



E-ISSN: 2664-8784
P-ISSN: 2664-8776
Impact Factor: RJIF 8.26
IJRE 2026; 8(1): 01-03
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www.engineeringpaper.net
Received: 05-10-2025
Accepted: 12-11-2025

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Chemical composition of hemp determined by IR spectroscopy

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DOI: <https://www.doi.org/10.33545/26648776.2026.v8.i1a.153>

Abstract

In this article, we determined the composition of hemp fibers by FTIR spectroscopy using a Perkin-Elmer spectrophotometer. The spectrum of hemp fibers is between 3336 and 662cm⁻¹ wavelengths. Hemp fibers contain the following functional groups: OH stretching, C-H symmetrical stretching, C=O stretching vibration, OH bending of absorbed water, C=C aromatic symmetrical stretching, C-C, C-OH, C-H ring and side group vibrations.

Keywords: Hemp fibres, FTIR, spectroscopy

Introduction

Fourier-transform infrared spectroscopy (FTIR) ^[1] is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid, or gas. An FTIR spectrometer simultaneously collects high-resolution spectral data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer, which measures intensity over a narrow range of wavelengths at a time.

The term Fourier-transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical process) is required to convert the raw data into the actual spectrum.

The goal of absorption spectroscopy techniques (FTIR, ultraviolet-visible ("UV-vis") spectroscopy, etc.) is to measure how much light a sample absorbs at each wavelength ^[2]. The most straightforward way to do this, the "dispersive spectroscopy" technique, is to shine a monochromatic light beam at a sample, measure how much of the light is absorbed, and repeat for each different wavelength (This is how some UV-vis spectrometers work, for example.).

Fourier-transform spectroscopy is a less intuitive way to obtain the same information. Rather than shining a monochromatic beam of light (a beam composed of only a single wavelength) at the sample, this technique shines a beam containing many frequencies of light at once and measures how much of that beam is absorbed by the sample. Next, the beam is modified to contain a different combination of frequencies, giving a second data point. This process is rapidly repeated many times over a short time span. Afterwards, a computer takes all this data and works backward to infer what the absorption is at each wavelength ^[2].

The beam described above is generated by starting with a broadband light source—one containing the full spectrum of wavelengths to be measured. The light shines into a Michelson interferometer—a certain configuration of mirrors, one of which is moved by a motor. As this mirror moves, each wavelength of light in the beam is periodically blocked, transmitted, blocked, transmitted, by the interferometer, due to wave interference. Different wavelengths are modulated at different rates, so that at each moment or mirror position the beam coming out of the interferometer has a different spectrum ^[2].

As mentioned, computer processing is required to turn the raw data (light absorption for each mirror position) into the desired result (light absorption for each wavelength) ^[2]. The processing required turns out to be a common algorithm called the Fourier transform. The Fourier transform converts one domain (in this case displacement of the mirror in cm) into its inverse domain (wavenumbers in cm⁻¹). The raw data is called an "interferogram".

The first low-cost spectrophotometer capable of recording an infrared spectrum was the Perkin-Elmer Infracord produced in 1957 ^[3]. This instrument covered the wavelength range from 2.5 μm to 15 μm (wavenumber range 4,000 cm⁻¹ to 660 cm⁻¹).

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The lower wavelength limit was chosen to encompass the highest known vibration frequency due to a fundamental molecular vibration. The upper limit was imposed by the fact that the dispersing element was a prism made from a single crystal of rock-salt (sodium chloride), which becomes opaque at wavelengths longer than about 15 μm ; this spectral region became known as the rock-salt region. Later instruments used potassium bromide prisms to extend the range to 25 μm (400 cm^{-1}) and caesium iodide 50 μm (200 cm^{-1}). The region beyond 50 μm (200 cm^{-1}) became known as the far-infrared region; at very long wavelengths it merges into the microwave region. Measurements in the far infrared needed the development of accurately ruled diffraction gratings to replace the prisms as dispersing elements, since salt crystals are opaque in this region. More sensitive detectors than the bolometer were required because of the low energy of the radiation. One such was the Golay detector. An additional issue is the need to exclude atmospheric water vapour because water vapour has an intense pure rotational spectrum in this region. Far-infrared spectrophotometers were cumbersome, slow and expensive. The advantages of the Michelson interferometer were well-known, but considerable technical difficulties had to be overcome before a commercial instrument could be built. Also an electronic computer was needed to perform the required Fourier transform, and this only became practicable with the advent of minicomputers, such as the PDP-8, which became available in 1965. Digilab pioneered the world's first commercial FTIR spectrometer (Model FTS-14) in 1969 [1]. Digilab FTIRs are now a part of Agilent technologies's molecular product line after Agilent acquired spectroscopy business from Varian [4]. [5].

Materials and Methods

Composition of hemp fibres was examined by using Fourier Transform Infrared Spectroscopy (FTIR) measurement which uses a Perking-Elmer spectrometer and the standard KBr pellet technique. A total of 16 scans were taken for the sample between 650 cm^{-1} and 4000 cm^{-1} , with a resolution of 2 cm^{-1} . Hemp fibres were ground and mixed with KBr

and then pressed into a pel-let for FTIR measurement.



Fig 1: Perking-Elmer spectrometer

Results and Discussions

Infrared spectrum of hemp fibres is displayed in Figure 2. The typical functional groups and the IR signal with the possible sources are listed in Table 1 for a reference. It could be observed from Table 1 that five components exist in the hemp fibres after retting pretreatment. Figure 2 shows a weak absorbance around 1729 cm^{-1} in the FTIR spectrum of hemp fibre, which might be attributed to the presence of the carboxylic ester (C=O) in pectin and waxes. Intensities of some bands in IR spectra have been found to be sensitive to variations of cellulose crystallinity and have been used to evaluate Crystallinity Index (CI) of cellulose. The ratios of peaks at 1423 cm^{-1} and 896 cm^{-1} , 1368 cm^{-1} and 2887 cm^{-1} and 1368 cm^{-1} and 662 cm^{-1} are normally used to measure CI e.g. [1-4]. In this study, the ratio of 1368 cm^{-1} and 2887 cm^{-1} is above 1 which seems to be unsuitable for evaluation, while the ratios of 1423 to 896 cm^{-1} and 1368 to 662 cm^{-1} are 55.7% and 49.3% respectively. The value calculated by using Segal empirical method is 56%, indicating that the ratio of 1423 to 896 cm^{-1} is more suitable for CI evaluation [5-12].

Table 1: Main infrared transition for hemp fibre.

Wavenumber (cm^{-1})	Vibration	Sources
3336	OH stretching	Cellulose, Hemicellulose
2887	C-H symmetrical stretching	Cellulose, Hemicellulose
1729	C=O stretching vibration	Pectin, Waxes
1623	OH bending of absorbed water	Water
1506	C=C aromatic symmetrical stretching	Lignin
1423	HCH and OCH in-plane bending vibration	Cellulose
1368, 1362	In-the-plane CH bending	Cellulose, Hemicellulose
1317	CH ₂ rocking vibration	Cellulose
1246	C=O and G ring stretching	Lignin
1202	C-O-C symmetric stretching	Cellulose, Hemicellulose
1155	C-O-C asymmetrical stretching	Cellulose, Hemicellulose
1048, 1019, 995	C-C, C-OH, C-H ring and side group vibrations	Cellulose, Hemicellulose
896	CO, CCO and CCH deformation and stretching	Cellulose
662	C-OH out-of-plane bending	Cellulose

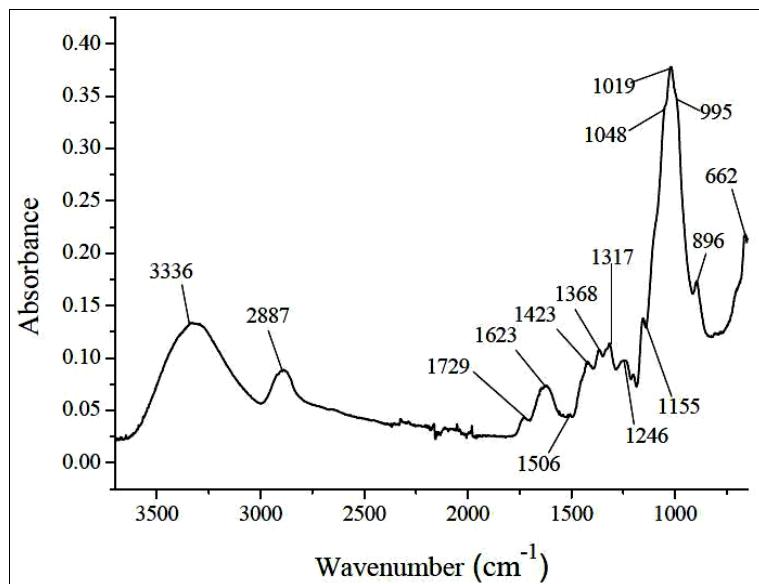


Fig 2: Spectrul IR al fibrelor de canepă

Conclusion

The composition of hemp fibers was determined using a Perking-Elmer spectrophotometer. The spectrum of hemp fibers is between wavelengths 3336 and 662cm⁻¹ and contains the following functional groups: OH stretching, C-H symmetrical stretching, C=O stretching vibration, OH bending of absorbed water, C=C aromatic symmetrical stretching, C-C, C-OH, C-H ring and side group vibrations [13-17].

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