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Near Infra Red Spectroscopy- An Overview

Hari Prasad Reddy Aenugu, D.Sathis Kumar*, Srisudharson, N. Parthiban, Som Subhra Ghosh, David Banji

Nalanda College of Pharmacy, Nalgonda, Andhra Pradesh, India – 508001

Corres.author: satmpdina@yahoo.co.in,
Phone: +91 9966796051

Abstract: Spectroscopy is the chief experimental technique of atomic and molecular physics and involves determining the energy states of atoms or molecules by looking at the light absorbed or emitted when they change states. Measuring the frequency of light absorbed or emitted which is determined by the energy difference between the two states, can provide a sensitive probe of interactions which perturb those energy states. Based on the principle and the range of electromagnetic radiation spectroscopy is classified into several types. Among those in this review we revealed that the principle, instrumentation and applications of Near Infrared spectroscopy.
Key words: Near Infrared spectroscopy.

Introduction:

Infra red spectrum is an important record which gives sufficient information about the structure of a compound. In recent years, NIR spectroscopy has become so widespread in process analysis and within pharmaceutical industry for raw material testing, product quality control and process monitoring[(1)]. Not only in the pharmaceutical industry it has gained wide acceptance in biotechnology, genomics analysis, proteomic analysis, interactomics research, inline textile monitoring, food analysis, plastics, textiles, insect detection, forensic lab application, crime detection, various military applications, and is a major branch of astronomical spectroscopy and so on[(2)]. NIR absorption bands are typically broad, overlapping and 10–100 times weaker than their corresponding fundamental mid-IR absorption bands. NIR spectroscopy is a vibrational spectroscopic method belongs to the infrared light spectrum which is very close to the visible region (from about 750 to 2500 nm), where the most of organic and some inorganic compounds shows good reflectance or transmission properties. That means they are exhibiting good absorption of light at the NIR region[(3)]. Figure 1 express the range of electromagnetic radiation.

History:[(4)]

The history of NIR is begins with William Herschel in 18th century. He found the radiant heat beyond the red end while using large glass prism to disperse the sunlight onto the three thermometers having carbon blackened bulbs. The heat is known as “NIR radiation” and the spectrum as “NIR Spectrum”(figure 2). In the early 1880s, the photographic plate, invented in 1829 by Niepce and Daguerre, had some NIR sensitivity. Abney and Festing recorded the spectra of organic liquids in the range 1 to 1.2 μm in 1881 and they have recognized both atomic grouping and the importance of the hydrogen bond in the NIR spectrum. First commercial fiber optic spectrophotometers were introduced in the 1980’s[(5)]. Karl Norris from the U.S. Department of Agriculture recognized the potential of this analytical technique and introduced modern NIRS into industrial practice. Coblentz WW constructed a spectrometer using a rock-salt prism and a sensitive vibration and thermal disturbances and recorded the spectra of several hundred compounds in the 1- to 15-

µm wavelength region. Coblentz discovered that no two compounds had the same spectrum, even when they had the same complement of elements (e.g., the isomers propan1-ol and propan2-ol). Each compound had a unique "fingerprint." However, Coblentz noticed certain patterns in the spectra; for example, all compounds with OH groups, be they alcohols or phenols, absorb in the 2.7 µm region of the spectrum. In this way, many molecular groups were characterized.

Theory & Principles:[6]

Near-IR (NIR) is a spectroscopic method is based on molecular over tones and combination vibrations of C-H, O-H and N-H. Although the three techniques (MIR, NIR & RAMAN) are very different in several aspects, their basic physical origin is same. Combinations arise by interaction of two or more vibrations taking place simultaneously. For a given molecule, a normal mode of vibration corresponds to internal atomic motions in which all atoms move in phase with same frequency but with different amplitude. Additionally to these normal vibrations transitions corresponds to be called overtones. Such transitions are forbidden by the selection rules of quantum mechanics. As a result the molar absorptivity in the near IR region is very small.[7] There are two laws which govern the basics of vibrational spectroscopy, they are Hooke's law and Frank condon principle. Hooke's law states that, for two body harmonic oscillator, the frequency of vibration is

$$\tilde{\nu}\,(\text{in cm}^{-1}) = \frac{1}{2\pi c}\sqrt{\frac{k(m_1+m_2)}{m_1 m_2}}$$

Where, C = speed of light, K = force constant (5×10^5 dynes/cm).

Hooke's law can be used to calculate the fundamental vibrations for diatomic molecules in IR. Transition (**figure 3**) from the ground state to the first excited state absorbs light strongly in IR region and give rise to intense bands called the fundamental bands. Transition from the ground state to the second excited state with the absorption of NIR give rise to weak bands called 1st overtone in NIR. Transition from the ground state to the third excited state with the absorption of NIR give rise to weak bands called 2st overtone in NIR. Like wise 3rd and 4th overtone bands will occur based on the transition to the fourth and fifth excited state with the absorption of NIR.

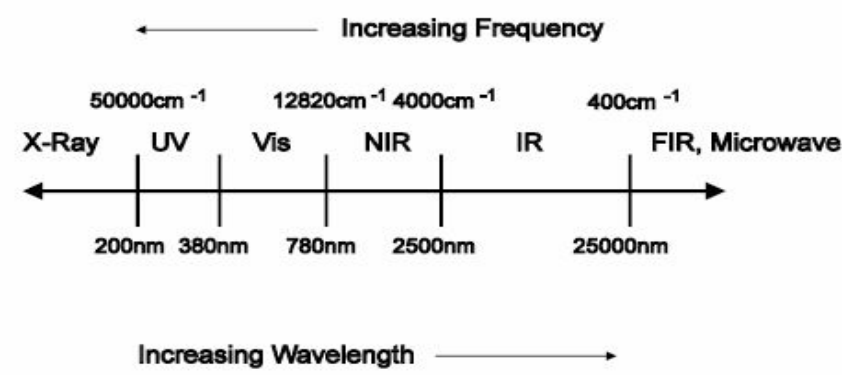

Figure 1: Range of Electromagnetic radiation

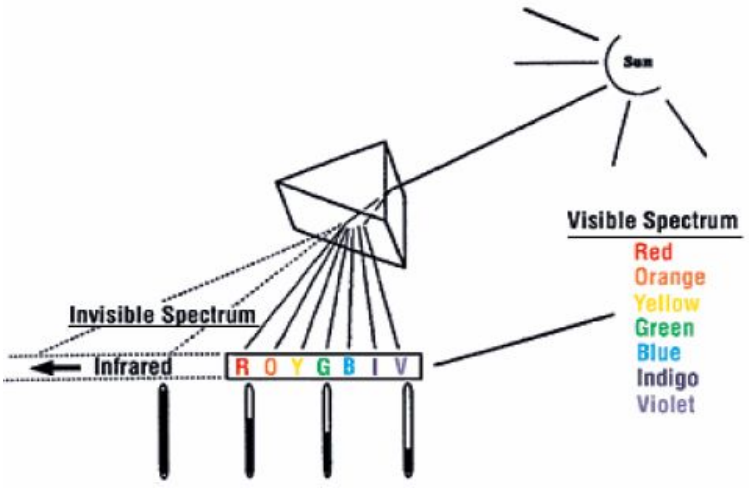

Figure 2: Dispersion of Electromagnetic radiation

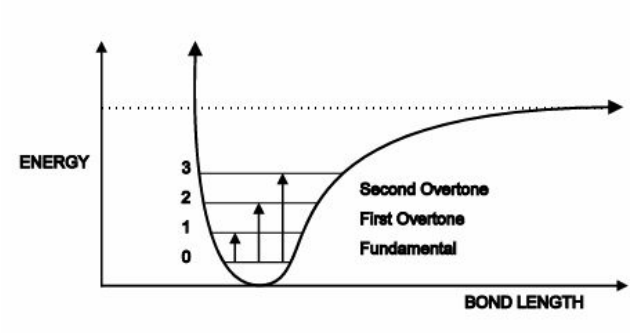

Figure 3: Vibrational transition

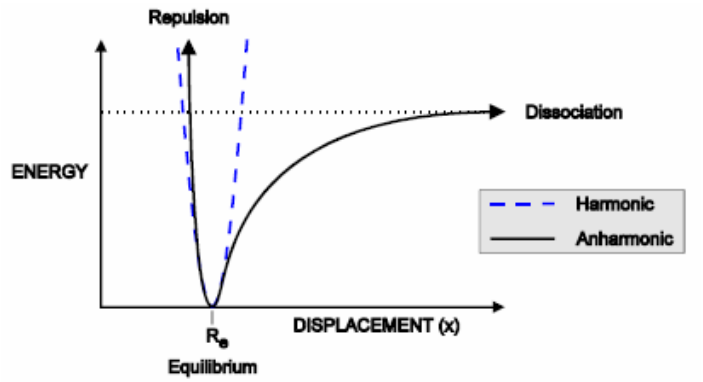

Figure 4: Harmonic and anharmonic oscillation

NIR is comprised of combinations and overtones that is anharmonic oscillation. Most molecules contain covalent bonds which share electrons between atoms. Although bonds are elastic, they do not obey Hooke's law exactly. The model of anharmonic oscillation is more precise. Harmonic oscillator can not be retained at larger amplitudes of vibrations owing to repulsive forces between the vibrating atoms and possibility of dissociation. Figure 4 has shown the harmonic and anharmonic oscillation.

Franck Condon Principle: When a molecule vibrates the probability of finding of given atom at a certain point is inversely proportional to its velocity. There fore the atoms in a vibrating molecule spend most of their time in configuration in which the kinetic energy is low, that is the configuration in which potential energy is nearly identical with total energy or at inter section of the vibrational energy level with the potential energy surface of the molecule. Thus the photon is most likely to be absorbed when the nuclei are stationary or moving slowly. The excitation resulting from the absorption of photon can not be transferred immediately to the nuclei. The nuclei will therefore tend to continue moving slowly after absorption. The nuclear configuration also tends to be close to intersection of the vibrational energy level with the potential energy surface of the molecule. Therefore transitions tend to take place between vibrational levels in which nuclear configurations are same in both states. Thus these small variations give rise to anharmonicity which causes the combination and over tone bands. The spectrum can be measured based on the sample nature either in transmission or reflection. Transparent materials are usually measured in transmittance, solids and turbid, semi solid solutions measured in diffuse reflection, transflection or transmission.[1] In the 1100–2500nm region, the amount of scattering makes the path length so great that transmittance through 1 cm of most samples, such as wheat meal, flour or milk powder, is negligible. This situation is called diffuse reflectance because most of the incident radiation is reflected. Hybrid mode of transmittance and reflectance called transflectance, where the radiation is transmitted through the sample reflected from a ceramic tile beneath the sample and then transmitted back through the sample before finally reaching the detector.[3]

Instrumentation: [3]

The most important source of Near Infra red light for scanning the spectrum of an organic compound is light emitting diodes(LED) which consists Gallium arsenide is used as semiconductor for near infrared light

emission, which emits the radiation at specific wavelength. These do not produce the radiation in the region of 1700 and 2500nm, which is very useful and active to NIR. The advantages of this source are they require low power so that these are used for production of portable spectrometers and have Long life expectancy. The tungsten lamps (incandescent bulbs) are also used as light source which produce heat up to 1100k. These lamps emit visible radiation along with considerable NIR radiation. So filters are needed in order to eliminate visible radiation which helps in prevention of unnecessary sample heating. The advantages of this source are cheap in cost and are readily available and these lamps can not increase their energy out put as the voltage of source is increased.

Most of the applications requires only selected wavelength. In some applications there is need to measure the response at many wave lengths. The existing method of wave length selection is a tool to determine the capacity of NIR instrument because it is possible to select the wavelength of interest. LEDs emit radiation at specific wavelength, but it is relatively broad energy emitted. LEDs combined with filters are using as wavelength selector and these are very efficient, cheap and ideal for portable, so low cost instruments can be produced without having moving parts. Gratings comprises metal or glass engraved surface with many fine parallel lines. When the light beam strikes the surface it divides into various wavelengths by diffraction. In interferometer wavelength selector, the light beam is split into two beams with a beam splitter. The two separated beams strikes the fixed and moving mirrors respectively and they are reflected back to beam splitter. They are then recombined and exit the interferometer in the direction of sample. Based on the material (KBr, CaF_2 or quartz) used for the beam splitter interferometer (figure 5) operate at specific wavelength.

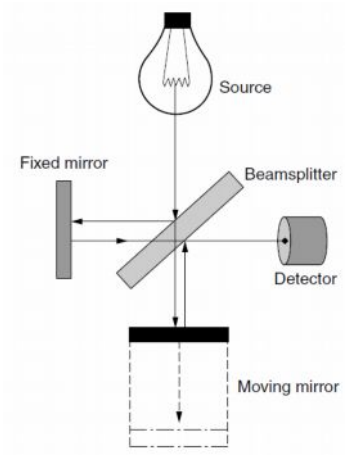

Figure 5: Diagram of beam splitter interferometer

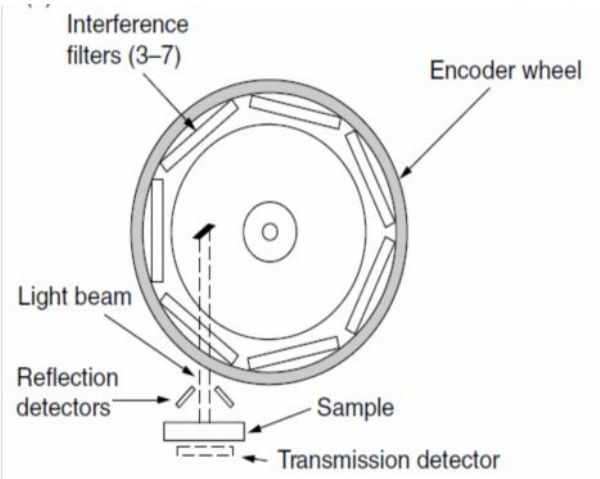

Figure 7: Flow diagram of Tilting filter

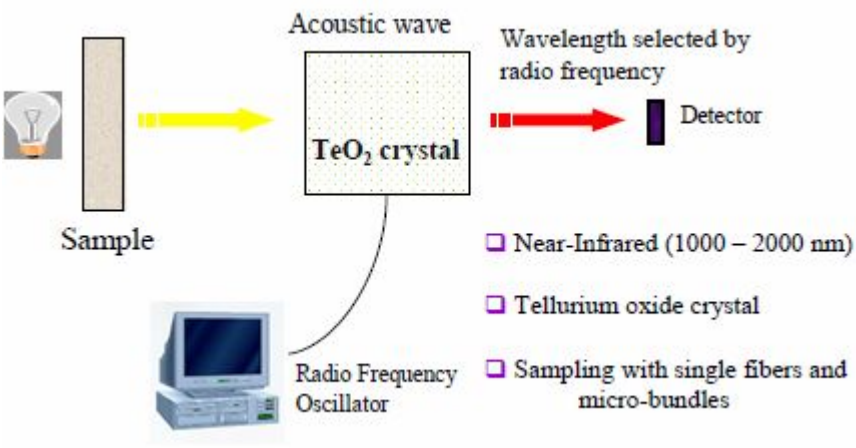

Figure 6: Flow diagram of AOTF used NIR spectroscopy

In AOTF (Acoustic Optical Tuneable Filter) wavelength selectors light is directed into a crystal of TeO_2 (figure 6). A high-frequency acoustic wave in the radio frequency range is coupled into the crystal by the use of a piezoelectric material bonded to the crystal. These acoustic waves quickly propagate through the crystal, interact with the broadband light and generate two monochromatic beams of light, each polarized in a different direction. These monochromatic beams are coupled by using optical fibres and can be used as a source of NIR light and sent to the sample. Advantages of AOTF are, it has no moving parts, adjustable intensity and gives narrow beams. The disadvantages include, it covers spectrum at limited wavelength range (1000-2000nm) and difficulties when measuring highly absorbing samples.

Tilting filter (figure 7) is the first type of interference filter. The transmitted energy at varied wavelength depends on the incident angle of light passing through the filter. In this, the filters were mounted in an encoder wheel for greater accuracy i.e. wavelength reproducibility.

Sample holder Cells made up of quartz or glass to perform transmission with liquids. Depending on the design of the instrument the cells are of varied sizes and designs. For example, some instruments use round sample cups for dry solid and grained samples. Transport cells are designed for analyzing the bulk samples. Round cups are often rotated in order to scan maximum amount of sample and to eliminate the lack of sample homogeneity. Transport cells move vertically where the scanning beam remains stationary.

The choice of detectors depends on Wavelength range, Spectrometer design characterstics, detector characteristics such as photosensitivity (responsivity), noise equivalent power (NEP), detectivity. Photosensitivity measures the voltage output per unit of incident radiant at a particular wavelength when noise is not considerable. NEP measures the quantity of light when the signal to noise ratio is 1. Detectivity is a parameter used to compare the performance of different detectors. Best detector should possess the higher in the signal of detectivity. Detectity is the signal to noise ratio at particular electrical frequency and in a 1HZ bandwidth when 1 watt of radiant on a $1cm^2$ active area detector. Detectors (**table 1**) using in NIR spectrometers are Lead sulphide detectos (PbS), Lead selenide detectors (PbSe), Silicon detectors, Indium antimonide detectors, InGaAs, InSb, Common Charged Coupled Devices (CCD).

Table 1: Detectors used in NIR spectroscopy

Detector	Wavelength range (nm)	Region	*Responsivity/**detectivity	Remark
PbS	1100-2500 400-2600 1100-4500	NIR UV-NIR NIR-MIR	Intermediate/intermediate	PbS 'sandwiched' with silicon photodiodes, are often used for VIS-NIR
PbSe	1100-5000	NIR-MIR	Fast/ high	The detector must be cooled with liquid nitrogen
InGaAs	700-1700	NIR NIR Raman	Fast/very high	Linear arrays high sensitivity, dynamic range, signal-to-noise performance and stability FT-NIR Diode arrays spectrometers
InSb/InAs	1000-5500	NIR MIR IR	Fast/ very high	High quality detector Detector photodiodes
CCD	800-2200	NIR	Fast/high	High performance detector Applied in cameras Diode arrays spectrometers

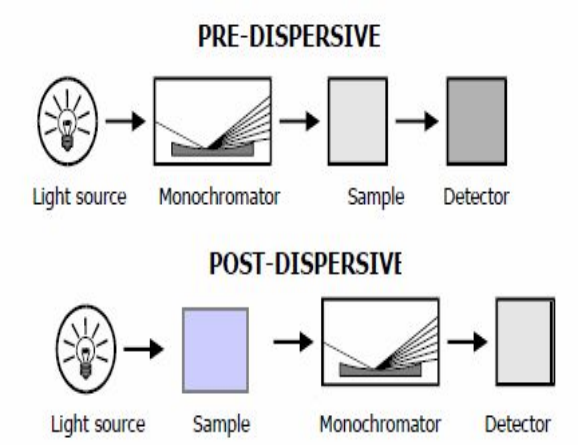

Figure 8: Scanning spectrophotometer

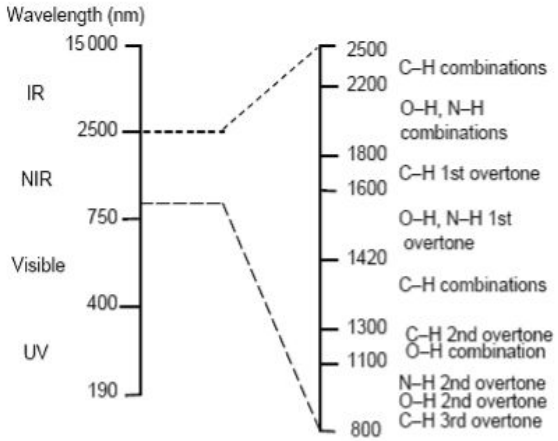

Figure 9: Characterisation of group absorption region

Types of Instruments: [5]

The types of NIR instruments are Scanning spectrophotometers, Fourier transform spectrophotometers, Acoustic Optical Tuneable Filter spectrophotometers, Photo diode array spectrophotometer.

In Scanning spectrophotometers (figure 8**)** the light projects from optical fibre onto difraction grating. The grating disperses the light into its constituent wavelength. The grating is mechanically rotated so that a narrow group of wavelength is allowed through the narrow slit. In predispersive instruments monochromatic light is sent to gratings through fibre optic cable. The dispersed light is transmitted through sample. The transmitted light passes through slit & reaches the detector. But in post dispersive instruments light is sent directly to the sample. The returning light from the fibre optic cable is directed to grating where it is dispersed and passes through slit placed before detector.

Fourier transform spectrophotometer is based on michelson interferometer. Acoustic Optical Tuneable Filter spectrophotometers are based on Acoustic Optical Tuneable Filters. In Photo diode array spectrophotometers detector is placed at an appropriate distance from a diffraction grating to analyze the complete sequence of wavelengths. Silicon PDAs can only be used below 1000nm. The more recent availability of InGaAs detector material makes the PDA more useful for NIR analysis. These detectors cover the range from 900-2200nm. In this spectrophotometer optical interference filters are used. These filters allow light only within a narrow wavelength range.

Characteristic Group Absorption Regions[4]

Near Infrared spectrum of compound provides more information than is normally available from electronic spectra. In this technique, some groups absorb characteristically within a definite range. The shift in position of absorption for a particular group may change with the changes in the structure of molecule. The force constant is responsible for the absorption peaks can be used to calculate bond distances and bond angle in simple cases. When the near infrared spectrum of unknown compound is scanned numbers of questions come to our mind such as which groups are present in the compound, what environments are influencing it or what type of carbon skeleton is present in the compound. The characteristic groups absorb light in definite frequency. So this technique is quite useful to predict the presence of functional groups and to identify the compounds. Figure 9 and table 2 express the characterization of group absorption region in NIR.

Advantages:

After the advent of fiber optics, bright light sources and sensitive detectors, NIR solved some of the difficult sampling techniques characteristic of IR, because NIR optical paths through liquid samples may be millimeters or even centimeters, rather than microns. Due to the much greater penetration depth of NIR radiation into the sample there is no need of sample dilution, matrices such as KBr or mineral oil and sample preparation. So that it minimizes sample preparation errors and sample destruction. [8]

Disadvantages:

A disadvantage of process NIR is the characteristics of the spectra, which are typically comprised of broad, overlapping peaks in comparison to IR spectra. No structural elucidation and not recommended for studying wetted samples (water bands are too strong). [8]

Table 2: Characterisation of group absorption region

Wavelength	Bond vibration	structure
1143	C-H second overtone	Aromatic
1160	C==O stretch fourth overtone	C==O
1170	C−−H second overtone	HC==CH
1195	C−−H second overtone	CH_3
1215	C−−H second overtone	CH_2
1225	C−−H second overtone	CH
1360	C−−H combination	CH_3
1395	C−−H combination	CH_2
1410	O−−H first overtone	ROH Oil
1415	C−−H combination	CH_2
1417	C−−H combination	Aromatic
1420	O−−H first overtone	ArOH
1440	C−−H combination	CH_2
1446	C−−H combination	Aromatic
1450	O−−H stretch first overtone	StarchH_2O
1450	C==O stretch third overtone	C==O
1460	Sym N−−H stretch first overtone	Urea
1463	N−−H stretch first overtone	$CONH_2$
1471	N−−H stretch first overtone	CONHR
1483	N−−H stretch first overtone	$CONH_2$
1490	N−−H stretch first overtone	CONHR
1490	O−−H stretch first overtone	Cellulose
1490	Sym N−−H stretch first overtone	Urea
1492	N−−H stretch first overtone	$ArNH_2$
1500	N−−H stretch first overtone	NH
1510	N−−H stretch first overtone	Protein
1520	N−−H stretch first overtone	Urea
1530	N−−H stretch first overtone	RNH_2
1540	O−−H stretch first overtone	Starch
1570	N−−H stretch first overtone	CONH
1620	C−−H stretch first overtone	$==CH_2$
1685	C−−H stretch first overtone	Aromatic
1695	C−−H stretch first overtone	CH_3
1705	C−−H stretch first overtone	CH_3
1725	C−−H stretch first overtone	CH_2
1740	S−−H stretch first overtone	--SH
1765	C−−H stretch first overtone	CH_2
1780	C−−H stretch first overtone	Cellulose
1780	C−−H stretch/HOH deformation combination	Cellulose
1790	O−−H combination	H_2O
1820	O−−H stretch/C−−O stretch second overtone combination	Cellulose
1860	C−−C1 stretch sixth overtone	C-Cl
1900	C==O stretch second overtone	$--CO_2H$
1908	O−−H stretch first overtone	P—OH
1920	C==O stretch second overtone	CONH
1930	O−−H stretch/HOH deformation combination	Starch
1940	O−−H bend second overtone	H2O
1950	C==O stretch second overtone	$--CO_2R$
1960	O−−H stretch/O−−H bend combination	Starch
1980	Asym N−−H stretch/amide IIb combination	$CONH_2$
1990	N−−H stretch/N−−H bend combination	Urea

2030	C==O stretch second overtone	Urea
2055	Sym N−−H stretch/amide Ib combination	Protein
2060	N−−H bend second overtone or N−−H bend	Protein
2070	N−−H deformation overtone	Urea
2070	O−−H combination	Oil
2090	O−−H combination	OH
2100	O−−H bend/C−−O stretch combination	Starch
2170	Asym C−−H stretch/C−−H deformation combination	HC==CH
2180	N−−H bend second overtone C−−H stretch/C==O stretch combination C==O stretch/amide IIIb combination	Protein
2200	C−−H stretch/C==O stretch combination	-CHO
2270	O−−H stretch/C−−O stretch combination	Cellulose
2280	C−−H stretch/CH2 deformation	Starch
2300	C−−H bend second overtone	Protein
2310	C−−H bend second overtone	Oil
2322	C−−H stretch/CH2 deformation combination	Starch
2330	C−−H stretch/CH2 deformation combination	Starch
2335	C−−H stretch/C−−H deformation	Cellulose
2352	CH2 bend second overtone	Cellulose
2380	C−−H stretch/C−−C stretch combination	Oil
2470	C−−H combination	CH_2
2470	Sym C−−N−−C stretch first overtone	Protein
2488	C−−H stretch/C−−C stretch combination	Cellulose
2500	C−−H stretch/C−−C and C−−O−−C stretch	Starch
2530	Asym C−−N−−C stretch first overtone	Protein

Applications:

Determination of pharmaceutical dosage forms

In 1966, Sinsheimer and Keuhnelian investigated a number of pharmacologically active amine salts both in solution and in the solid state by NIR spectroscopy. Quantitative analysis of the samples in solution was performed using the 2150 to 2320 nm region. Spectrum of the solid samples collected in the 1050 to 2800 nm region was analyzed qualitatively using peak assignments. Several spectral features were noted as showing promise for the quantitation of drugs in the solid state. The first use of NIR for tablet drug content was reported in 1968 by Sherken. In this study, an assay for meprobamate in tablet mixtures and commercially available preparations was established. A range of standard solutions of meprobamate analyzed by the NIR method was used for calibration development. In 1977, Zappala and Post investigated the use of NIR in the analysis of meprobamate in four pharmaceutical preparations: tablets, sustained-release capsules, suspensions, and injectables. [(4)]

Mark S. Kemper et al., were prepared Gels using Carbopol 980 with 0%, 1%, 2%, 4%, 6%, and 8% ketoprofen and analyzed with an FT-NIR spectrophotometer operated in the transmission mode. The correlation coefficient of the calibration was 0.9996, and the root mean squared error of calibration was 0.0775%. The percent relative standard deviation for multiple measurements was 0.10%. The results prove that FT-NIR can be a good alternative to other, more time- consuming means of analysis for these types of formulations. [(9)]

Alba Eustaquio et al., had taken the Production batch samples of paracetamol tablets having percentage purity 90–110% of the stated amount (500 mg) were analysed by the BP official UV assay and by NIR transmittance spectroscopy. NIR measurements were made on 20 intact tablets from each batch, over the spectral range 6000–11 520 cm. An average spectrum was calculated for each batch. Partial least squares (PLS) regression models were set up using a calibration set (20 batches) between the NIR response and the reference tablet paracetamol content (UV). analysis based on this mathematical transformation is partial least squares (PLS) regression. The method is easy to use and does not require knowledge of the mass of the tablets. [(10)]

Cellulose and lactose are the most frequently used excipients in illicit ecstasy production. Ines Baer et al., were used near infrared reflectance spectroscopy (NIRS) for the determination of the different chemical forms of these two substances, as well as for the differentiation of their origin (producer). It was possible to distinguish between the different chemical forms of both compounds, as well as between their origins (producers). First, a few cellulose and lactose samples were chosen to make mixtures with amphetamine at three degrees of purity (5, 10 and 15%), in order to study the resulting changes in the spectra as well as to simultaneously quantify amphetamine and identify the excipient. A PLS model could be build to predict concentrations and excipient. [(11)]

M. Donoso et al., used NIRS method as an alternative method to current tablets hardness and porosity equipment. Seven theophylline tablet formulations of same composition but different hardness values (3, 6, 8, 10, 12, 15 and 17 Kp) were prepared. Seven formulations of same composition but different porosity values (47.7%, 35.5, 24.6, 22.6, 22.1, 18.7, and 15.9 %) were also prepared. Five placebo tablet formulations with different hardness and porosity values were also prepared. Measure the diffuse reflectance from tablet surface. Calibrations curves between hardness or porosity values of theophylline tablets using lab instruments versus NIR absorbance were plotted. Model equations (PLS) were developed to predict tablet hardness and porosity from NIR spectra. An increase in hardness produced a decrease in porosity and increase in absorbance of NIR spectra. [(12)]

L Maric et al., determined the content of the active pharmaceutical ingredient (metformin hydrochloride) in tablets by using NIRS. The API content is determined by the validated HPLC method. NIR spectra were analyzed by the method of partial least squares. The best result for drug content determination was obtained for the raw spectrum, where the coefficient of correlation for calibration model was 0.99945, RMSEC 0.552, and RMSEP0.570. it was concluded that both models ensured a high coefficient of correlation as well as low level of error of the calibration model and prediction. [(13)]

Neville W. Broadet al., had taken the tablets produced containing 5, 10, 15, 20 and 30 mg. Reference values for the individual tablets used in the NIR calibration models and test set were measured by reversed-phase HPLC. Partial least squares regression using standard normal variate transformed second-derivative spectra over the range 800 to 1040 nm gave the optimum calibration model with a standard error of calibration of 0.52 mg per tablet[(14)].

Aditya S. Tatavar et al., were used near infrared (NIR) spectroscopy for the determination of content uniformity, tablet crushing strength (tablet hardness), and dissolution rate in sulfamethazine veterinary bolus dosage forms. Eight different formulations consisting of sulfamethazine, corn starch, and magnesium stearate were prepared Content uniformity can be modeled based on the absorbance at a specific wavelength or a combination of wavelengths. The data is analysed by multi linear regression (MLR) and partial least square (PLS) methods. For the sulfamethazine boluses, as the crushing strength increases, diffuse reflectance decreases and absorbance increase[(15)].

Matheus P. Freitas et al., compared dissolution profiles obtained by using a dissolution apparatus (conventional method) and the NIR diffuse reflectance spectra of a series of clonazepam. Ten different formulations with fixed amount of clonazepam and varying proportions of excipients were analyzed at seven dissolution times and three different media. The percentages of dissolution of each sample were correlated with the NIR spectra of three tablets of each batch, through a multivariate analysis using the PLS regression algorithm. The squared correlation coefficients for the plots of percentages of dissolution from the dissolution apparatus versus the predicted values, in cross-validation, varied from 0.80 to 0.92, indicating that the NIR diffuse reflectance spectroscopy method is an alternative, nondestructive tool for measurement of drug dissolution from tablets[(16)].

Simin Hassannejad Tabasi et al., studied dissolution behavior of sustained release theophylline matrix tablets using near infrared (NIR) diffuse reflectance spectroscopy and multivariate calibration models. Eudragit NE 30D was used as a granulation binder to prepare theophylline sustained release tablets. A total of 117 tablets from 5 batches containing different proportions of Eudragit NE 30D were scanned using a NIR spectrometer. The release characteristics of the tablets were investigated in the acetate buffer for 4 h. The percentage release at 1, 2, 3 and 4 h was used to build the PLS calibration models. For $PLS_{1\,h}$, the standard error of calibration (SEC), and standard error of prediction (SEP) were 2.8 and 3.4%. For $PLS_{2\,h}$, the SEC and SEP were 2.7 and 3.5%. For $PLS_{3\,h}$, the SEC and SEP were 2.6 and 3.5% and for $PLS_{4\,h}$, the SEC and SEP were 3.0 and 3.5%, respectively[(17)].

The identification and quality control of incoming materials is a straight forward application, which can be performed directly on the platform without any further time consuming tests. Organic solvents have different functional groups like alkyl-, hydroxyl-, aromatic CH-links or others. These show characteristic absorption bands, which support the automatic identification[18].

Due to the variability of the result in the UV/VIS determination of hydrocortisone sodium succinate, K. Nikolich et al., ascertained the use of NIRS analysis and was shown to be a more appropriate quantitative spectroscopic tool for such determinations. Hydrocortisone sodium succinate concentration in the range from 89.05%to 95.83 %, were analysed by NIR and UV/VIS spectroscopy. A correlation coefficient of 0.9758 and a standard error of cross validation (RMSECV) of 1.06%were found between the UV/VIS and the NIR spectroscopic results of the hydrocortisone sodium succinate concentration in the samples[19].

C. Bodson et al .,Validated the manufacturing process of Diltiazem Diltiazem HCl tablets(which were prepared by Direct compression) First, a partial least squares model was built to calibrate the NIR spectrometer. Then, this model was validated and compared with a validated UV spectrophotometry reference method.The manufacturing process was validated by producing three batches at three different concentration levels. The NIR analysis of these batches was performed during 3 days. This study shows that NIRS can be used to validate the whole manufacturing process[20].

Analysis of polymorphs[4]

Gimet and Luong were used NIR spectroscopy for the quantitative determination of polymorphs in a formulation matrix. Most polymorphs exhibit spectral differences in the mid-IR, since NIR spectra arise from overtones and combinations of mid-IR absorbances, NIR was also reasoned to be suitable for the analysis of polymorphs.

Determination of Adulteration of African Essential Oils[21]

near-infrared (NIR) technologies using for the assessment of essential oil components and in the identification of individual essential oils. NIR can be used to discriminate between the ravintsara(adultrant) and ravensara(authentic) essential oils. It was shown that two commercial oils labelled as R. aromatica were actually ravintsara (C. camphora).

Monitoring of PVC industrial blending process:[22]

Lidia Maria Bodecchi et al., had a work which deals with a feasibility study on the monitoring of the industrial production of PVC based semifinished products, namely dry blends, employed in turn for the manufacturing of disposable circuitries for extra-corporeal haemodialysis. A sample of dry blend, for each batch corresponding to different lot of resin, was then collected. The instrumental technique selected for the examination of all materials was the Near Infra Red Spectroscopy (NIR). The collected NIR signals were processed by using Principal Component Analysis (PCA) algorithm. The combination of NIR and PCA was able to: i) discriminate resins claimed as identical but coming from different suppliers and different production plants ii) detect batch-to-batch special variations probably due to different polymerization technologies of the PVC resins.

Identification of inorganic preservative-treated wood: [23]

Chi-Leung So et al., distinguished the treated and untreated waste wood using NIR. near infrared (NIR) spectroscopy with multivariate analysis (MVA) can distinguish preservative types and retentions. Within the range of preservative concentrations available, partial least squares (PLS) regression was also performed on the NIR data, from which retention levels were predicted. The results highlight the potential for this technique to predict the concentration, as well as identify the type, of inorganic preservatives present.

Protein quantification within lipid implants[24]

Lipid implants have been proposed as promising sustained release devices for the parenteral application of pharmaceutical proteins. Tilo schon brodt et al., used Near infrared spectroscopy (NIRS) as tool for drug quantification within controlled release matrix systems based on poly-(lactic-co-glycolic) acid (PLGA). Bovine serum albumin (BSA) and rh-interferon α-2a (IFN α-2a) were initially lyophilized with trehalose and then blended with tristearin (matrix material) and optionally with polyethylene glycol 6000 (PEG, release modifier). Implants were prepared by compression. NIR transmittance spectra were measured on a NIRTab® spectrometer. Partial least squares regression (PLSR) calibration models were developed to predict protein content in implants from the NIRS results.

Determination of Moisture Content in Freeze-dried Materials[25]

FT-NIR analysis determines the concentration of moisture in lyophilized materials quickly and with no

sample preparation or reagents . Eight batches of lyophilized Leucovorin Calcium were obtained. The moisture content of each batch was measured by gravimetry (loss on drying). These values are taken as standard values. Moisture in lyophilized preparation of folinic acid calcium salt(Leucovorin Ca) was also determined by FT-NIR spectra. Stepwise multiple linear regression (SMLR) was used for calibration. The calibration results showed that FT-NIR spectroscopy is a suitable method for quantification of moisture in freeze dried materials.

Validation of manufacturing process of Diltiazem HCl tablets [25]

Intravascular near-infrared spectroscopy:

Franziska H Bernet et al.,studied with fifteen patients using OPCAB (off-pump coronary bypass) technique indicated that intravascular NIR spectroscopy is technically feasible. Nevertheless, a refinement and standardization of NIR-data acquisition is necessary and more work is required to understand the behavior of NIR light in myocardial tissue. [26]

Parsons et al., used NIR spectroscopy for myocardial ischemia detection in open chest studies with dogs, concluding that the application of NIR spectroscopy is suitable for assessment of the tissue oxygenation relation to contractile function during ischemia[27]. Kupriyanov et al. examined changes in hemoglobin oxygenation to study tissue perfusion and flow in pigs with NIR spectroscopy[28].

Conclusion:

We concluded that this near infra red spectroscopy is also one of the best techniques due to its applications.

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