



Raman spectroscopic investigation of tannin-furanic rigid foams



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ABSTRACT

Tannin-furanic rigid foams are innovative polymeric materials made of inexpensive organic ingredients, and are usually produced via an acid catalyzed polycondensation reaction between furfuryl alcohol and condensed flavonoids (e.g. Mimosa tannin). These bio-friendly foams have already been proposed e.g. as insulating material for eco-sustainable buildings (green building technology), as their most important physical properties are their low thermal conductivity and their high fire resistance.

The aim of the present study is the Raman spectroscopic characterization of the tannin-furanic rigid foams in order to establish a tool complementary to infrared spectroscopy for comparing their spectral signature with that of the precursor materials furfuryl alcohol, polymerized furfuryl alcohol, and Mimosa tannin. The discussion deals with similarities and differences to the spectral signatures of sp² carbon-based materials, the still preserved organic nature of the tannin-furanic foam, and with similarities and differences to recently reported infrared spectra.

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1. Introduction

One of the most innovative polymeric materials proposed in the last years are tannin-based rigid foams (see Fig. 1), [1–13] which can be produced from naturally occurring resources through different formulations of compounds. In the case of tannin-furanic rigid foams these are condensed tannin (e.g. Mimosa tannin or Quebracho tannin) [14,15], furfuryl alcohol [14–18], and an acid as polymerization catalyst.

The density of these blackish appearing foams generally governs their physical properties [1,13]. Compression resistance (a tannin-furanic foam of 200 kg/m³ density has a mechanical compression resistance of up to 1 MPa) and water absorption (a lighter foam of 50 kg/m³ can absorb water more than seven times the dry weight of the foam) are properties, among others, which have already been studied in depth [1,2,5,9,11–13].

Further basic characterization of tannin-furanic foams was carried out considering more application-oriented properties [2,8,11,13]. These foams have very low thermal conductivity (0.030–0.040 W m⁻¹ K⁻¹). They can easily resist strong acidic and basic conditions, hence showing remarkable chemical resistance. Moreover, tannin-furanic foams show high fire resistance, extreme fireproof behavior being obtainable using phosphoric acid as polymerization catalyst [2].

According to these interesting properties, several applications for these materials have been investigated or proposed [3,4,19], e.g. as insulation material for eco-sustainable buildings (green building technology) [5,11], or as adsorbent for wastewater treatment [12]. Present developments include the use of tannin as precursor for carbon polymerized High Internal Phase Emulsions (carbon polyHIPE or carboHIPEs) [20].

The most important precursor for the formation of the tannin-furanic foam polymer is the tannin itself [13–15,21–24], in the present case Mimosa tannin, which is a representative for condensed tannins. It is a complex oligomer mostly composed of flavan-3-ol repeating units, in this case predominantly prorobinetidin flavonoids, having a great amount of aromatic carbons (resorcinolic A-ring and pyrogalllic B-ring) and of hydroxyl groups [21].

Characterizations using MALDI-ToF [6,7,16] and solid state ¹³C NMR [11,25,26] were performed on tannin-furanic foams in order to find information about the copolymerization between flavonoid groups in tannin, furanic groups derived from furfuryl alcohol, and formaldehyde used as hardener, showing that the skeleton of these polymers is constituted by flavonoids and furans randomly arranged during the condensation reaction that produces them [1,7]. Additional information was deduced regarding the dimensional arrangement with a networked crosslinking and the presence of some methylene bridges.

Since Raman spectroscopy [27,28] revealed itself also advantageous in order to analyze flavonoids [29–33], tannins and wood extracts rich in flavonoids [34,35], and tannin-impregnated wood

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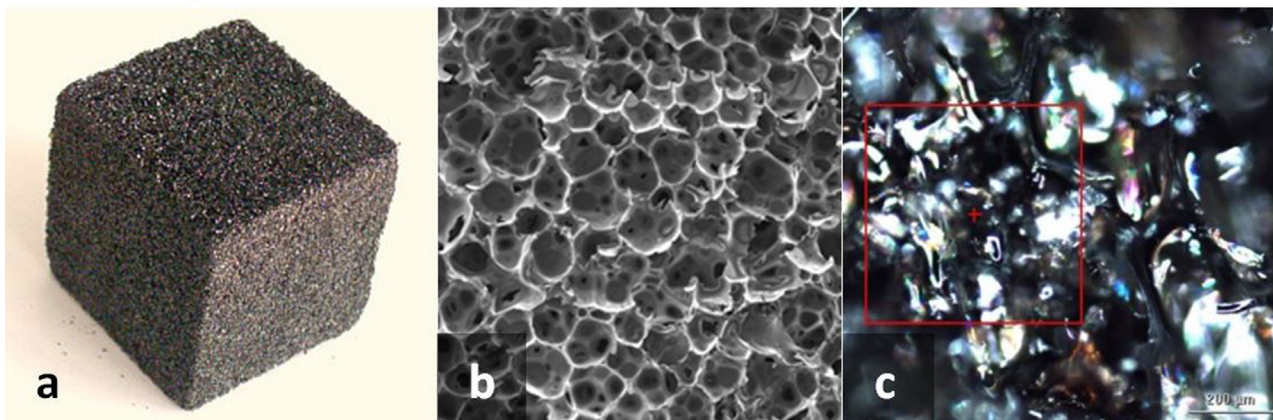


Fig. 1. a) Tannin-furanic rigid foam. b) SEM image of a tannin-furanic foam c) optical microscope image (10x magnification, dark field).

species [36], or in order to discriminate the content of different flavonoids and carotenoids in petals of pansy cultivars [37], and in order to distinguish between recent and fossil resins [38], or between lignin parts and leather parts in wood-leather panels [39], we decided to apply this spectroscopic technique to receive

additional information on the chemical rearrangement of flavonoids and furans after formation of the tannin-furanic rigid foam, and possibly establish a link to results obtained by infrared spectroscopy in similar materials [12,40,41], and by Raman spectroscopy in sp^2 carbon-based materials [42–45].

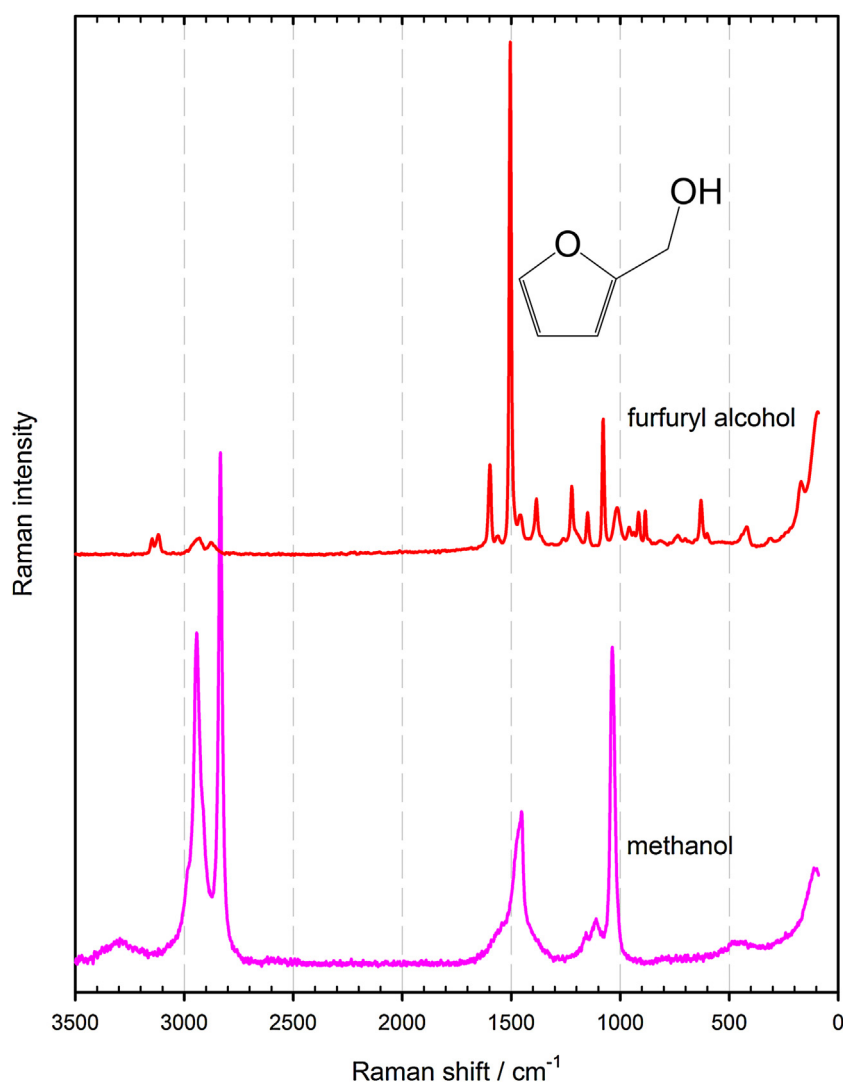


Fig. 2. Normalized Raman spectrum of furfuryl alcohol, compared to the normalized Raman spectrum of methanol, both obtained with laser excitation at 1064 nm. See Ref. [18,47,48] for an analogous comparison.

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