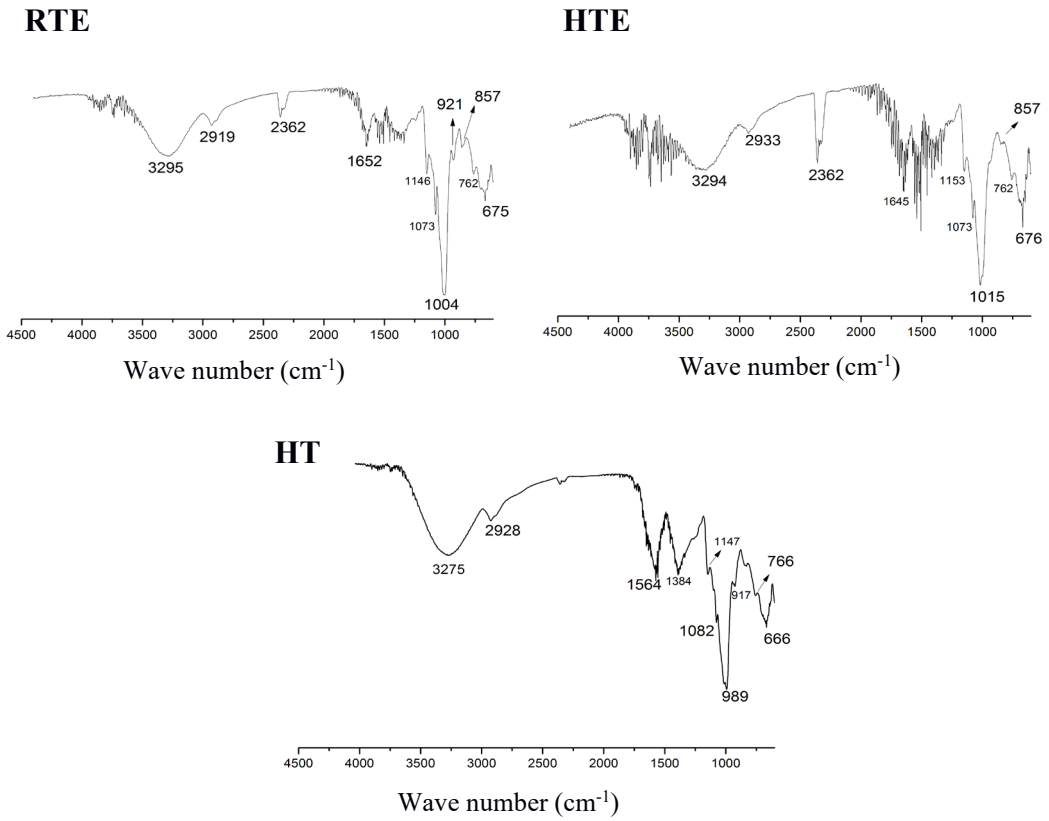

Figure 1 – Infrared spectra taro mucilage extracted through different techniques. RTE: mucilage extracted at room temperature and precipitated with ethanol; HT: mucilage extracted at high temperature; HTE: mucilage extracted at high temperature and precipitated with ethanol.

In Figure 1, the infrared spectra of the mucilage extracted at room temperature and precipitated with ethanol (RTE), of the mucilage extracted at high temperature (80 °C) (HT), and of the mucilage extracted at high temperature and precipitated with ethanol (HTE) can be found.

The bandwidths present in 3,295, 3,294, and 3,275 cm^{-1} correspond to the axial deformation of the hydroxyl groups (-OH) in intermolecular alcohol hydrogen bonds, commonly found in polysaccharides. This confirms the presence of carbohydrates in the mucilage obtained (Mothé & Correia, 2002; Andrade et al., 2020; Andrade et al., 2015).

The bandwidths in 2,919, 2,933, and 2.928 cm^{-1} correspond to the axial deformation of the C-H bond, which is found in the region between 3,000 and 2,840 cm^{-1} (Mothé & Correia, 2002). According to Ghadiri Alamdari et al. (2021), these bandwidths are attributed to the C-H stretching of polysaccharides. This also confirms the presence of carbohydrates.

The bandwidths between 1,233 e 1,000 cm^{-1} can be observed in every mucilage. This is similar to what was observed by Lin and Huang (1993). Thus it can be a result of the C-OH alcohol groups, in particular structures such as carbohydrates. Thus, the infrared spectra prove the structure of a polysaccharide with a C-O-C bond, which is a characteristic of carbohydrates, between 1.200 cm^{-1} e 900 cm^{-1}. This confirms the bond between the polymer-forming monomers as observed by Dalonso et al. (2009).

The region between 1,750 cm^{-1} and 1,500 cm^{-1}, attributed to the C=O stretching of the nucleic acid amide I and amide II of the proteins, is present in the mucilage obtained (Santos et al., 2012). It confirms the presence of proteins as in the work of Andrade et al. (2015), which studied the taro mucilage extracted at room temperature without precipitation. Andrade et al. (2021) detected the presence of protein in this mucilage through the Biuret test.

According to the infrared spectra, it can be inferred that there is the presence of carbohydrates and proteins in the mucilage studies. The standing out difference among the spectra is the presence of the bandwidth in 1,384 cm^{-1} present in HT. According to Andrade et al. (2015), the locust and guar gums and the taro mucilage extracted at room temperature have bandwidths that are in close proximity with 1.380 cm^{-1}, which can indicate the presence of a methyl (-CH_3) group (Silverstein, Webster, & Kiemle, 2006). The presence of this group can provide a hydrophobic portion of the mucilage. This may favor its emulsifying activity and stability.

Other analyzes such as mineral determination through atomic absorption, amino acids and monosaccharides through liquid chromatography (HPLC), thermal analysis, and X-ray diffractometry are interesting possibilities for future works since they may allow us to better understand the chemical composition and predict behaviors in the mucilage studies. In the case of mineral determinations, monosaccharides and amino acids are important because they can better explain the stability, emulsifying activity, and rheological behavior of the mucilage. The thermal analysis is interesting because it makes it possible to understand the hydrocolloid behavior in high temperatures once the mucilage can be added to bakery industry products. The x-ray diffractometry can infer if the mucilage is crystalline, semicrystalline, or amorphous, thus explaining its behavior in high temperatures.

4. CONCLUSION

With the spectra analyzed, it can be inferred the presence of carbohydrates and proteins in every mucilage. The infrared spectra of HT stands out due to the presence of a bandwidth in close proximity to 1,380 cm^{-1}, which can indicate the presence of the methyl group.

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