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Production and Characterization of Second Generation Ethanol from Potato Peel (*Solanum tuberosum*) using Raman Spectroscopy

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Abstract: Among the many barriers to commercialization of lignocellulosic ethanol production, e.g. potato peels, the lack of ways of monitoring and characterizing can be cited. Non invasive techniques, such as IR and Raman Spectroscopy, are ways of enabling such control. The aim of this study was to produce second generation ethanol from English potato peels (Solanum tuberosum) and identify its composition through Raman Spectroscopy. The hydrolysis was followed by neutralization, fermentation with Saccharomyces Cerevisiae and distillation. Subsequently, a comparison was made with anhydrous ethanol and Raman spectroscopy's sample. Both showed spectral features referred to alcohol, with peaks at 438 and 1051 cm^{-1} assigned to Anhydrous Ethanol and peaks at 440 and 1046 cm^{-1} assigned to Second Generation Ethanol from Potato Peel. However, the sample produced from Second Generation Ethanol exhibits a distinct peak attributed to the presence of water. It is concluded that the production of second generation ethanol from potato peel is feasible and Raman spectroscopy is a good way of characterizing.
Keywords: Second Generation Ethanol; Raman Spectroscopy; Fermentation; Molecule Vibration

Produção e Caracterização de Etanol de Segunda Geração a partir de Casca de Batata (Solanum tuberosum) utilizando Espectroscopia Raman

Resumo: Entre as muitas barreiras à comercialização da produção de etanol lignocelulósico, por ex. cascas de batata, pode-se citar a falta de formas de monitoramento e caracterização. Técnicas não invasivas, como Espectroscopia IR e Raman, são formas de possibilitar esse controle. O objetivo deste estudo foi produzir etanol de segunda geração a partir de cascas de batata inglesa (Solanum tuberosum) e identificar sua composição através de Espectroscopia Raman. A hidrólise foi seguida de neutralização, fermentação com Saccharomyces Cerevisiae e destilação. Posteriormente, foi feita uma comparação entre etanol anidro e amostra de espectroscopia Raman. Ambos apresentaram características espectrais referentes ao álcool, com picos em 438 e 1051 cm-1 atribuídos ao Etanol Anidro e picos em 440 e 1046 cm-1 atribuídos ao Etanol de Segunda Geração de Casca de Batata. Porém, a amostra produzida a partir do Etanol de Segunda Geração apresenta um pico distinto atribuído à presença de água. Conclui-se que a produção de etanol de segunda geração a partir da casca de batata é viável e a espectroscopia Raman é uma boa forma de caracterização.
Palavras-chave: Etanol de Segunda Geração; Espectroscopia Raman; Fermentação; Vibração Molecular

Introduction

In recent decades, concerns about the scarcity of fossil resources and the increasing

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emissions of greenhouse gases have intensified the pursuit of renewable energy sources. Production of biofuels, such as ethanol, from renewable biomass is a promising alternative to meet this demand [4]. English potato peels present themselves as a potential raw material source for second generation ethanol production due to their abundance, low cost, and ability to reduce agricultural waste.

The composition of English potato peels is rich in cellulose and hemicellulose, making them an ideal candidate for lignocellulosic ethanol production. This process involves breaking down the lignin, cellulose, and hemicellulose in the peels into fermentable sugars that can be converted into ethanol by fermenting microorganisms [1].

To optimize the ethanol production process, it's crucial to understand the chemical composition and structure of English potato peels. Raman spectroscopy is a valuable tool for this characterization because it allows for the identification and quantification of the supracited components. Additionally, it enables the real-time monitoring of changes in the biomass structure during the conversion process, which is crucial for optimizing ethanol production efficiency [2].

The use of potato peels in second generation ethanol production is a promising research field in the pursuit of renewable energy sources [3]. The integration of Raman spectroscopy in the characterization of these peels offers valuable insights for the improvement of the production process. However, challenges, such as process optimization and ethanol production monitoring, need to be addressed for this approach to become economically viable on a commercial scale.

The objective of this study was to produce second-generation ethanol from English potato peels (Solanum tuberosum) and identify its composition through Raman spectroscopy.

Materials and methods

As a pre-treatment, 30 potatoes were peeled to obtain 1 Kg of peel, which were refrigerated for 24 hours in a household refrigerator. The peels were dried in an oven at 65°C for 72 hours. After drying, they were ground in a processor, sieved, and stored in a becker.

For hydrolysis, a batch reactor with reflux was used. A 30.75g of potato peel powder were weighed and placed in a round bottom glass flask, to which 153.75g of sulfuric acid (H_2SO_4) were added. The flask was heated using a heating mantle system. Once it reached 90°C, the mixture was submitted into heating for 30 minutes and then filtered and neutralized

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with sodium hydroxide to achieve pH of 5.

The neutralized must was transferred to an Erlenmeyer flask, and 5g of yeast (*Saccharomyces Cerevisiae*) were added to the mixture. The system was closed with a stopper that had a silicone tube attached to allow for the release of air and gases (Fig. 01). The mixture was stirred to start the process and then left to rest for 72 hours.



Fig. 01 – Fermentation system

After the resting period, it underwent a fractional distillation. The condensate was observed through the Vigreaux column at a temperature of 79°C. During the process, the distillate becker was submerged in a flask cooled with water and pieces of ice to prevent evaporation. The process continued for about 80 minutes, until the temperature reached exactly 94°C.

In the end, a transparent distillate was obtained. It was transferred to a small plastic container and sealed to be tested into Raman spectroscopy.

During the spectroscopic analysis, the Raman spectra were acquired in triplicate by pipetting 400 µL of the sample into an aluminum sample holder. A dispersive Raman spectrometer (CORA 5200) was employed for this purpose, utilizing a 785 nm excitation laser with a power of 450 mW. The spectrometer's spectral resolution covered the range from 400 to 1800 cm^{-1}. Each spectrum was collected over a duration of 30 seconds, involving 3 seconds and 10 accumulations.

Results and discussion

Figure 2 presents the mean Raman spectra of the Anhydrous Ethanol and Second Generation Ethanol from Potato Peel, showing similar spectra between them. Both samples

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showed spectral features referred to alcohol, with peaks at 438 and 1051 cm^{-1} assigned to Anhydrous Ethanol and peaks at 440 and 1046 cm^{-1} assigned to Second Generation Ethanol from Potato Peel.

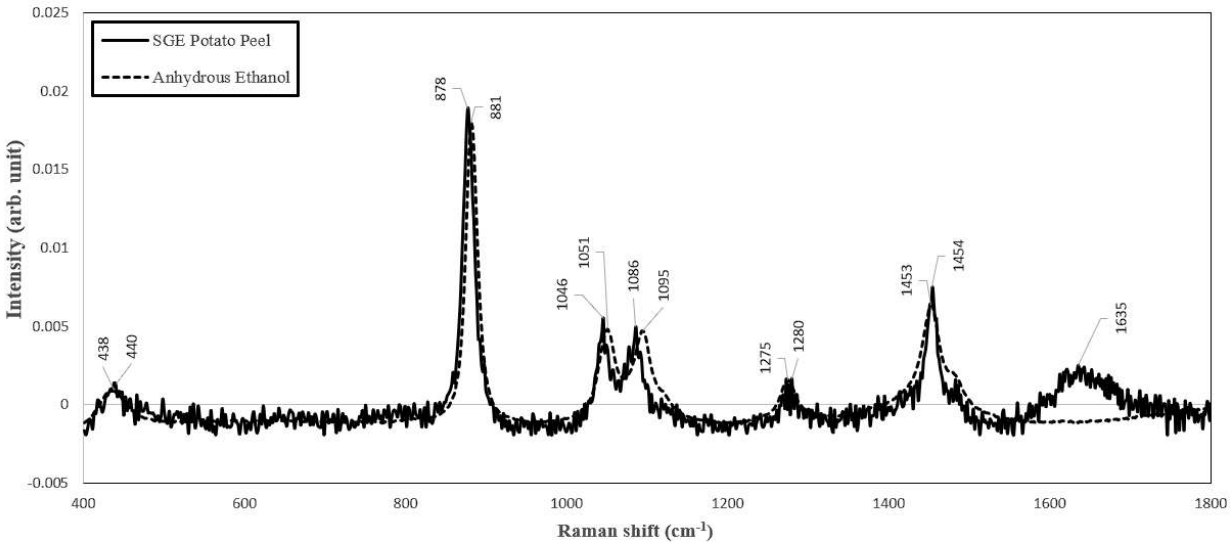

Fig.02. Raman Spectrum of Anhydrous Ethanol and SGE Potato Peel

The sample produced from Second Generation Ethanol exhibits a distinct peak in the region at 1635 cm^{-1}. This phenomenon is attributed to the presence of water in the sample, as complete purification of the produced ethanol is not achievable during distillation. The positions of sample's peaks and their respective molecular vibrations are detailed in table 01.

Table 01. Definition of assignment for peak position

Peak position (cm^{-1})	Assignment
440 - 438	Bending vibrations of C-C-O
881 - 878	Stretching vibrations C-C
1051 - 1046	Stretching vibrations C-O
1095 - 1086	Stretching vibrations C-C
1280 - 1275	Torsion and rotational vibrations CH2
1454 - 1453	Bending vibrations of CH3 and CH2

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1653 Bending vibrations of water

Conclusions

According to the obtained results, it demonstrates the successful production of second generation ethanol from potato peels. Additionally, characterization was carried out using Raman spectroscopy, with a particular emphasis on the alcohol functional group. This characterization was then compared with the spectral profiles of anhydrous ethanol at 440 and 1051 cm^{-1}.

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