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Fourier Transform Infrared Spectroscopy for Natural Fibres

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1. Introduction

Infrared spectroscopy is nowadays one of the most important analytical techniques available to scientists. One of the greatest advantages of the infrared spectroscopy is that virtually any sample in any state may be analyzed. For example, liquids, solutions, pastes, powders, films, fibres, gases and surfaces can all be examined with a judicious choice of sampling technique. The review by Annette, Sudhakar, Ursula and Andrea [1-2] also demonstrates the applicability of dispersion infrared spectroscopy for natural fibres studies.

Fourier transform infrared spectroscopy (FTIR) has facilitated many different IR sampling techniques, including attenuated total reflection and diffuses reflectance infrared Fourier transform (DRIFT) spectroscopy. It has dramatically improved the quality of infrared spectra and minimized the time required to obtain data. The increased speed and higher ratio of signal-to-noise of FTIR relative to dispersion infrared has lead to a substantially greater number of applications of infrared in natural fibres research. In addition, the constant advancing of computer and computing science has made infrared spectroscopy techniques striding further: The availability of a dedicated computer, which is required for the FTIR instrumentation, has allowed the digitized spectra to be treated by sophisticated data processing techniques and increased the utility of the infrared spectra for qualitative and quantitative purposes. With interferometric techniques, the infrared spectroscopy is being launched into a new era and interest in this technique is at an all time high.

Cellulose, which acts as the reinforcing material in the cell wall, is the main constitute in natural fibres. The cellulose molecules are laid down in microfibrils in which there is extensive hydrogen bonding between cellulose chains, producing a strong crystalline structure. Much work has been published on the characterization of the hydrogen bonds in cellulose by using various techniques, among which FTIR has proved to be one of the most useful methods [3-6]. Furthermore, FTIR can provide researchers with further information on the super-molecular structure. FTIR can also be used to determine the chemical compositions of native natural fibres and the modified natural fibres.

This chapter of the book describes the application of FTIR in the hydrogen bonds analysis, determination of structures and chemical compositions, and the morphology characterization for natural fibres.

2. Hydrogen bonds analysis of natural celluloses by using FTIR

A hydrogen bond is the attractive interaction of a hydrogen atom with an electronegative atom, such as nitrogen, oxygen or fluorine, that comes from another molecule or chemical group. Cellulose occurs in the form of long, slender chains, polymer of 1-4 linked β-D-glucose (Figure 1). Hydroxyl groups in C2, C3 and C6 contribute to the formation of various kinds of inter- and intra-molecular hydrogen bonds. The formation of inter- and intra- molecular hydrogen bonds in the cellulose not only has a strong influence on the physical properties of cellulose, including solubility [7-8], hydroxyl reactivity [9-10] and crystallinity [11-12], but also plays an important role in the mechanical properties of the cellulose [13]. Calculated by Tashiro and Kobayashi [14] showed that hydrogen bonds contribute about 20% the strain energy to the cellulose. It is apparent that the investigation of hydrogen bonds on cellulosic fibres and other materials gives rise to great benefits for the research on all other aspects of natural fibres and related materials.

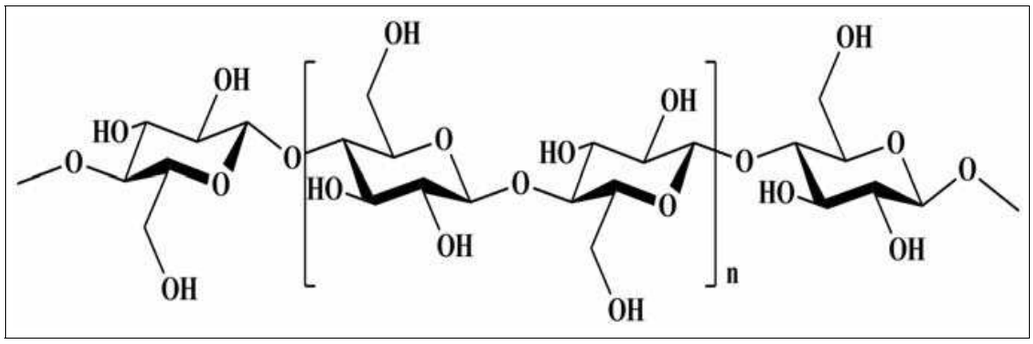


Fig. 1. Chemical structure of cellulose

X-ray diffraction has been a powerful tool [15-19] to investigate hydrogen bonds visualization, lengths and angles. FTIR is even a more advanced tool to study hydrogen bonds in cellulose. IR was firstly used to investigate hydrogen bonds in cellulose in the 1950s [e.g. 20-22] and then the whole area of OH stretching wave-number in IR spectra for cellulose I and cellulose II [23-24]. The OH stretching region always covers 3-4 sub-peaks and these sub-peaks cannot be determined in the original data set. Some mathematical methods (e.g. deconvolution [25-27] and second-derivative [28-30]) were used to identify the exact peak for hydrogen bonds. Hinterstoisser and Salmén [3, 31] recently used DMA-FTIR to investigate OH stretching vibration regions between 3700 and 3000 cm^{-1} in the cellulose. In their experiments, cellulose sheets were stretched sinusoidally at low strains while being irradiated with polarized infrared light. For the obtained dynamic IR signals (the in-phase and the out-of-phase responses of the sample), the dynamic IR cross-correlation can be defined. The responses of the OH-groups to an external perturbation can be recorded as in-phase and out-of phase spectra. The cross correlation of these spectra gave the 2D synchronous (Figure 2) and asynchronous (Figure 3) plots, clearly showing the separated bands in the OH-vibration range and the relation of the OH-groups among them. It is apparent that most of the researchers have focused on the establishment of cellulose structure by investigating hydrogen bonds with FTIR. These (the structure of nature fibres) will be discussed in the next section. Few reports have described the correlation of hydrogen bonds with other characteristics of cellulose by using FTIR technologies.

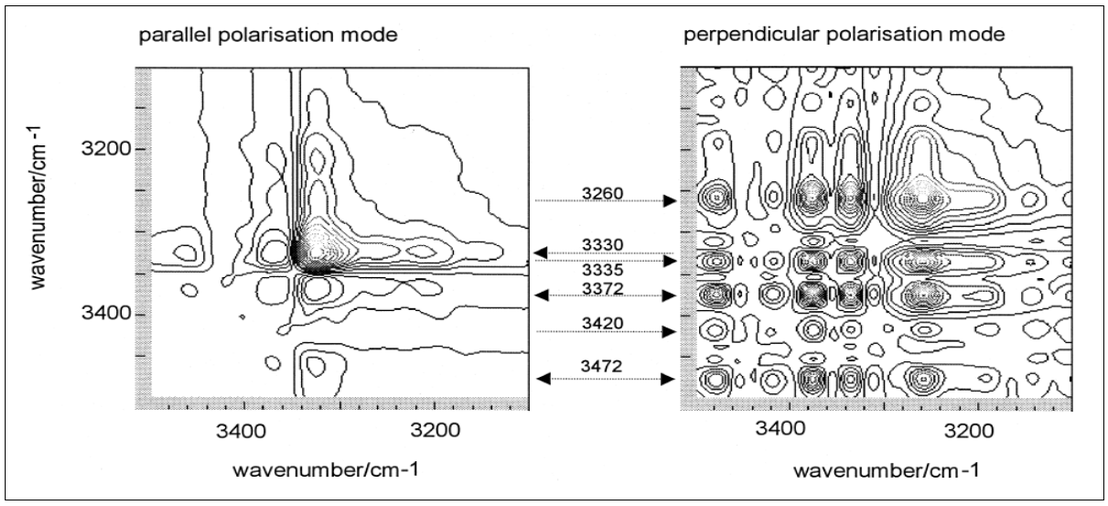


Fig. 2. Synchronous 2D plot-cross-correlation of in- and out-of-phase spectra [3]

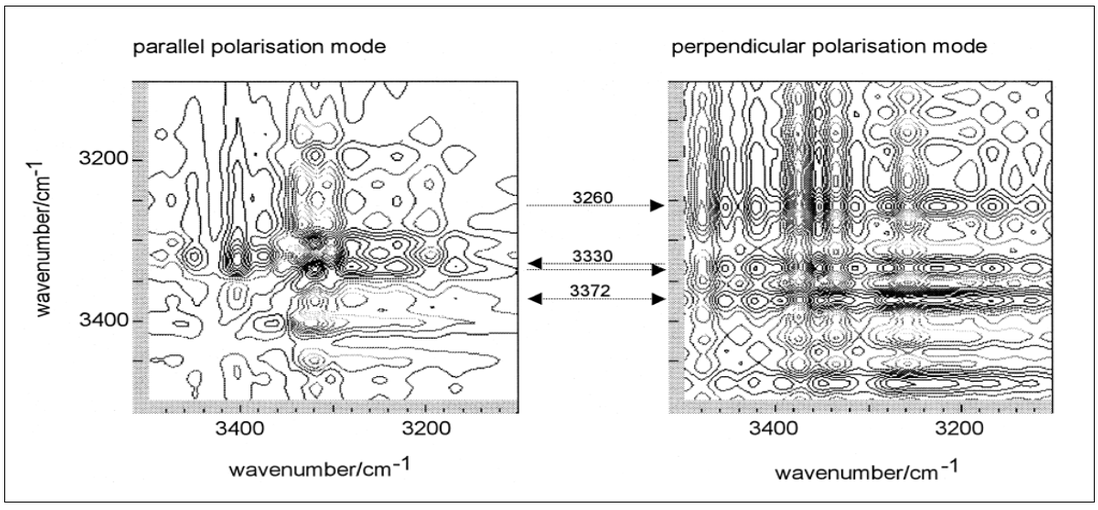


Fig. 3. Asynchronous 2D plot-cross-correlation of in- and out-of-phase spectra [3]

FTIR is very useful for examining the variation of hydrogen-bonds due to various defects [32]. The Nano-cellulose and Bio-composite Research Centre at Brunel University has investigated dislocations in natural fibres (hemp fibres) by using hydrogen-bonding characteristics under FTIR procedure. The test pieces were made from dislocation cluster (region) with the size of a single dislocation from a few microns to 100μm. The test pieces were then processed and examined by using FTIR measurement by using a Perkin-Elmer spectrometer and the standard KBr pellet technique. The recorded FTIR spectra (e.g. 3660–3000cm^{-1}) were deconvolved using Peak Fit V.4.12 software (Figure 4) and the peak positions of the major IR bands can be summarized and compared (Table 1). It can be found that the absorbance of hemp fibres without dislocations in the X–H (O–H and C–H) stretching region is much higher than that with dislocations. The peak positions of the four bands for hemp fibres with and without dislocations are 3450cm^{-1}, 3346cm^{-1}, 3262cm^{-1} and 3161cm^{-1} for the hemp without dislocations, and 3451cm^{-1}, 3350cm^{-1}, 3264cm^{-1} and 3167cm^{-1} for the dislocation regions. These bands are related to the valence vibration of hydrogen

bonded OH groups [26]: i.e. band 1 to the intra-molecular hydrogen bond of O(2)H---O, band 2 to the intra-molecular hydrogen bond of O(3)H---O, band 3 to the intermolecular hydrogen bond of O(6)H---O and band 4 to the O---H stretching respectively.

Peak wavenumber (without dislocation) (cm^{-1})	Peak wavenumber (with dislocation) (cm^{-1})	$\Delta\nu$(cm^{-1})	Bonds
3327	3332	5	OH stretching
2883	2882	-1	C–H symmetrical stretching
1724	1724	0	C=O stretching vibration
1623	1624	1	OH bending of absorbed water
1506	disappear	-	C=C aromatic symmetrical stretching
1423	1423	0	HCH and OCH in-plane bending vibration
1368, 1363	1367,1363	-1/0	In-the-plane CH bending
1325	1325	0	S ring stretching
1314	1313	-1	CH_2 rocking vibration at C6
1259	1261	1	G ring stretching
1245	1244	-1	C-C plus C-O plus C=O stretch; G condensed > G etherfied
1232	1231	-1	COH bending at C6
1204	1199	-5	C-O-C symmetric stretching, OH plane deformation
1152	1156	4	C-O-C asymmetrical stretching
1046	1043	-3	C-C, C-OH, C-H ring and side group vibrations
1020	1018	-2	C-C, C-OH, C-H ring and side group vibrations
994	996	2	C-C, C-OH, C-H ring and side group vibrations
895	894	-1	COC,CCO and CCH deformation and stretching
662	663	1	C-OH out-of-plane bending

Table 1. Bonds wavenumber related to regions without and with dislocations

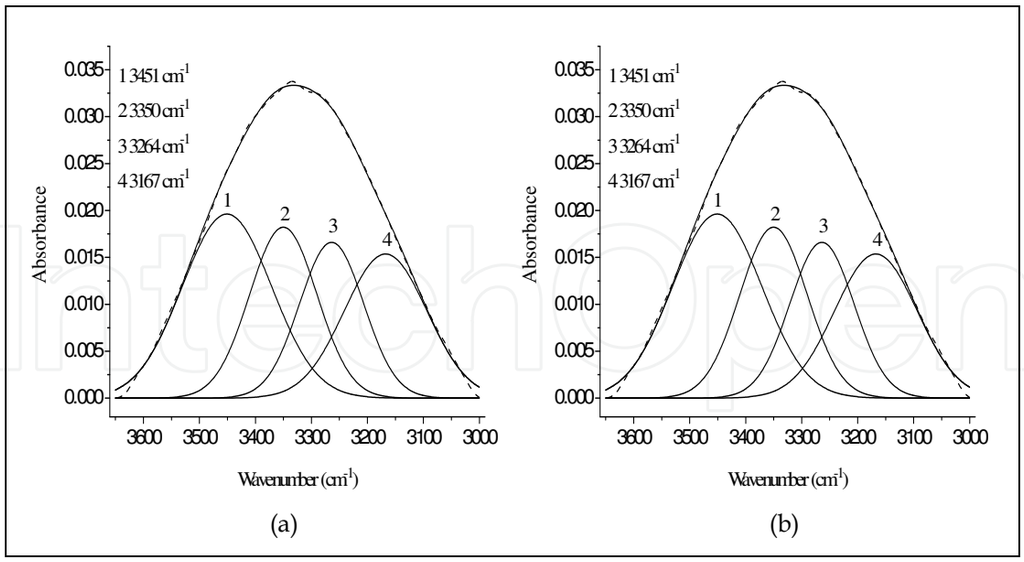


Fig. 4. Deconvolved FTIR spectra of the υ_{OH} region of hemp without dislocation (a) and dislocation regions (b). (Solid curves=calculated data; dotted curves=experimental data) [32]

It can be seen that the wave-numbers of peak position of dislocations are higher than those of hemp fibre without dislocation. This indicates that the degree of hydrogen bonding in dislocation regions is lesser than that in without dislocation regions. Furthermore, the absorbance of these bands in the dislocation regions is much lower than that in the regions without dislocations: for dislocation regions being about 79.3% for band 1, 64.4% for band 2, 64.9% for band 3 and 75.7% for band 4 those without dislocations respectively. These mean that the number of hydrogen bonds in dislocations is lower than without dislocation regions according to Beer–Lambert law.

3. Structure of natural fibres determined by using FTIR

The structure of cellulose has a remarkable and complex influence on the course of chemical reactions of the polymer (cellulosic materials). Generally, the structure of cellulose consists of three structural levels: namely (i) the molecular level of the single macromolecule; (ii) the supramolecular level of packing and mutual ordering of the macromolecules; (iii) the morphological level concerning the architecture of already rather complex structural entities, as well as the corresponding pore system [33]. This section only focuses on the molecular level and supramolecular level, and the morphological level will be discussed in the final section of this chapter.

Molecular orientation is one of the most important parameters, affecting the physical properties of macromolecular systems. It is often introduced in natural macromolecules by the mechanical deformation incurred during their processing. By using FTIR equipped with a microscopic accessory, Kataoka and Kondo [34] determined the molecular orientation of cellulose during the formation of wood cell wall by virtue of the C-O-C stretching mode parallel to molecular chains [23] (Figure 5). It was found that the molecular orientation of cellulose in the primary cell wall coincided with the direction of enlarging cellular growth. It is therefore that the cellulose in the (nascent) primary cell wall might be oriented during

crystallization and subsequent formation of microfibrils due to the drawing stress/effect exerted during cellular enlargement. This force, distributed along molecular chains, can cause β-glucose chains in the nascent cellulose to crystallize in the I_α phase with a higher crystallinity, making the molecules orientated in the enlarging direction.

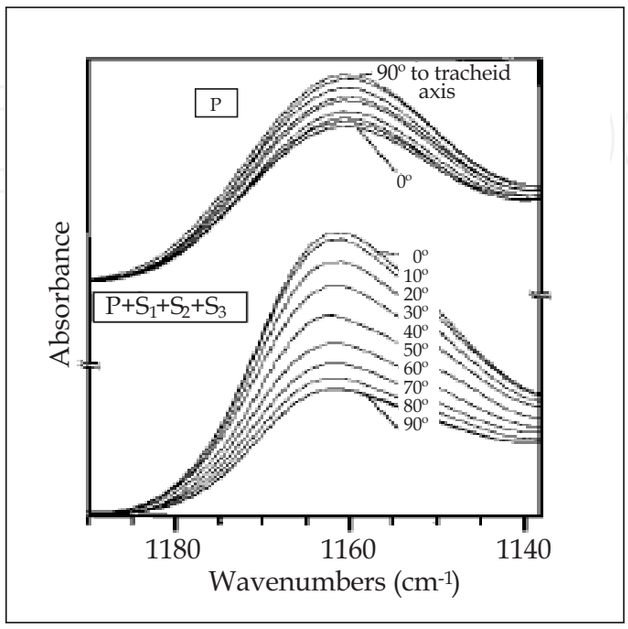


Fig. 5. Changes in FTIR spectra with a rotation of IR polarizer to the tracheid cell axis due to the C-O-C stretching mode: the primary (P) and the mature (P + S1 + S2 + S3) [34]

In order to better understand wood and wood fibres for their potential utilization in advanced materials, some researchers have employed FTIR in conjunction with mechanical loading to study the molecular responses to the stress/load, such as for spruce wood and cellulose paper materials [35] (Figure 6), illustrating the shift of the absorption peak at 1,160 cm-1, C–O–C vibration when the materials successively loaded from 0 up to 24 MPa at 0% RH. The decrease of the shift of absorption peak as the stress increased can be observed (6 wavenumbers in Figure 6). This decrease in wavenumber signifies an increase in the length of the covalent bonds involved in the vibration absorption, i.e. a decrease in the force constant of the bond. This demonstrates that FTIR-spectroscopy may be used to monitor molecular straining of cellulosic material under load and the molecular deformation is linearly related to the macroscopic load of the material. Using FTIR technologies, it was found that spectral deformations occurred in cellulose related groups, but no molecular deformation detected for the lignin or hemicelluloses of wood constituents. The molecular straining of the cellulose molecule resulted in greater macroscopic force under moist conditions compared to dry conditions, but an equal macroscopic strain under both conditions. This may be interpreted that moisture accessible regions are arranged tending in parallel with the cellulose load bearing entities, suggesting that the cellulose disordered regions may not exist as large regions across the cellulose aggregate structure, rather that are spread out. In addition, the moisture absorbing area of the cellulose structure is probably related to the surface areas of the cellulose.

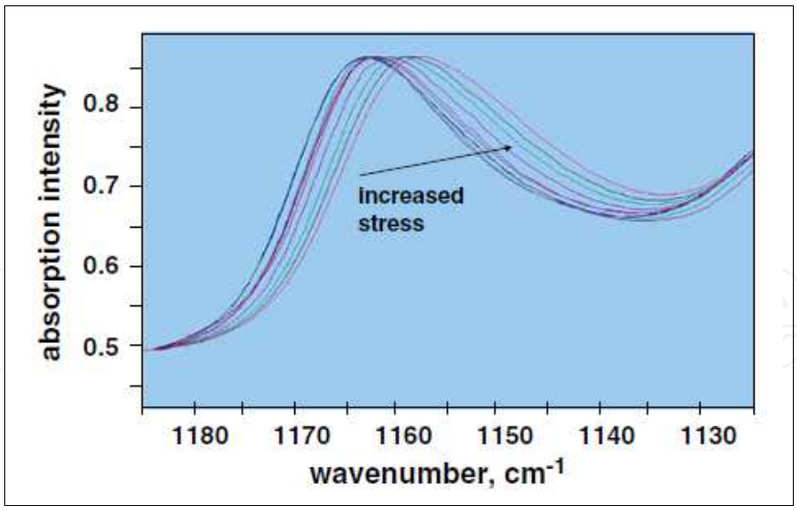


Fig. 6. Absorption spectra of the C–O–C vibration peak with increasing stress levels [35]

Polarized FTIR accompanied with a vapor-phase deuteration has been used to characterize orientation of the main chains and hence to study the molecular orientation of Nematic Ordered Cellulose (NOC) [36]. A ratio (R) of the absorbance of the band due to the particular molecular moiety for radiation polarized perpendicular to to parallel to the stretching direction was introduced to evaluate the orientation behaviour of the main chains and OH groups. Computation of the FTIR spectra (e.g. Figure 7) shows that R values for the main chain are 0.32, and OH group 0.81 for Intramolecular and 0.91 for intermolecular H.B.

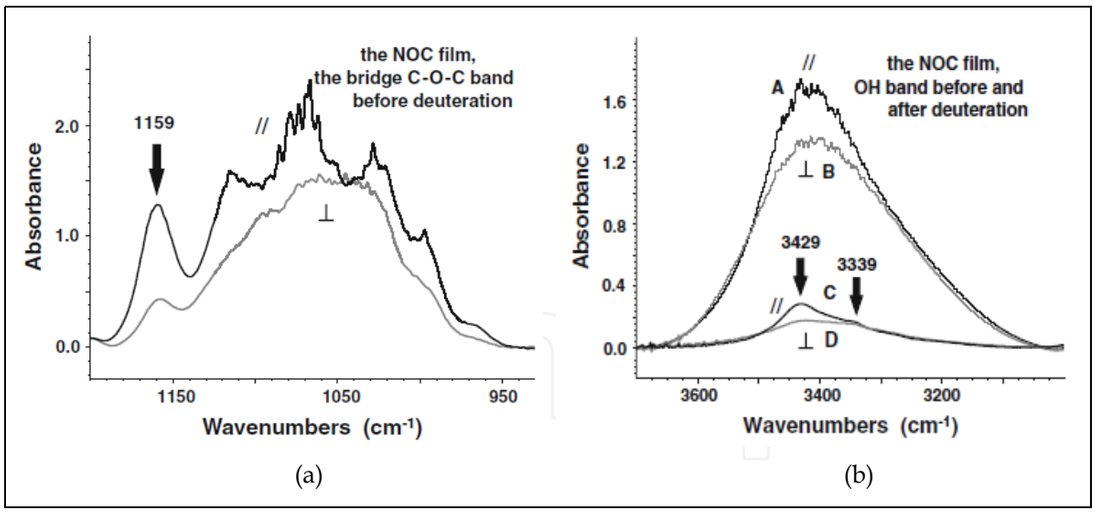


Fig. 7. The bridge C–O–C (a) and OH (b) stretching band for the NOC film before deuteration [for (b) (A, B)=before, (C, D)=after, (//)=electric vector parallel to and (⊥)=perpendicular to the stretching direction]

It is apparent that: (1) the R value for the β-glucan main chains of cellulose molecules is not necessarily in agreement with that for the side chains of OH groups; (2) the uniaxial drawing process to prepare the NOC film gave rise to the oriented main chains toward the stretching direction; (3) the nonoriented OH groups in the noncrystalline regions which

occupy more than 80% of the drawn film samples could be the key for discouragement of the crystallization.

Supramolecular level investigated with FTIR mainly focuses on the crystal structure, which includes: 1) hydrogen bonding, 2) crystallinity measurement and 3) cellulose I_α and I_β determination. Kondo, Togawa and Brown [37] proposed a concept to describe how various states of molecular association can be categorized in cellulose. Figure 8 demonstrates the schematic representation of their concept.

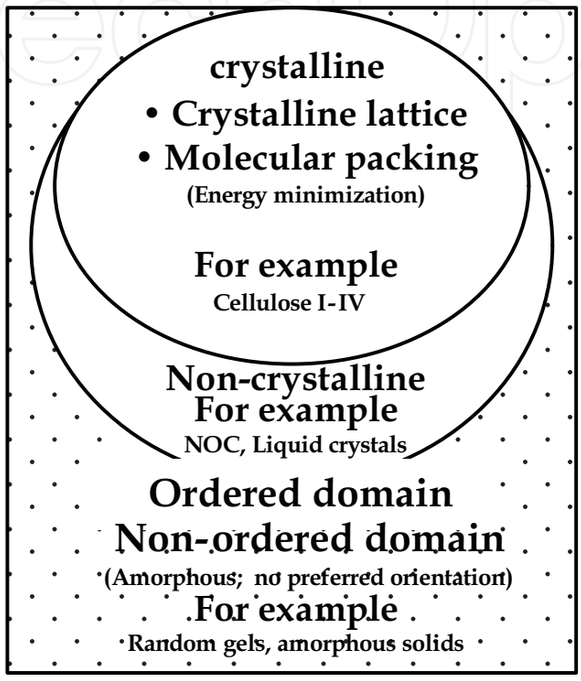


Fig. 8. Concept of glucan chain association for cellulose

According to two-phase model theory [38], there exist two regions in cellulose chain, namely amorphous and crystalline regions. Crystalline region in cellulose is an idealistic assembly of cellulose molecules in the biological system. There exist four different crystalline forms in cellulose. Researchers have developed various techniques to characterize the crystalline structure of cellulose, e.g. XRD, FTIR, Raman spectroscopy, and ^{13}C CP/MAS NMR. Among them FTIR is a more advanced tool for investigating the structure of cellulose. As mentioned above, since 1950s, some important work had been carried out by researchers and there are a number of literatures reporting on the IR/FTIR data of natural fibres [39].

The hydrogen bonds in cellulose mainly distribute in crystal domains and amorphous domains. It is possible to establish relation between the OH-bands and the cellulose structure. In 1913, Nishikawa and Ono [40] firstly revealed the crystalline nature of cellulose with X-ray diffraction. Cellulose has four polymorphic crystalline structures from cellulose I to cellulose IV. However, cellulose I and cellulose II have been most extensively studied. The other crystalline structures are still in question and yet to be studied further. According to Gardner-Blackwell model [41], hydrogen bonds for cellulose I include two intramolecular bonding, namely, O(2)H---O(6) bonding and O(3)H---O(5) bonding and one intermolecular bonding, O(6)H---O(3) (Figure 9a). Based on the Kolpak-Blackwell model [42], hydrogen

bonds in cellulose II contains three intramolecular bonds: O(2)H---O(6) bonding, O(3)H---O(5) bonding and O(2)H---O(2) bonding, and two intermolecular bonding: O(6)H---O(2) and O(6)H---O(3) (Figure 9b). The IR assignments for OH regions in cellulose I and II are summarized in Table 2.

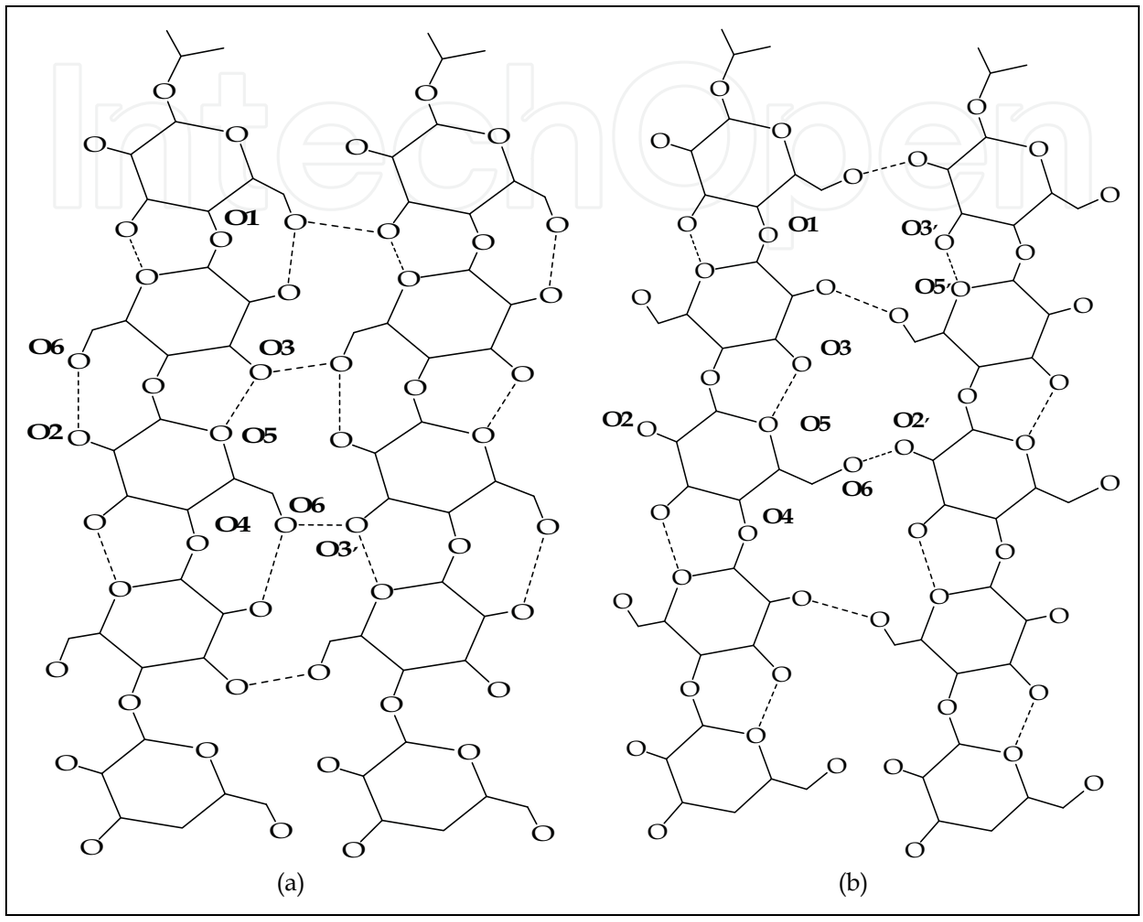


Fig. 9. Hydrogen-bonding network: (a) parallel to the *bc* plane (cellulose I); (b) in the centre chains (Cellulose II)

Peak wavenumber (cellulose I) (cm^{-1})	Peak wavenumber (cellulose II) (cm^{-1})	Bonds
	3175	OH stretching
3230-3310		O(6)H---O(3)
	3308	OH Inter H-bond
	3309	OH Inter H-bond
	3315	OH Intra H-bond
3340-3375		O(3)H---O(5)
	3374	OH Intra H-bond
3405-3460		O(2)H---O(6)
	3486	OH Intra H-bond

Table 2. Correlation of bonds and celluloses (structure) [43], [44]

Hatakeyama and his coworkers firstly studied the hydrogen bond in the amorphous regions of cellulose. These studies focus on investigating the effect of temperature on the formation of interchain hydrogen bonds [45], and the effect of hydrogen bonds on the temperature dependence of the dynamic modulus and the mechanical loss tangent [46]. In 1996, Kondo and Sawatari systematically examined the formation of hydrogen bonds in amorphous cellulose. The substituted amorphous cellulose derivatives, 6-O-, 2,3-di-O-, and tri-O-substituted methylcellulose, were used to model the components of amorphous cellulose. An artificial spectrum for amorphous cellulose was then quantitatively constructed by using compound IR spectra in order to investigate hydrogen bond formation in cellulose. The typical absorption wavenumber for the real and artificial spectra were summarized in Table 3.

Peak wavenumber (real) (cm^{-1})	Peak wavenumber (artificial) (cm^{-1})	Absorbance	Bond stretching
669	671	W	OH out-of-phase bending
899	892	M	Nonsymmetric out-phase ring
1040	1040	S	C-O
1070	1075	S	Skeletal vibrations C-O
1108	1108	S	Nonsymmetric in-phase ring
1159	1154	S	Nonsymmtric bridge C-O-C
1374	1375	M	CH bending
1420	1425	W	CH_2 symmetric bending
2892	2903	M	CH
3420	3457	S	OH

Table 3. Absorption wavenumber between the real and synthesized IR spectra of amorphous cellulose [43, 47]

The traditional two-phase cellulose model describes cellulose chains as containing both crystalline (ordered) and amorphous (less ordered) regions. A parameter termed the crystallinity index (CI) has been used to describe the relative amount of crystalline material in cellulose. The CI of celluloses has been measured using several different techniques including XRD, solid-state ^{13}C NMR, infrared (IR) spectroscopy and Raman spectroscopy. The determination of CI using FTIR spectroscopy is the simplest method. It should be noted that this method gives only relative values, because the spectrum always contains contributions from both crystalline and amorphous regions. In 1958, O'Connor [49] proposed Lateral Order Index (LOI, A_{1420}/A_{893}) to calculate the CI for cellulose. Later, Nelson and O'Connor [49-50] introduced Total Crystallinity Index (TCI, A_{1375}/A_{2900}) to evaluate the CI of cellulose. The absorbance ratio A_{1420}/A_{893} was defined as an empirical CI. The absorbance at 1420 and 894 cm^{-1} are sensitive to the amount of crystalline versus amorphous structure in the cellulose, that is, broadening of these bands reflects more disordered structure. As for TCI, various reports seem not to show a coherent result [51-52].

4. Chemical composition of natural fibres by using FTIR

Compositional variation and physical organization at the microscopic level determine the ability to perform a desired function for most materials. Lignocellulosic fibres from different lignocellulosic materials appear quite different, but the chemical composition is fairly similar although with different magnitudes of constituents. The major compositions of lignocellulosic fibres are cellulose, hemicellulose and lignin (see Figure 10), while the minor constituents include minerals, pectin, waxes and water-soluble components. The application of infrared spectroscopy in lignocellulosic fibres has a long history: The infrared spectroscopy was used to investigate the hydroxyl groups of cellulose in the 1930's [53] and significant efforts were made in the 1950's to assign the different absorption maxima in the IR spectrum of cellulose [54-59]; The absorption maxima in the IR spectra of lignin were investigated from 1940's [60-61] through 1950's [62-64]; The characteristic absorption maxima of hemicellulose were studied during the 50's [65-66].

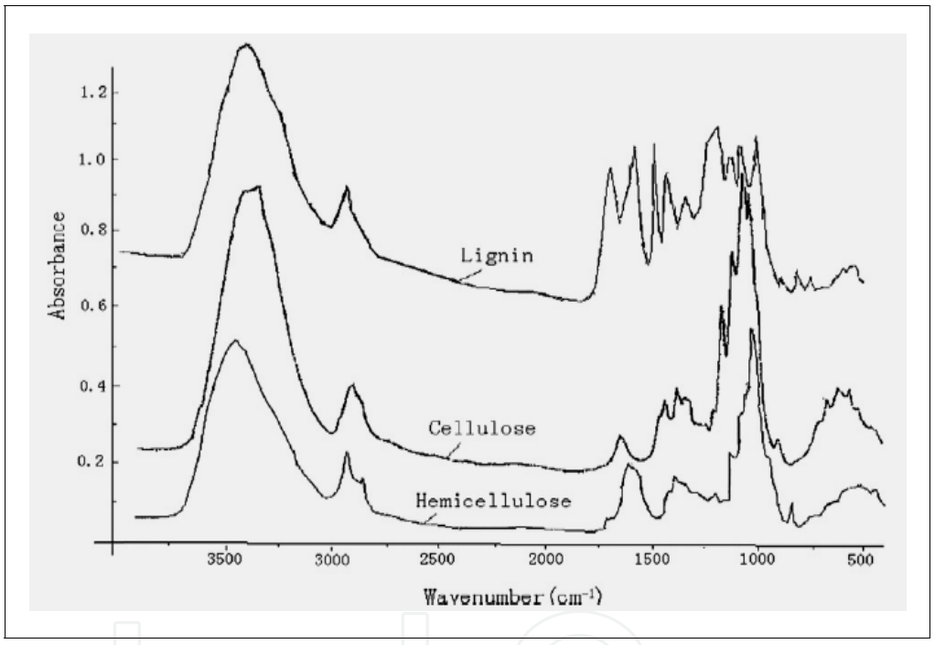


Fig. 10. IR spectra of cellulose, hemicellulose and lignin of natural fibres [60]

FTIR has been commonly used to characterize natural fibres with various treatments, e.g. grafting [67-68], coupling [69-71], mercerization [72-74]. With the aid of FTIR, researchers are able to obtain much more in-depth information of natural fibres after various modifications. FTIR is also an efficacy technique for the surface and interface characterizations of lignocellulosic fibres [75]. This allows further interpretation of the nature of adhesion between lignocellulosic with other substances. For example, Felix and Gatenholm [76] modified the lignocellulosic fibers with polypropylene–maleic anhydride copolymer. The spectrum of untreated fibres from the spectrum of treated fibres showed two peaks: one located at 1739 cm^{-1} and one at 1746 cm^{-1}, and the FTIR analysis indicated that the reaction between fibres and copolymer can be divided into two main steps: the copolymer is firstly converted into the more reactive anhydride form and then esterification takes place on the surface of cellulose fibres.

FTIR has recently been found most promising to examine the change of the chemical compositions of natural fibres (hemp fibres) due to inherent defects. An example of the results is given in Figure 11. A scrutiny of the IR spectra from 1370cm^{-1} to 1330cm^{-1} shows that the band at 1368cm^{-1} and 1363cm^{-1} almost disappears in dislocation regions (Figure 11a). These two bands, assigned as the in-plane CH bending, may be from hemicelluloses or cellulose, the near disappearance of these may be due to the removal of the hemicelluloses in dislocation regions. Hemicelluloses can form a linkage between cellulose and lignin, and lignin-carbohydrate complex with lignin by ether bonds [77]. The removal of hemicelluloses in dislocation regions may cause the decrease of transfer of shear stress under tensile loading and loss of lignin as well.

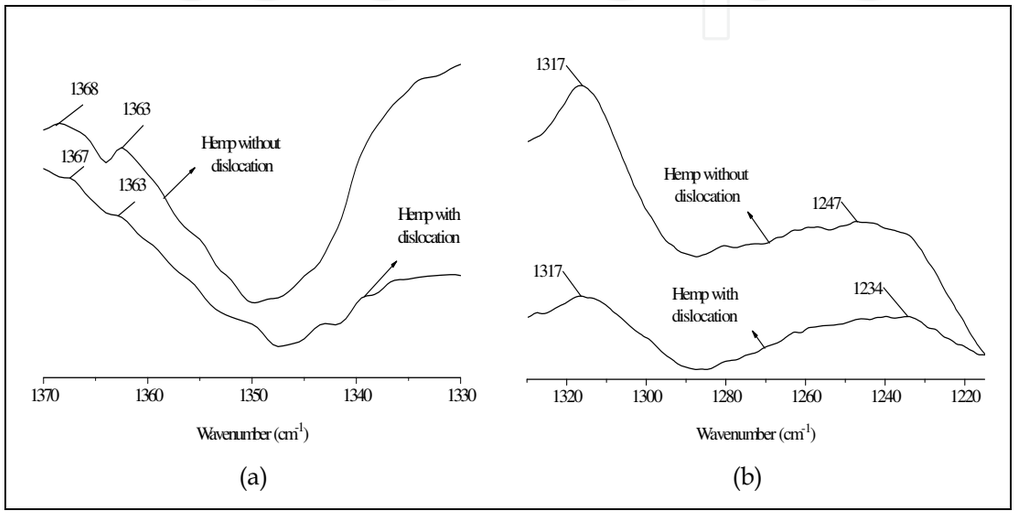


Fig. 11. FTIR spectra of hemp fibres from 1370 cm^{-1} to 1330 cm^{-1}(a) and from 1330 cm^{-1} to 1215 cm^{-1} (b) with and without dislocation [32]

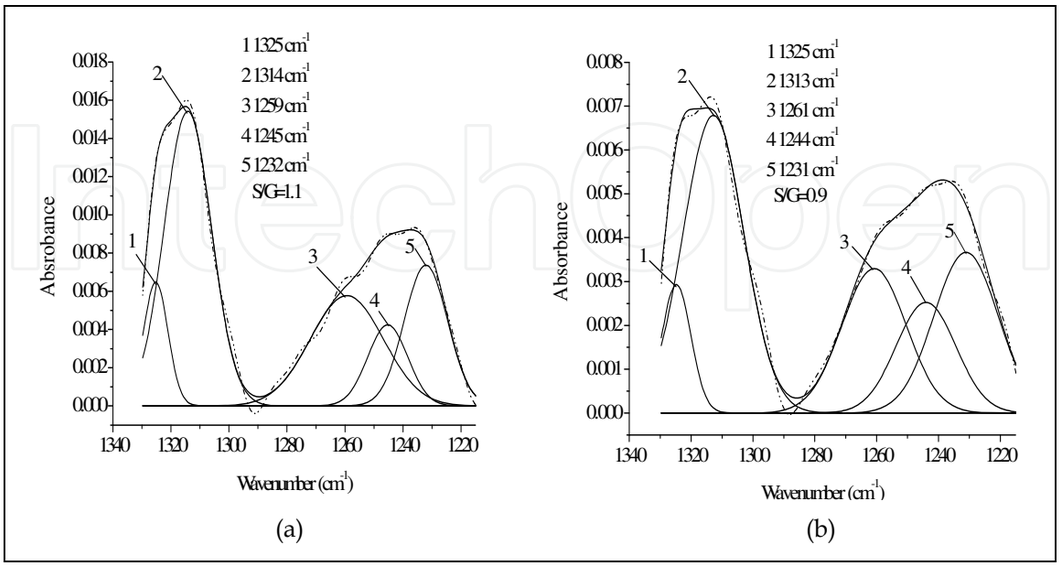


Fig. 12. Deconvolved FTIR spectra without dislocation (a) and dislocation regions (b). (Solid curves=calculated data; dotted curves=experimental data) [32]

The S ring (CH2 rocking at C6 in cellulose) and G ring stretching (C–C plus C–O plus C O stretch and COH bending at C6 in cellulose) could normally be observed in bands at 1325, 1314, 1259, 1245 and 1232cm^{-1} respectively for the hemp fibres without dislocation. Due to the overlapping of bands, only two peaks can be seen in Figure 11b. Lignin is composed of three basic units, namely p-hydroxyphenyl (H), guaiacyl (G) and syringyl (S) [78]. Guaiacyl (G) and syringyl (S) are the main units of lignin, but the ratio of S/G varies from one to another plant. It was reported recently by del Río et al. [79] that S/G values calculated upon FTIR were in agreement with those calculated upon Py-GC/MS at the bands of 1271cm^{-1} and 1327cm^{-1} respectively. However, the study on hemp fibre showed that the bands at 1271cm^{-1} and 1327cm^{-1}, assigned as G-ring stretching and S ring stretching respectively, were shifted to lower wavenumbers: for the hemp fibres without dislocations (Figure 12a), the G ring and S ring stretching appear at the bands of 1259cm^{-1} and 1325cm^{-1} and for the dislocation regions at 1261cm^{-1} and 1325cm^{-1} (Figure 12b).

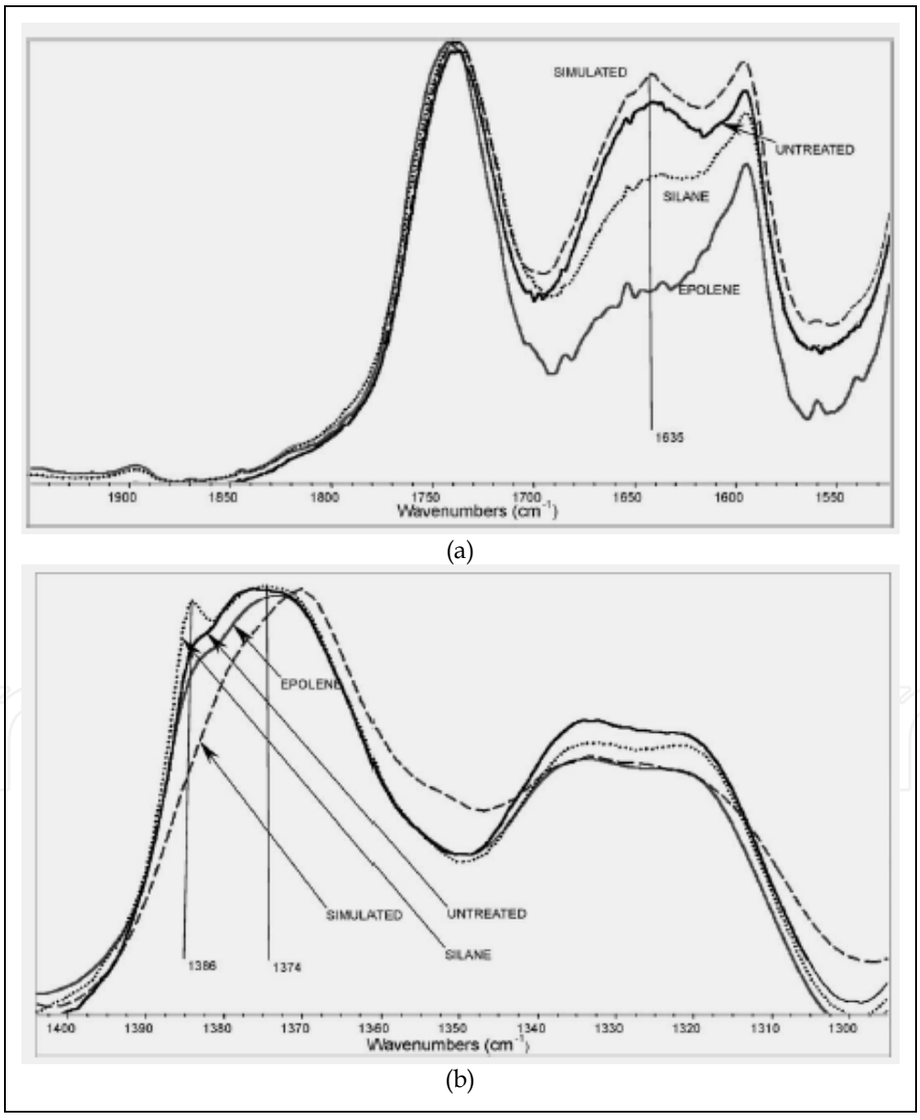


Fig. 13. FTIR spectra of various types of composites

The different molar contents of G-lignin and S-lignin of the hemp with and without dislocations gave rise to the ratio of S/G 0.9 for the former and 1.1 for the latter fibres. The lignin network in the parts without dislocations would be more rigid than that in dislocation regions. The lower absorbance in dislocations means that the lignin was removed from dislocation regions, and such the cellulose content in dislocations would be higher than that without dislocations.

FTIR can further be used to investigate the interfacial properties of natural fibre composites [80]. For example, Figures 13a and b exhibits the spectra for different types of composites containing 40% aspen fibres. The highest absorbance value corresponds to the untreated composites and the lowest value to that of composites modified with maleated polypropylene. The FTIR examination on the interface of wood fibre-reinforced polypropylene composites has also confirmed the efficacy of the technique [81]. The spectra are able to illustrate that the coupling agent was located around the wood fibers rather than randomly distributed in the polypropylene matrix, and the compatabilizer was attached to the wood fibers either by ester or hydrogen bonds.

5. Morphologies of natural fibres by using FTIR

FTIR spectroscopic imaging is the complete synthesis of FTIR spectroscopy with sample visualization and greatly extends the capabilities of conventional FTIR spectroscopy. Figure 14 illustrates a general configuration of an FTIR imaging micro- spectrometer. Spectral data can be represented as a picture, showing chemical information simultaneously from thousands of pixels. The main advantages of FTIR imaging are noninvasiveness, fast data collection and the ability to create visually appealing display. FTIR imaging not only provides new scientific capabilities, but it is also a compact and informative way to present results. It can collect more than 10,000 spectra in a few minutes. FTIR imaging has been

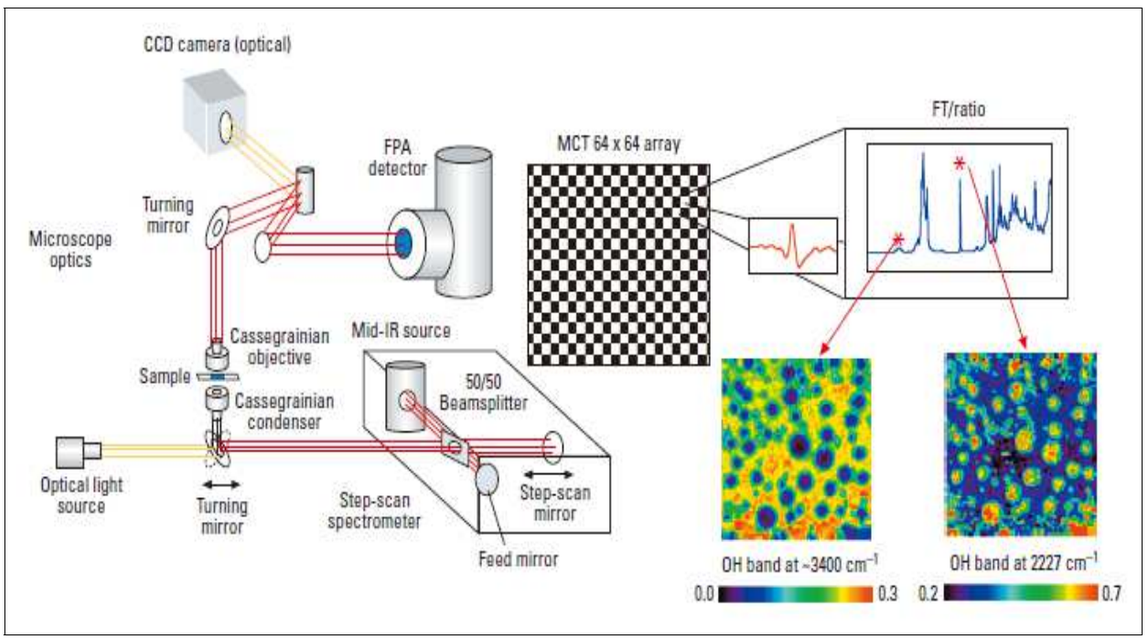


Fig. 14. Schematic of a typical FTIR imaging spectrometer [82]

found to be a remarkable tool for biological and materials analysis. It can be used extensively to investigate the chemical composition of stem [83-85] and cell wall structure [86] of natural fibres, and natural fibre composites [87].

FTIR imaging in conjunction with pyrolysis molecular beam mass spectrometry (py- MBMS) can work as a rapid analysis tool to evaluate difference in the chemical composition, for example, from the bark to the pith of wood stern (Figure 15) [85], and the data can statistically be processed to establish the correlation of the change in chemical features and the distance across the xylem (Figure 16).

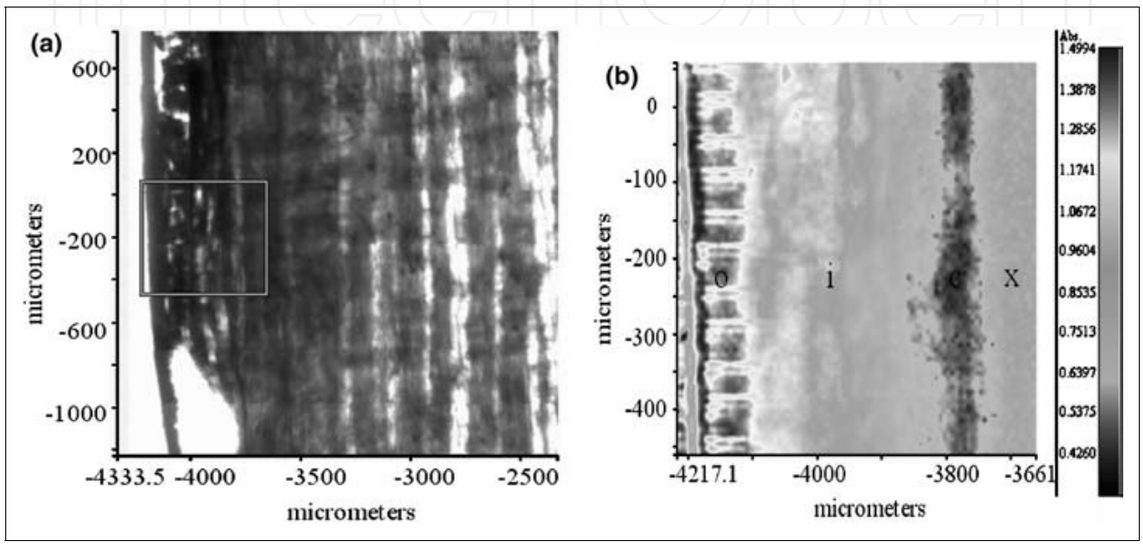


Fig. 15. (a) Visible image of the bark, cambium, and xylem of the control aspen stem. The area in the box was selected for FT-IR spectral analysis. (b) Spectral image of a portion of the outer bark [o], inner bark [I], cambium [c], xylem [x] showing the relative concentration of phenolic in these anatomical features [85]

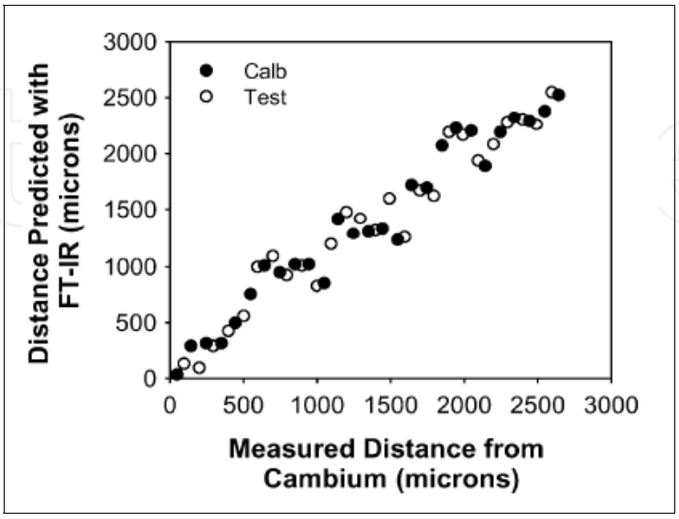


Fig. 16. PLS model predicting the distance from the bark to pith based on changes in the chemical composition. (Filled circles=calibration and open circles =test set) [85]

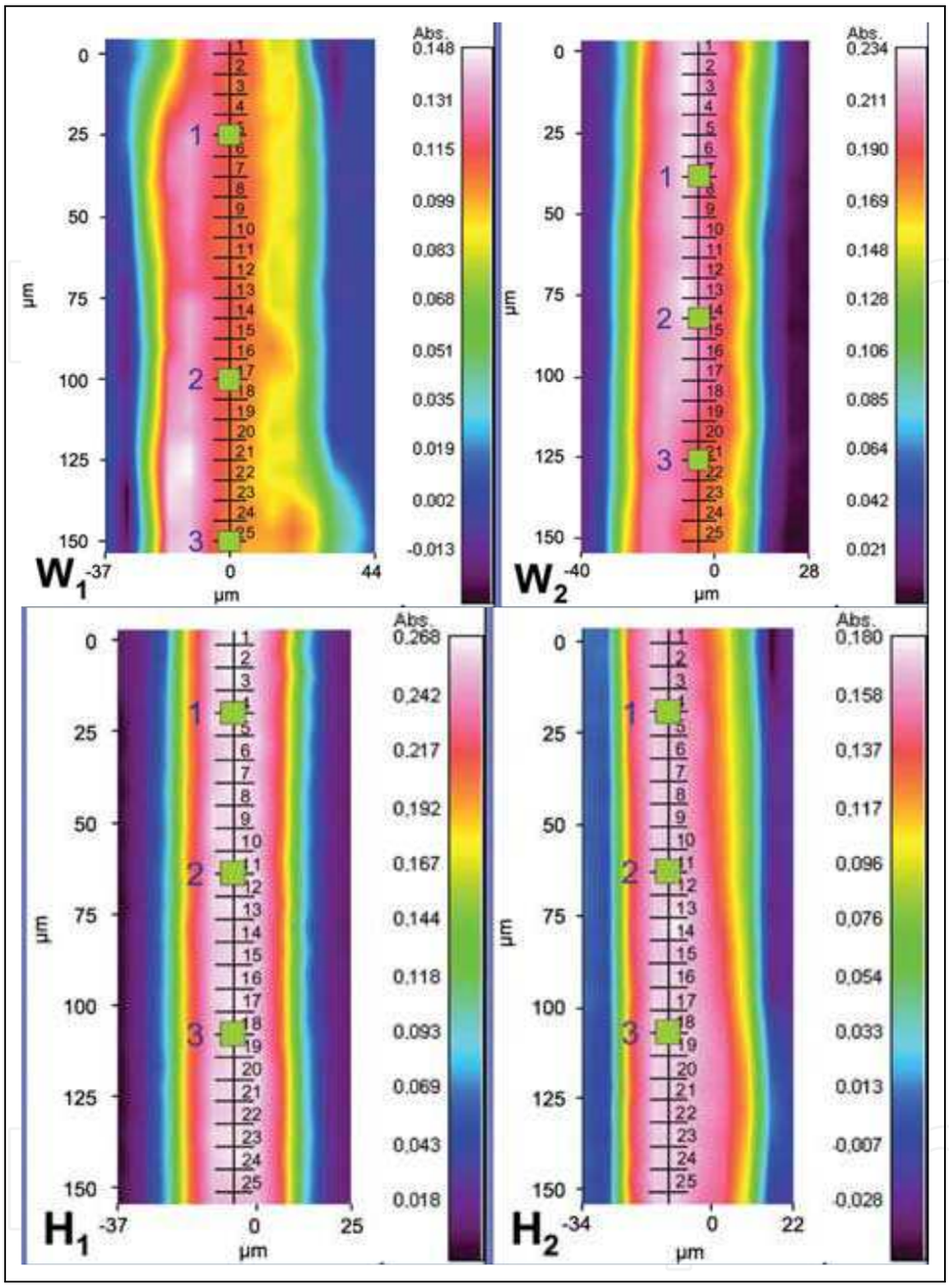


Fig. 17. Total IR absorbance full-spectral images of the two *W fibres* (*W*1 and *W*2) and the two *H fibres* (*H*1 and *H*2), showing the 25 pixel positions for each fibre used for evaluating the average orientation spectra as well as the three pixel positions for each fibre selected for evaluating the orientation of the different wood polymers in the fibres [86]

FTIR spectroscopy imaging has also been used to examine the orientation of the main wood compositions in transverse and longitudinal directions of wood fibres. For example, the examination by using FTIR on spruce fibres (Figures 17 and 18) [86] is able to illustrate that 1) glucomannan and xylan show a predominant orientation in the S_2 layer of cell wall,

2) hemicelluloses are arranged in parallel with the cellulose microfibrils and accordingly more or less in parallel with the longitudinal axis (the S_2 layer of the cell wall) of fibres, 3) only a little degree of orientation can be observed for lignin and 4) the variation in the molecular orientation along the fibres seems to be uniform in the pore-free regions. These results gave rise to a conclusion that all of three main components within fibres may have a clear anisotropic behaviour under mechanical stress, that is, their properties will be different in the longitudinal direction (along the fibre axis) and the transverse direction.

FTIR can be used to examine the structure of natural fibre based composites, such as, examining the surface distribution of polyacrylamide (PAM) or the in-plane distribution of cellulose within a paper sheet [87].

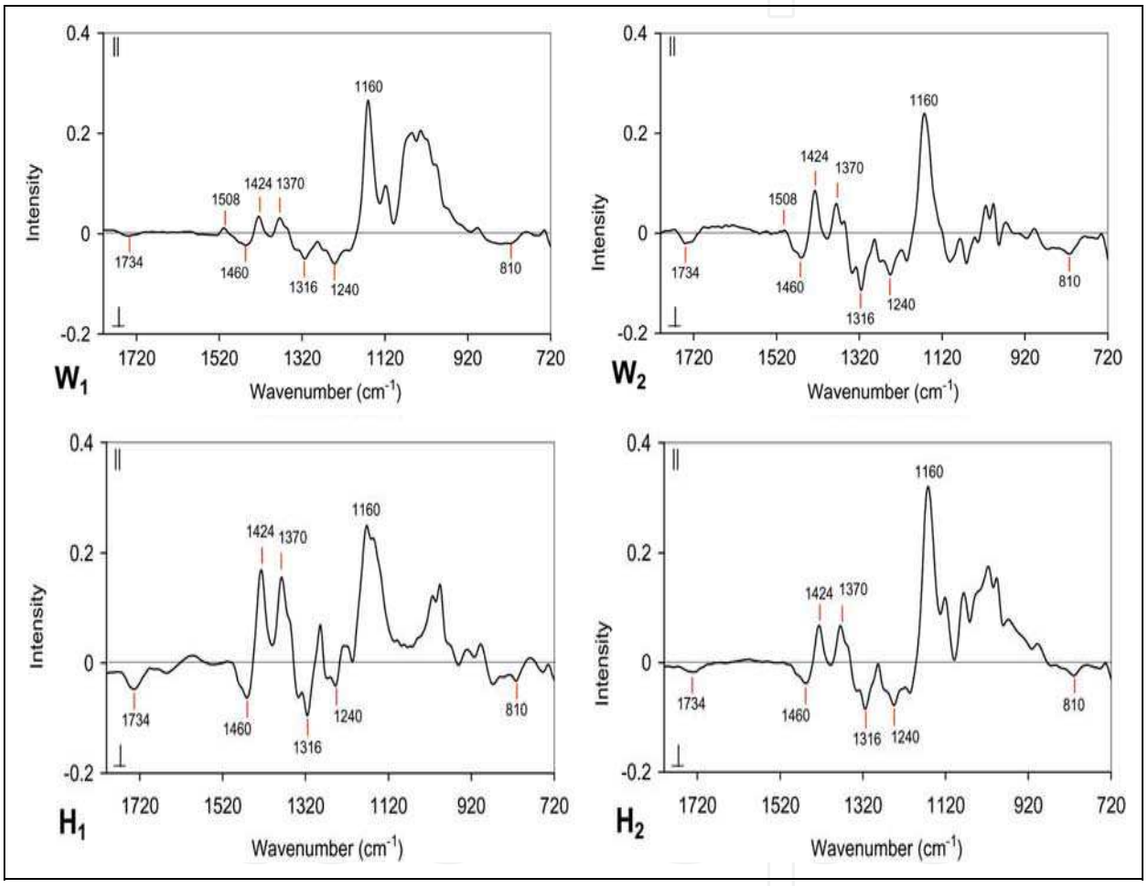


Fig. 18. Average orientation spectra of the two *W fibres* (*W*1 and *W*2) and the two *H fibres* (*H*1 and *H*2): *cellulose* 1160 cm^{-1}, 1316 cm^{-1}, 1370 cm^{-1} and 1424 cm^{-1}, *glucomannan* 810 cm^{-1}, *xylan* 1734 cm^{-1}, 1460 cm^{-1} and 1240 cm^{-1} and *lignin* 1508 cm^{-1} [86]

6. Conclusions

FTIR offers scientists an excellent range of solutions for understanding natural fibres and their related modification technologies and products, such as chemical compositions, microstructures, fibre architectures, characterisation of interface, and properties of both natural fibres and related composites.

FTIR is a powerful technique to examine the formation of inter- and intra- molecular hydrogen bonds in cellulose. The detailed database allows the establishment of strong correlation between the nature of hydrogen bonds and physical (e.g. solubility, hydroxyl reactivity, crystallinity) and mechanical properties of cellulose. The capability of accurate examination of hydrogen bonds has lead to an ever increasing uses of FTIR for investigating the defects (e.g. dislocation of hemp fibre) or deterioration (e.g. perturbation) of natural fibres and change of materials after modification.

The structure of cellulose has a profound influence on the course of chemical reactions of cellulose materials and the resulted properties. The molecular orientation and crystallization and formation of microfibrils not only vary from one plant to another, but could also change due to various environmental or other physical effects. FTIR is able to examine the nature of molecular chains, crystallinity and their correlations with various bonds.

In commons with other materials, the chemical composition at microscopic level determines the ability to perform various functions for the usefulness of natural fibres. FTIR has been mostly successful in accurate analysis of both major (cellulose, hemicellulose and lignin) and minor (mineral, pectin, waxes) constituents of natural fibres. Change in chemical compositions, interface and hence properties of natural fibres and composites could also be effectively identified by using FTIR.

FTIR is the most interesting and versatile of all analytical techniques and are well placed to become the technology of the century.

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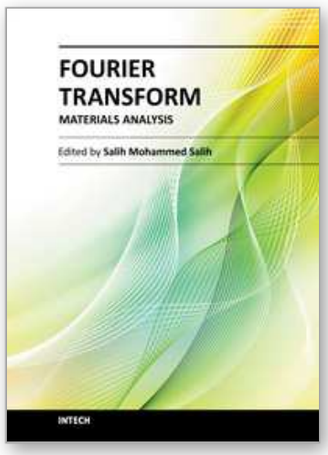
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The field of material analysis has seen explosive growth during the past decades. Almost all the textbooks on materials analysis have a section devoted to the Fourier transform theory. For this reason, the book focuses on the material analysis based on Fourier transform theory. The book chapters are related to FTIR and the other methods used for analyzing different types of materials. It is hoped that this book will provide the background, reference and incentive to encourage further research and results in this area as well as provide tools for practical applications. It provides an applications-oriented approach to materials analysis written primarily for physicist, Chemists, Agriculturalists, Electrical Engineers, Mechanical Engineers, Signal Processing Engineers, and the Academic Researchers and for the Graduate Students who will also find it useful as a reference for their research activities.

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