Thermostructural and fluorescence properties of enterolobium cyclocarpum extract embedded in a silica xerogel matrix

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Abstract

This work deals with the fluorescence behavior exhibited by extracts of parota wood (enterolobium cyclocarpum) when are embedded in a silica matrix, and the effect of the temperature on the emission and structural properties. The organics were incorporated into a silica matrix using the sol-gel method, and the resulting hybrid composite was subjected to different heat treatments to study its thermal stability and the effect on their structural and fluorescence properties. The structural properties were studied using x-ray diffraction (XRD), and the organics were analyzed by infrared spectroscopy (FT-IR) and fluorescence spectroscopy. Results show that the silica matrix avoids the thermal decomposition of the organics, and the structural changes of the silica impact on the fluorescence properties. In this sense, the maximum emission obtained at 400 °C was three times higher than the RT. This behavior was attributable to the strong interactions between the cellulosic compounds and the silica. Also, the cellulosic compounds promote the formation of open Si-O arrangements.

1. Introduction

Enterolobium cyclocarpum, also called parota or huanacaxtle, is a leguminous tree widely distributed in tropical regions of Mexico and the North of South America. The parota tree is one of the most important species due it reaches between 20 to 40 m in length, 3 m in diameter, and has a very extended crown spread [1–3]. The enormous amount of biomass obtained from these types of trees and their properties, such as hardness, low-weight, and resistance to decomposition, makes the parota suitable for a large variety of uses [4]. Parota trees also have applications in pharmaceutics and medicine. The leaf poultice is used to treat inflammatory tumors, the bark syrup in flu treatments, and the exudation of the tree bark in bronchitis remedies [5–7]. The food industry uses the parota seeds due to their starch properties, high nutritional value, and high protein content [8–10]. The parota gum also is used in food production because it confers some desirable characteristics such as density and texture [11–13].

Wood is the most abundant biopolymer on Earth [14], and the extracts obtained from there mainly contains cellulosic compounds. These extracts have an enormous potential for advanced applications in various engineering fields because exhibit properties allowing to manage the electromagnetic radiation [15–20]. Then, the ability to fabricated highly stable hybrid composites based on cellulosic compounds are useful in optoelectronic to develop optical fibers, organic light emitting diodes, and solar cells [20]. Besides wood extracts...
could serve as an agent to protect historic items based on paper [21, 22]. In this sense, the silica-based hybrid composites show high thermal stability [23–27], although the functional properties are closely dependent on the characteristics of the silica matrix [24–26]. In this work, a hybrid composite based on the wood extracts having a silica matrix was developed using the sol-gel method. The structural and fluorescence properties have been studied as well as their behavior with the temperature.

2. Experimental

2.1. Materials and methods

All chemicals used to fabricate the hybrid composite were analytical reagent grade, and they were used directly without further purification. Deionized water, tetraethyl orthosilicate Si(OC2H5)4 (Sigma-Aldrich, 98%), and ethyl alcohol (Sigma-Aldrich) were used as reagents. Also, some pieces of parota obtained from the central region of Mexico were employed to extract the wood components. Then, 30 g of dirt-free parota wood without any treatment was left into 20 ml of ethyl alcohol in a moderate stirring at room temperature. After two days, the solution became brown, indicating that the ethyl alcohol achieved the extraction of the wood components. Then, the sol-gel method was used to embed the parota components in the silica matrix. The molar ratios of water to the alkoxide were experimentally determined to ensure the hydrolysis reaction and obtain an amorphous silica structure [28]. In this case, the better molar ratios of ethanol to TEOS and water to TEOS were 4:1 and 11:66, respectively. After, 22.2 ml of TEOS and 17.0 ml of the ethyl alcohol with the parota extract were mixed at constant stirring for 15 min. Then, 20.2 ml of deionized water was slowly dropped over the TEOS/ethyl alcohol mixture at constant stirring for 30 min. Finally, the gel was left to dry at ambient conditions, and the dried silica incorporating the Parota extract was finely grounded and calcined each 100 °C up to 1000 °C for 1 h.

2.2. Characterization

The structural and optical properties were studied using x-ray diffraction (XRD), infrared spectroscopy (FT-IR), and fluorescence spectroscopy. X-ray data acquisition was made with an Inel Equinox 2000 diffractometer (cobalt source, \( \lambda = 1.7890 \) Å), from 10° to 60° in 2\( \theta \) using a step of 0.02°. The FT-IR spectra were carried out using a Perkin Elmer Frontier spectrometer with the fully attenuated reflectance technique ATR (PerkinElmer, Waltham, MA, USA). The fluorescence spectra were obtained with a Perkin Elmer LS55 spectrophotometer using an excitation wavelength \( \lambda_{ex} = 300 \) nm.
3. Results and discussion

3.1. Effect of the parota extract on the silica matrix

Figure 1(a) shows the XRD pattern of the hybrid composite revealed a broad band at 27.43°, indicating a short-range ordering of the silica matrix. This XRD pattern is related to an amorphous-like structure of the α-quartz, and it is obtained at room temperature and ambient pressure. Figure 1(b) shows the XRD pattern of the parota wood having a semicrystalline structure with an amorphous component. The XRD pattern of woods varies mainly with the amount of cellulose, hemicellulose, and lignin [29–35]. The cellulose chains give a semicrystalline XRD pattern, while the lignin monomers are responsible for the amorphous component observed in figure 1(b) [36]. Parota wood exhibits reflections at 18.75°, 25.82°, and 40.95°. However, those
Reflections are barely observable in the hybrid composite XRD pattern because of the low amount of cellulosic compounds extracted from the wood.

Figure 2 shows the FT-IR spectra of the hybrid composite, the parota extract, and the pure silica. The infrared spectrum of the parota extract (figure 2(a)) shows the presence of O–H and C–H of the ethyl alcohol because it is used to extract the cellulosic components from the parota. The wood extract is constituted of cellulose, hemicellulose, and lignin, as table 1 shows. Besides those components, there are identified syringyl, and guaiacyl which are lignin units present in hardwoods [37, 38]. On the other hand, at 3400 cm⁻¹ the presence of physic adsorptive water is related to an amorphous silica arrangement [39].

| Vibrational modes [14, 40–44] | Wood extract | Silica |
|-----------------------------|--------------|-------|
| CH₂ and CH₃ of the cellulose. | 2970         | Stretching vibrational absorption of silicon hydroxyl and adsorbed water. |
| C–H vibration of –OCH₃ groups of lignin. | 2880         | Si–O–Si asymmetric stretching vibrations. |
| C=O vibration of non-conjugated ketones and free aldehyde present in lignin and hemicellulose. | 1740         | Silanol groups (Si–OH). |
| C–H deformations, asymmetric bending vibration of –CH₃ and –CH₂ groups from lignin. | 1450         | Si–O–Si bending vibrations. |
| CH₂ aromatic skeletal vibrations related to the structure of cellulose. | 1420         | Si–O rocking vibrations. |
| δC–H from cellulose and hemicellulose. | 1380         | 1090                  |
| C–H of cellulose and Cl–O of syringyl derivatives (characteristic band of the hardwoods). | 1330         | 960                   |
| Acyl-oxygen CO–OR stretching vibrations of hemicelluloses and C–O stretch vibrations of the lignin guaiacyl rings. | 1265         | 1090                  |
| Syringyl rings of lignin | 1230         | 1265                  |
| C=O–C vibration in cellulose and hemicellulose. | 1230         | 1230                  |
| Aromatic C–H in-plane deformation and symmetrical C–O stretching. | 1087         | 1087                  |

Table 1. Vibrational modes of the wood extract and the silica.

Figure 4. Infrared spectra of the hybrid composite subjected to different heat-treatments from RT to 1000 °C. (Dashed lines are the FT-IR of the α-quartz and the parota extract measured as references).
The narrowing of the main silica band showed in figure 2(b) with embedded wood extracts is attributable to a structural rearrangement of Si-O rings in open structures with six members instead of the rings with four members usually formed by the silica \( [27, 47] \). Then, rings with many Si-O members improve the range in which these structures are ordered, which results in narrower bands than those formed with few-member rings. The cellulosic compounds extracted from the parota wood induce this structural rearrangement of the silica, although its behavior could depend on the specific amounts of lignin, cellulose, and hemicellulose having in the wood extract. In this sense, the structural changes that occurred in the silica matrix are evaluated through the behavior of its main band as well as other minor contributions. On the other hand, the thermal stability of the organics can be monitoring through bands in 1230, 1380, and 1740 cm\(^{-1}\), while the bands at 1040 and 1087 cm\(^{-1}\) are obscured by the silica.

### 3.2. Thermostructural behavior of the hybrid composite

Figure 3 shows the XRD pattern of the hybrid composites at room temperature (RT), and the ones heat-treated at 200 °C, 400 °C, 600 °C, 800 °C, and 1000 °C. The position and the full width at half maximum (FWHM) of the wood reflections also are indicated in the diffractograms. Sample at RT exhibits a broad band centered at 27.4° (dashed red line) associated with an amorphous-like structure of the \( \alpha \)-quartz. This band shifts to lower angles as the temperature increases indicating a structural rearrangement of the silica bonds. However, a broadening of the band is observed at 600 °C, which is associated with the coexistence of two isomorphous silica formations, one related to \( \alpha \)-quartz, and the other one based on \( \beta \)-tridymite \([26]\). At 1000 °C, the band suffer a narrowing and the center shifts to 25.0°, which reveals the disappearance \( \alpha \)-quartz, persisting only the \( \beta \)-tridymite with a crystallinity enhancement. Then, the cellulosic components extracted from the parota wood avoid the silica crystallization maintaining an amorphous arrangement. This behavior contrasts with others results reported using different organics. In that cases, the silica reaches crystalline structures from 800 °C \([25, 26]\). On the other hand, all the XRD patterns show the presence of the parota wood components, indicating that the cellulosic compounds have been thermally stabilized.

Figure 4 shows the heat treatments effects on the FT-IR spectra. The dashed lines show the FT-IR spectra of the \( \alpha \)-quartz and the parota extracts measured as references. The hybrid composites show bands related to hemicellulose at 1740, and 1380 cm\(^{-1}\), cellulose at1380 cm\(^{-1}\), and lignin at 1230 cm\(^{-1}\); and bands related with the silica matrix at 480, 790, 945, and 1090 cm\(^{-1}\). Figure 4 shows that the bands associated with the cellulosic compounds increases as increasing the temperature. The C=O vibrations of the lignin and hemicellulose that
occurred at 1740 cm$^{-1}$ have a gradual intensity increment. Similarly, the bands at 1380 and 1230 cm$^{-1}$ of the cellulose and hemicellulose also increase with the temperature. This behavior is associated with the structural permanence of the cellulosic compounds extracted from the parota wood and due to their thermal stabilization promoted by the interaction with the silica matrix.

Below 400 °C, the main silica band shows a width reduction indicating a type of structural regularity based on rings with a small number of members. The silanols band at 945 cm$^{-1}$ is incorporated into the main silica band as increasing the temperature, and the band at 1090 cm$^{-1}$ corresponding to the -n-fold rings has an intensity increment with a width reduction. In this case, there could be contributions of phonons belonging to rings with many members [27]. Therefore, the higher intensity of these bands is understood due to the formation of open structures wherein prevails members with many rings, promoted by the presence of the organics. On the other hand, the band at 545 cm$^{-1}$ is strongly related to the presence of siloxane species, which have rings with four members like those observed in the early stages of the hydrolysis and condensation processes of alkoxysiloxanes [28]. This band disappears from 600 °C due to the formation of more ordered structures. The behavior at increasing temperature allows determining that the interaction of the organic with the silica matrix promotes the formation of open silica structures with many members. Also, the FT-IR spectra show the a clear presence of the lignin, cellulose, and hemicellulose at 1000 °C, indicating that the silica protects the cellulosic compounds thermally.

### 3.3. Fluorescence emission

Figure 5(a) shows the emission spectra obtained for the hybrid composite of the samples between RT and 500 °C, and figure 5(b) displays the spectra range from 600 °C to 1000 °C. The spectra have three contributions, from 350 to 380 nm, from 405 to 470, and from 480 to 500 nm associated with the type of cellulosic compounds. According to the literature, lignins present fluorescence at high wavelengths [21, 45], while cellulose emission occurs at lower wavelengths [21]. The fluorescence emission increases with the temperature up to 400 °C, where the emission reaches its maximum intensity, three times higher than the one exhibited by the sample without heat-treatment. At 500 °C, a diminishing of the fluorescence emission is observed, probably promoted by the α-quartz transformation into β-tridymite (amorphous-like structures). The other emission spectra do not show significant variations from 600 °C to 1000 °C because the silica structure also does not present relevant changes. Thus, the changes observed in the fluorescence spectra are promoted by a Si-O bonds rearrangement when temperature increases and because the cellulosic compounds are firmly attached to the silica, as the spectroscopic analysis also showed.

Figures 5(c) and (d) show the intensities and the fluorescence spectra areas, respectively. These graphs show a slight tendency to increase the fluorescence in the samples heat-treated from RT to 300 °C. Then, the maximum emission is reached at 400 °C, and after it is reduced again to stabilizing without changes up to 1000 °C. The fluorescence emission of this last sample confirms the organics prevalence in the heat treated samples, in agreement with the results obtained from XRD and FT-IR, wherein the interaction between the d-orbital and the π-bonds ensure the formation of hybrid structures between the cellulosic compounds and the silica matrix. The properties of the hybrid composite having cellulosic compounds and the high thermal resilience exhibited by the embedded organics allow using these organics in optoelectronics and other advanced applications.

### 4. Conclusions

The hybrid composite based on cellulosic compounds embedded in a silica matrix showed high thermostability, maintaining their properties functional at a temperature of 1000 °C. In this case, there was a clear interaction between the matrix and the organics. Silica protected and stabilized the organics, and the functional properties, such as the fluorescence emission, are closely related to the silica structure. However, the fluorescence properties varied at increasing the temperature because of the structural arrangement of the Si-O bonds. Also, the cellulosic compounds change how the silica is ordered in chains with four or six SiO2 members, which in turn modifies the crystallization behavior of the silica. The properties of the hybrid composite having cellulosic compounds and the high thermal resilience exhibited by the embedded organics will allow using these organics in optoelectronics and other novel applications.

### Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.
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