^{13}C NMR Study of Albemoschus Esculentus Compounds

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Abstract: *We investigated the main compounds present in Albemoschus Esculentus (AE) by using ^{13}C solution and solid state nuclear magnetic resonance spectroscopy (NMR) techniques. NMR data allowed us to characterize the main type of components in this sample. Four main components were found: cellulose in the shell; a polysaccharide between the shell and seeds; and a triacylglycerol and a startch in the seed. Our results revealed that these components are responsible by AE physico-chemical properties.*

Resumo: *Os componentes principais presentes no Albemoschus Esculentus (AE) foram investigados através da espectroscopia de ressonância magnética nuclear (RMN). As técnicas em solução e no estado sólido foram utlizadas. Os dados de RMN possibilitaram a caracterização dos principais tipos de componentes presentes neste tipo de amostra. Quatro componentes principais foram identificados: a celulose, na casca; um polissacarídeo, entre a casca e a semente, um triacilglicerídeo e um amido na semente. Os resultados revelaram que estes componentes são responsáveis pelas propriedades físico-químicas do AE.*

Introduction

The investigation of the main compounds presentin *Albemoschus Esculentus* (AE) can provide a better understanding of its application in water treatment as a clarifying agent. Among the many experimental techniques that can be employed to study chemical structures, solution and solid state nuclear magnetic resonance have proved to be particularly effective. It is well known that solution techniques provide detailed information on chemical structure and microstructure. Among these is solid state NMR, which provides information on chemical structure without interfering in the sample, as this spectroscopic technique is non-destructive[1-5]. Therefore, data on molecular dynamics can be also obtained by solid state NMR. Indeed, both solution and solid state NMR can provide reliable information on the materials[1-9].

The main purpose of this work is to obtain information on the main chemical components of AE *for a* better understanding of its behavior when used in water treatment. To carry out a more comprehensive analysis, we thus characterized the fruit by both ^{13}C solution and solid state NMR techniques.

Types of treatments: in the first, the AE vegetable was dried and powdered with subsequent polysaccharide water extraction: The solutions and the residues after solvent

Experimental

The methodology of analysis is described as follows. The sample was submitted to two

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evaporation were analyzed by ^{13}C solution NMR by using deuterated acetone, deuterated dimethylsulfoxide, KOH/D_2O, and deuterium oxide, and ^{13}C solid state NMR. In the second procedure, AE was dried and its seed isolated. The shell and seed were powdered, and the powdered seed was extracted with acetone. After evaporation of the solvent, an oil was obtained.

All NMR solution spectra were carried out on a VARIAN MERCURY 300, and the solid state experiments were obtained on a VARIAN INOVA 300. Both spectrometers operated at a ^{13}C resonance frequency of 75.4 MHz. The ^{13}C solution spectra were obtained in adequate quantitative conditions. All solid state NMR spectra were recorded with magic angle spinning (MAS), with short delay between 90 degree pulses. Cross-polarization magic angle spinning (CPMAS) with a contact time of 1 ms, and cross-polarization magic angle spinning with dipolar dephasing (CPMASDD) spectra, were obtained at the same conditions. and CPMASDD was applied with a dephasing time of 40 μs.

Results and Discussion

According to the methodology previously described, the dried and powder soluble fractions of AE were analyzed by ^{13}C NMR solution in different solvents to detect the polymeric component, (probably a polysaccharide) which can have properties to be applied in water treatment. The solubilization was carried out in deuterated acetone $(CD_3)_2CO$, deuterated dimethylsulfoxide DMSO, KOH/D_2O, and deuterium oxide. All extracts were analyzed, and the interpretation of the spectra indicated that the extract obtained in $(CD_3)_2CO$ had signals related to the aliphatic region.These signals were attributed to an oil, probably, a triacylglycerol, derivingfrom the seed. The ^{13}C spectrum obtained from the extractin DMSO shows signals from a polysaccharide located at 99 ppm (C-O anomeric); 68-82 ppm (CH-O) and 62 ppm (CH_2-O) and also from cellulose located at 104 ppm (C-O anomeric); 84 ppm (CH-O) and 60 ppm (CH_2-O) (Figure 1). The ^{13}C spectrum of the extract obtained by KOH/D_2O showed the same signals detected in DMSO solution. The water solution did not show any C-13 NMR signal.

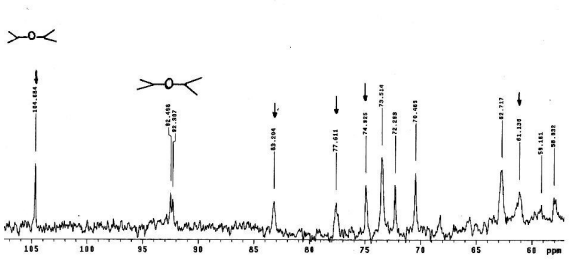

Figure 1. The ^{13}C spectrum obtained from the extract in DMSO.

^{13}C solid state NMR spectra were recorded using CPMAS and MAS techniques. Signals from polysaccharide, cellulose and oil were detected.After seed isolation, the oil extracted with acetone was characterized by ^{13}C solution NMR. All signals detected were attributed to a triacylglycerol, and such signals were the same found in the solid state NMR (Figure 2).

The CPMAS ^{13}C solid state NMR study of the seed flour showed that the main component would probably be a starch due to the detection of signals typical of mono, di and polysaccharides (Figure 3). MAS and CPMASDD techniques were also used, and the results obtained confirmed the data obtained by the analysis of the whole AE powder. These results are consistent with those found in the literature.

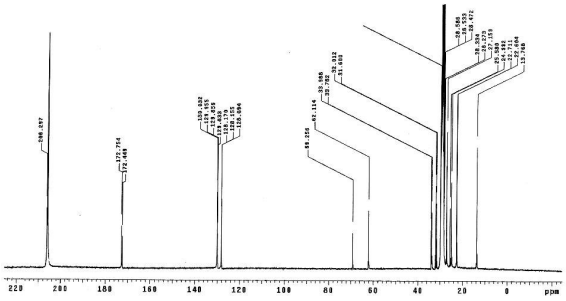

Figure 2. ^{13}C solution NMR of an oil extracted from AE, using $(CD_3)_2CO$ as a solvent

As shown in Figure 3, CPMAS ^{13}C main signals assigned in the spectrum were derived from the starch, and the weak lines located at about 34 and 145 ppm can be probably attributed to gluten proteins.

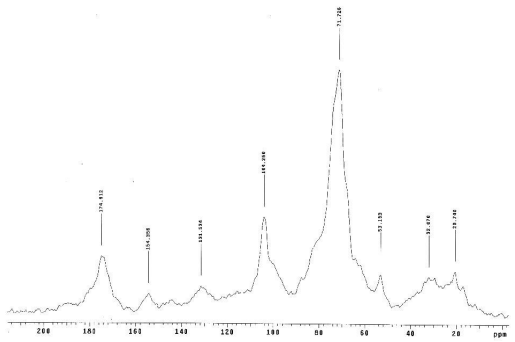

Figure 3 - CPMAS ^{13}C solid state NMR spectrum of AE seed starch.

Conclusions

Both solution and solid state NMR techniques provided valuable information on the main chemical components presentin the AE without any chemical treatment. The response of AE components, which was monitored by MAS, CPMAS, and CPMASDD showed that molecular mobility was directly influenced by the mixture of the components in the material, indicating sample heterogeneity.

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