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Qualitative and Quantitative Characterization of Textile Material by Fourier Transform Infra-Red

A Brief Review

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Abstract: Difference in the chemical properties of the textile materials is mainly attributed to the presence of different functional groups in their structure. Manufacturing process subjects the material to various treatments like mechanical stretching, bending, twisting, heating etc. They are liable to cause structural changes and therefore characteristics. So, knowledge of scientifically measured characterization of textile material before and after subjecting to various mechanisms of production pattern is needed. Fourier Transform Infra Red spectroscopy (FT-IR) has found preference over other techniques in this regards. It is successfully used in qualitative as well as quantitative analysis of textile substance.

Keywords: Infrared Spectroscopy, FT-IR, Textile, Polymer, Chemical structure.

I. INTRODUCTION

Infrared spectroscopy is a unique technique for materials analysis in the laboratory for over seventy years. [1] Most commonly used instruments in infrared spectroscopy are dispersive infrared spectrometer and Fourier transform infrared spectrometer. Fourier transform infrared (FTIR) spectrometer is preferred over dispersive spectrometer due to several reasons [1, 2, 3, 4, 5, 6 and 7]

- It is a non-destructive technique
- It provides a precise measurement method which requires no external calibration
- It can increase speed, collecting a scan every second
- It can increase sensitivity – one second scans can be co-added together to ratio out random noise
- It has greater optical throughput
- It is mechanically simple with only one moving part etc.

An infrared spectrum represents a fingerprint of a sample with absorption peaks which correspond to the frequencies of vibrations between the bonds of the atoms making up the material. Hence, different material has a unique combination of atoms; no two compounds produce the exact same infrared spectrum. Therefore, infrared spectroscopy can result in a positive identification of every different kind of material. This phenomenon can be used to qualitatively identify substances. Interaction of infrared light with matter, also determine the size of the peaks in the spectrum and gives direct indication of the amount of material present (quantitative analysis).

II. FTIR [FOURIER TRANSFORM INFRA-RED (FT-IR)]

FTIR spectrometers are the third generation infrared spectrometer [2]. They have been widely used in structure elucidation, which are either of natural origin or synthesized chemically. It is a measurement technique which collects spectra based on the measurements of the coherence of a radiative source, using time-domain or space-domain measurements of the electromagnetic radiation or other type of radiation.[3] It collects and digitizes the interferogram of a sample signal using an interferometer, performs the FT function, and displays the spectrum. [4, 5] It can be applied to a variety of types of spectroscopy including optical spectroscopy, infrared spectroscopy (FTIR, FT-NIRS), nuclear magnetic resonance (NMR) and magnetic resonance spectroscopic imaging (MRSI),mass spectrometry and electron spin resonance spectroscopy.[3,8]

III. BRIEF HISTORY OF DEVELOPMENT OF FTIR

Fourier [9] had developed mathematical transform (FT) method in 1700. Herschel [10] had developed mathematical IR radiation technique in 1800. Albert Michelson [9, 10, 11 and 12] had perfected FT-IR instrument and used it for several measurements in his study of light and relativity in 1887 .Michelson [10] experiments had led to IR published design of spectra of organic and his interferometer in 1891. Felgett analytical chemistry [9, 12, and 13] had discovered multiplexing advantage of FT in 1949. Jacquinot [9, 12, and 14] dispersive IR had discovered throughput spectrophotometer advantage of FT in 1954. Spectra-Tech and Digilab had developed the first commercial FT-IR micro spectrophotometer in 1983. FT-IR was combined with personal computers to make widely used, versatile, and cost-effective method of analysis in 1980s. Micro spectrophotometers had provided high-quality microanalysis [9].

IV. INSTRUMENTATION AND WORKING OF FTIR

FT-IR consists of a moving mirror, fixed mirror, beam splitter, IR radiation source and Detector. (Figure 1)[2, 3, 4] Michelson interferometer is used for analysis of IR radiation after passing through sample. [4] Radiation from IR source is collimated by mirror and the resultant beam is divided at beam splitter. Half of beam passes through mirror (fixed) and half refracted to moving mirror. Moving mirror is provided with mechanism, which allows this mirror to move a very short distance (typically a few millimeters) away from the beam splitter. After reflection by these two mirrors, two beams recombined at beam splitter. Because the path that one beam travels, is a fixed length and the other is constantly changing as its mirror moves. The signal which exits the interferometer is the result of these two beams "interfering" with each other, called an interferogram. The signal passes through cell and after that radiation is focused to detector. All the signals are amplified and converted to digital signals by the amplifier and analog-to-digital converter, respectively. The detector simultaneously measures all of frequencies that pass through the cell and routes the information to computer. This information is decoded by Fourier transformation and decoded spectrum is directed to read out device. Time from insertion of sample to recording of plot is about 2 min. [1, 3, 4, and 5]

V. REGION OF IR

The IR region of the electromagnetic spectrum ranges in wavelength from 2 -15 µm. Conventionally the IR region is subdivided into three regions, near IR, mid IR and far IR. Most of the IR used originates from the mid IR region. The table below indicates the IR spectral regions [2, 3, and 5]

Table 1: IR Spectral Regions

Region	Wavelength	Wave numbers (V), cm^{-1}	Frequencies (v), HZ
Near	0.78 -2.5	12800 – 4000	3.8×10^{14} - 1.2×10^{14}
Middle	2.5 - 50	4000 – 200	3.8×10^{14} - 1.2×10^{14}
Far	50 -100	200 -10	3.8×10^{14} - 1.2×10^{14}

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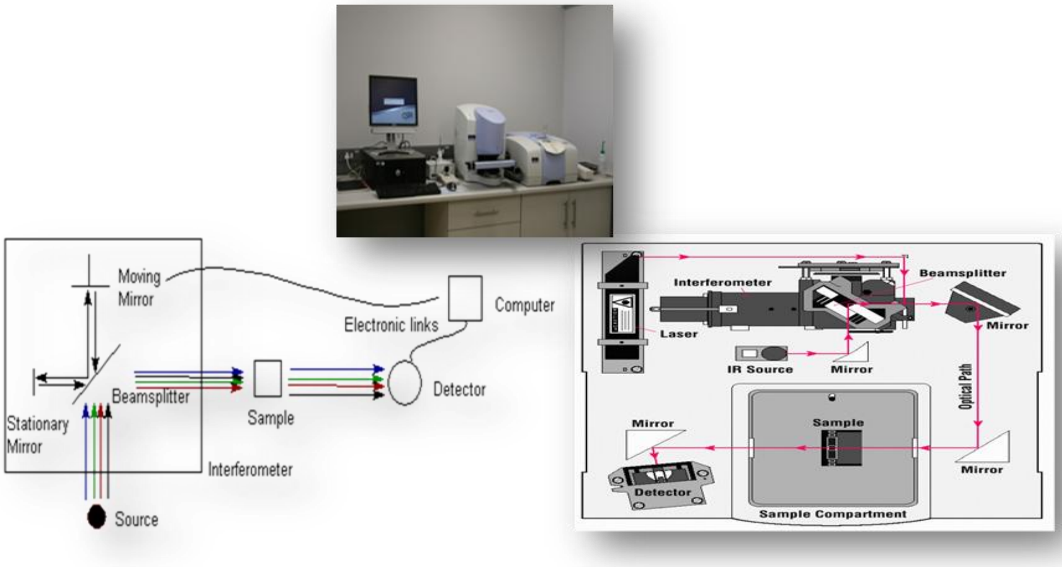

Figure 1: Working Model of FTIR; Courtesy to Reference [15]

VI. APPLICATION OF FT-IR IN TEXTILES

The speed and sensitivity of the FTIR spectroscopy allows rapid analysis of micro-samples down to the nanogram level in some cases, making the FTIR unmatched as a problem-solving tool in organic analysis. The FTIR microscope accessory allows spectra from a few nanogram of material to be obtained quickly, with little sample preparation, resulting in more data at lower cost. In some cases, thin films of residue are identified with a sensitivity that rivals or even exceeds electron or ion beam-based surface analysis techniques. This has enabled a wider use of this in textile field. [16]

A. Qualitative analysis

For qualitative identification purposes, the spectrum is commonly presented as transmittance versus wave number. Functional groups have their characteristic fundamental vibrations which give rise to absorption at certain frequency range in the spectrum (Figures 2(a-b)). However, several functional groups may absorb at the same frequency range, and a functional group may have multiple-characteristic absorption peaks, especially for 1500 – 650 cm-^{1}, which is called the fingerprint region [Figure 2(b)]. The fingerprint region is often the most complex and confusing region to interpret, and is usually the last section of a spectrum to be interpreted. However, the utility of the fingerprint region is that the many bands there provide a fingerprint for a molecule.

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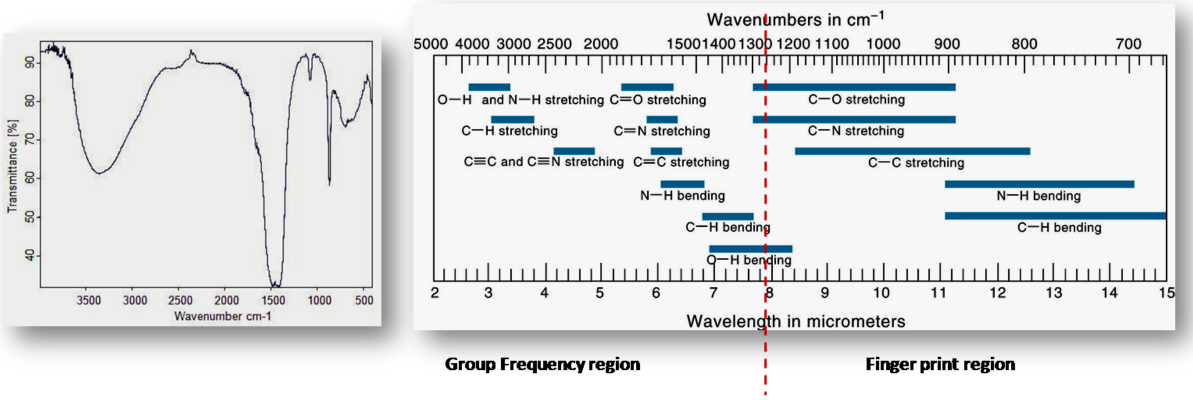

(b) Functional Group vibration and its relationship with regions of infrared absorption.

(a) Sample IR spectrum

Figure 2: IR Spectrum and Its Correlation In Characterization Of Functional Group In Substance.

Each band in a spectrum can be attributed to stretching or bending mode of a bond. Almost all the fundamental vibrations appear in the mid-infrared region. Hence, characterization of functional groups in substances according to the frequencies and intensities of absorption peaks is feasible, and also structures of molecules can be proposed.

A polymer is a long-chain molecule formed when individual units, called monomers, are linked together. Fibers are polymers that can be categorized into two broad groups, natural and synthetic. Due to the range of chemical compositions, different fibers exhibit a range of chemical and physical behaviors. Two of the most significant types of molecular motion are stretching and bending of bonds within the molecule. To cause a particular bend or stretch, a specific wave number of infrared light is required[17,18]. Other combinations of bonded atoms will absorb photons of other infrared frequencies. The collection of all absorbance between 600 and 4000 cm^{-1} constitutes the infrared spectrum for that molecule. This can be thought of as a molecular fingerprint. Just as people have unique fingerprints, molecules also have unique fingerprints. Identical molecules have identical fingerprints. Since every molecule has a unique arrangement of bonded atoms, every molecule absorbs a different set of infrared frequencies. [17]. FT-IR spectra for O-H group based cotton fiber and ester group based polyester has been given in figures 3(a-b) as an example. The bands represent cellulose [figure 3(a)] can be seen at 2906 cm^{-1} (CH, CH_2 stretch), 1478 cm^{-1} (H-C-H and H-O-C bend), 1379 cm^{-1}, 1334 cm^{-1} (H-C-C, H-C-O, and H-O-C bend), 1108 cm^{-1} (C-C and C-O stretch), 910 cm^{-1} (C-O-C in plane, symmetric), and 516-379 cm^{-1} (skeletal C-O-C, C-C-C, O-C-C and O-C-O bend). Types of bonds present in the chemical structure of polyester, has made execution of absorbency peaks and intensity totally different than cotton. This is purely influenced by the presence of ester group in its structure. Thus knowledge of chemical structure, natural as well as manmade fibers can be easily distinguished with FT-IR. Besides structural elucidation, another qualitative application of infrared spectroscopy is the identification of a fiber or finish (chemical) with a reference infrared spectrum. If all the peaks of the unknown match those of the reference, the fiber or finish can be identified. [2]

The sensitivity benefits also enable identification of even the smallest of contaminants. This makes FT-IR an invaluable tool for quality control or quality assurance applications whether it is batch-to-batch comparisons to quality standards or analysis of an unknown contaminant in liquid as well as solid form of textile material. Based on this FT-IR spectroscopy has been instrumental in identifying cotton trash components and foreign matter present along with cotton fiber in mix [19].

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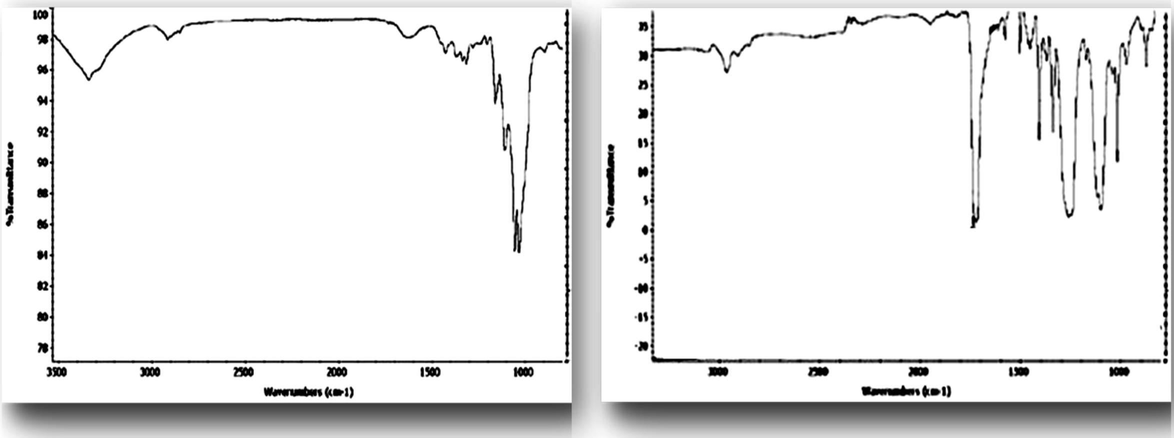

(a) Spectra of a single cotton fiber

(b) Spectra of a single polyester fiber

Figure 3: Identification of Chemical Structure of Fiber on the basis of FT-IR.

FT-IR can be used in chemical analysis by matching the spectra with a known database and monitoring the chemical reactions. Thus it makes possible the analysis of changes in molecular structure and intermolecular interactions [8]. This can provide authentic proof for polymer degradation, positive or negative effect of various finishes, even at Nano scale, on the performance of fiber, yarn or fabric etc.

Difference in the stretch band size can also help in defining the presence of crystalline and amorphous region in the chemical structure of the textile fibres [5, 10].

B. Quantitative analysis

Absorbance (A) is used for quantitative analysis due to its linear dependence on concentration. It is given by Beer-Lambert law [2, 23, 24, and 25] absorbance is directly proportional to the concentration and path length of sample:

$A=\epsilon cl$……… [11]

Where, *A is* absorbance, ε the molar extinction coefficient or molar absorptivity, which is characteristic for a specific substance, *c* the concentration and *l* the path length (or the thickness) of sample. Thus the intensity of the peaks in the FT-IR spectrum is proportional to the amount of substance present, for identical ε and *c*.

This concept is utilized in determining amount of components in a mix [11]. It also facilitates in defining the consistency of fiber blend or mix. The same way it determines Percentage of trash particles or foreign matter present in fiber, yarn or fabric. Similarly it is a reliable tool in identifying the fabric constituents and their density involved in the structure. It also verifies details of structural components, viz; blend components and their ratio used in the formation of constituent yarn [8, 20].

It can be used in measuring the degree of polymerization in polymer structure. [5]. It can be used to identify extent of polymer degradation [5, 10]. Changes in the character or quantity of a particular bond are assessed by measuring at a specific frequency over a time for the purpose.

Examination of textile material with added or subtracted value components can be done. [8] It is used in measuring capacity of optical conductivity [10]. It helps in defining degree of crystallinity attained by polymer during processing [5].

VII. LIMITATIONS OF FTIR

FTIR spectroscopy cannot be used to detect all the vibration modes in a molecule. It can be used only to study the non-symmetrical Vibrational state in an atom [16]. It is not possible to know molecular weight of substance

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with FT-IR spectroscopy. It is frequently non-adherence to Beer's law of complexity spectra. [21, 22] The narrowness of spectra and effect of stray radiations make the measurements of absorbance depended upon slit width and wavelength setting only, reduces accuracy. Generally, IR spectroscopy does not provide information of the relative positions of different functional groups on a molecule. From single IR spectrum of an unknown substance, it is not possible to know whether it is pure compound or a mixture of compound. [21, 22]

FTIR instruments do not measure spectra; they measure interferogram. Interferogram are difficult to interpret without first performing a Fourier transform to produce a spectrum. Accuracy of FT-IR remains true if there is no change in atmospheric conditions throughout the experiment. So, for highly sensitive work, and experiments which take long time, changes in infrared absorbing gas concentrations can severely affect the results. [22]

VIII. CONC LUSION

FT-IR spectrometers are hyphenated to chromatography, the mechanism of chemical reactions and the detection of unstable substances can be investigated accurately. One of the greatest advantages of the infrared spectroscopy is that virtually any sample in any state may be analysed. For example, liquids, solutions, pastes, powders, films, fibres, gases and surfaces can all be examined with a judicious choice of sampling technique. Thereby FT-IR has found unique place in various qualitative as well as quantitative analysis of wide spectrum textile field.

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