QUALITATIVE AND QUANTITATIVE ANALYSIS ON CIPROFLOXACIN AND TETRACYCLINE.

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Abstract: Modern spectroscopic investigational methods are very sensitive and effective tools for the analysis, both qualitative and quantitative of many drugs. The qualitative and quantitative investigation were made on two antibacterial drugs namely, Ciprofloxacin and Tetracycline through the spectroscopic studies. From the recorded FTIR and FT Raman spectra, an effective analysis was made by taking a sufficient vibrational tentative assignments of the fundamental mode of vibrations.

Key words: FTIR, FT RAMAN, Ciprofloxacin, Tetracycline, Antibacterial drugs.

I. Introduction:

Ciprofloxacin and Tetracycline are considerably made use of many microbial infection containing Gram negative, Gram positive bacteria, chlamydiae and rickettsiae (Ruiz,N.M and Rámirez-Ronda 1990, Ian Chopra and Marilyn Roberts, 2001, Jr Feder H.M. et al., 1981) . Ciprofloxacin is broadly used to treat large number of infections, which includes, respiratory track infections, urinary track infections, tuberculosis, endocarditis, gastroenteritis, etc., (Sean C Sweetman and Martindale 2009). It is employed to use before certain types of surgery, in which of need prevention from infection. It may happen, in cornear ulcer and eye infections.

Tetracycline influences both, bacteriostatic and bactericidal mode of action opposed to the majority of aerobic and anaerobic, Gram-negative and Gram-positive bacteria.

Pharmaceutical science treaty with the pinpointing, selection, presentation and standardization of various drugs. It is very crucial to model Pharmaceutical products that unremittingly deliver the purposeful performance, which needs to keep under observation of their quality constantly. Quality of drug takes part a prerequisite role which indicates the suitability of drug product for its important use. To examine the structure and analysis of certain compounds which are active in biological and Pharmaceutical, few spectroscopic approaches have been generally used.

The interactivity between Human Holo Transferrin and antibacterial drugs can be determined by several methods such as Fourier Transformation Infrared (FTIR) Spectroscopy, Raman Spectroscopy, etc. To understand the various functional groups and highly polar bonds of both pure HHT and antibacterial drugs and their chemical reactions, FTIR investigation was carried out. However, their foundation structure and symmetric bonds have been examined by Raman spectroscopy. Although, it is familiar that Raman and FTIR are interrelated vibrational spectroscopic methods, and some band intensity differences are there in between that two methods. That's why, both of the methods i.e. FTIR and Raman analyses were carried out.

II. Experimental:

2.1. Materials and Methods:

All chemicals (Human Holo Transferrin, Ciprofloxacin and Tetracycline) and solvents were reagent grade and were used as purchased without additional purification.

2.2. Fourier Transform Infrared Spectroscopy:

FTIR analysis was carried out by FTIR Spectrosphotometer accommodate with infrared (IR) microscope worked in reflection mode. The microscope is provided with a video camera, MCT detector (a liquid Nitrogen – cooled Mercury cadmium Telluride) and a translation stage which is controlled by computer and this computer is programmed in x and y directions. The collected spectra were in the region of 650 cm^{-1} to 4000 cm^{-1} its resolution is 4 cm^{-1} (Silverstein R.M and F.X.Webster, 2002, Dani V.R., 1995). The FTIR spectra in the present research work was recorded using a AGILENT CARY 630.

2.3. Raman Spectroscopic analysis:

FT Raman spectra were recorded using 1064 nm line of Nd:YAG laser working at 100 mw on BRUKER RFS 27 spectrometer in the range 4000-50 cm^{-1}, respectively at sophisticated Analytical Instrumentation Facility (SAIF), IIT Chennai, India.

III. Results And Discussion:

3.1. Vibrational spectral Analysis:

The Infrared spectra were registered on Fourier Transform spectrometer in the mid-infrared region (MIR) and the range was (400-4500 cm^{-1}). Because of the complex interaction of atoms within the molecule, The functional groups of IR absorption may vary over a wide range. However, it has been seen that many functional group supply with characteristic IR absorption at specific narrow frequency range. Multiple functional group may absorb at one particular frequency range but a functional group often gives rise to several characteristic absorptions. Thus, the spectral elucidations should not be cramped to one or two bands only, actually the entire spectrum should be examined.

While the FTIR bands at 4000 – 1300 cm^{-1} represented functional group region, the arrival of strong absorption bands in the region of 4000 – 2500 cm^{-1} was due to stretching vibration of hydrogen and more atoms with a mass of 19 or less. The O–H and N–H stretching frequencies were in the 3700 to 2500 cm^{-1} region with various intensities. Hydrogen bonding has a notable impact on the peak shape and intensities, usually causing peak broadening and shift in absorption to lower frequencies. The C–H stretching vibration obtained in the region of 3300 to 2800 cm^{-1} (Silverstein R.M. 2002, and Dani V.R., 1995).

FTIR spectra of protein reveal a number of amide bands, amide I band at 1600 – 1700 cm^{-1} (mainly C=O stretching and amide II band at 1500 - 1600 cm^{-1} (C – N stretching coupled with N –H bending modes), respectively (Fig.1). These FTIR distinguished peak are corrected with structural changes in proteins.

Additionally, the amide I band is the most immensively employed in the research of protein conformation as it is more reactive to the alternations in protein environment than amide II. It also should be, mentioned that its sub-peaks details are more developed than that of amide II.

Usually, the absorption band observed at nearly 1610-1632 cm^{-1}, 1632-1640 cm^{-1}, 1644-1662 cm^{-1}, 1670-1680 cm^{-1}, and 160-1700 cm^{-1} in the amide I region are assigned to β-sheet, random cool, α-helix, turn and β-antiparallel structures, respectively (Ahmed A. et al., 1995)

In FTIR spectra of HHT and Ciprofloxacin complex, one notable characteristic peak was found between 3500 and 3450 cm^{-1} which was attributed to stretching vibration of OH groups and intermolecular hydrogen bonding (Fig.2). one more band at 3000-2950 cm^{-1} characterized alkene and aromatic C–H stretching. The 1956 to 1450 cm^{-1} region revealed FTIR absorption from a large variety of double-bonded functional groups. The band at 1750 to 1700 cm^{-1} revealed the carbonyl C=O stretching. The peak between 1650 and 1600 cm^{-1} was allocated to quinolones. A strong absorption peak between 1050 and 1000 cm^{-1} was allocated to C – F group (Table.1).

In FTIR spectra of HHT and Tetracycline complex, vibrational peaks at 3342 – 3325 cm^{-1} that were ascribed to N-H and O-H stretching vibrational peaks at 3064 – 3003 cm^{-1} and 2955 – 2835 cm^{-1} were ascribed in C-H and CH_3 (methyl) stretching respectively, C=C stretching peaks were shown at 1622-1569 cm^{-1} Bending vibrational peaks for C-H and CH groups were appeared at 1454 and 1357 cm^{-1}, respectively. Vibrational peaks at 1247-1000 cm^{-1} and 995 cm^{-1} were assigned to C-H in plane deformation and C-N stretching respectively. The out plane ring deformation peaks were shown at 567 – 501 cm^{-1} (Fig.3).
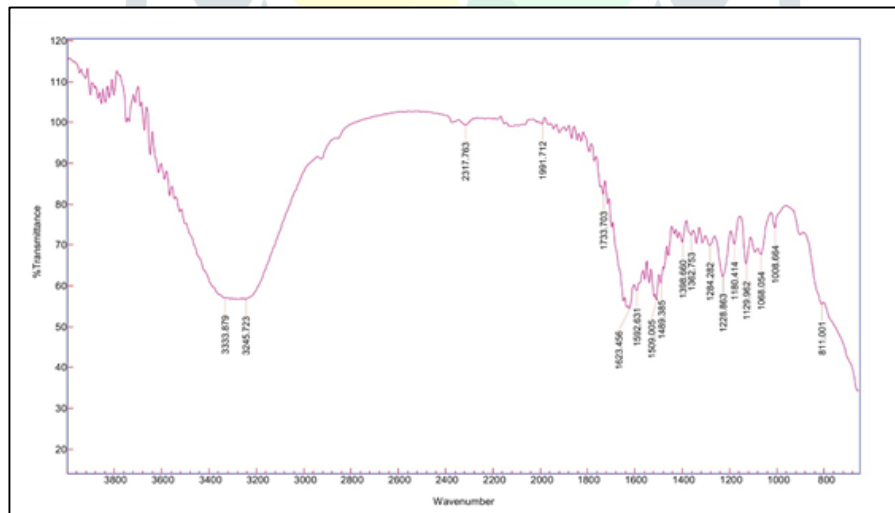

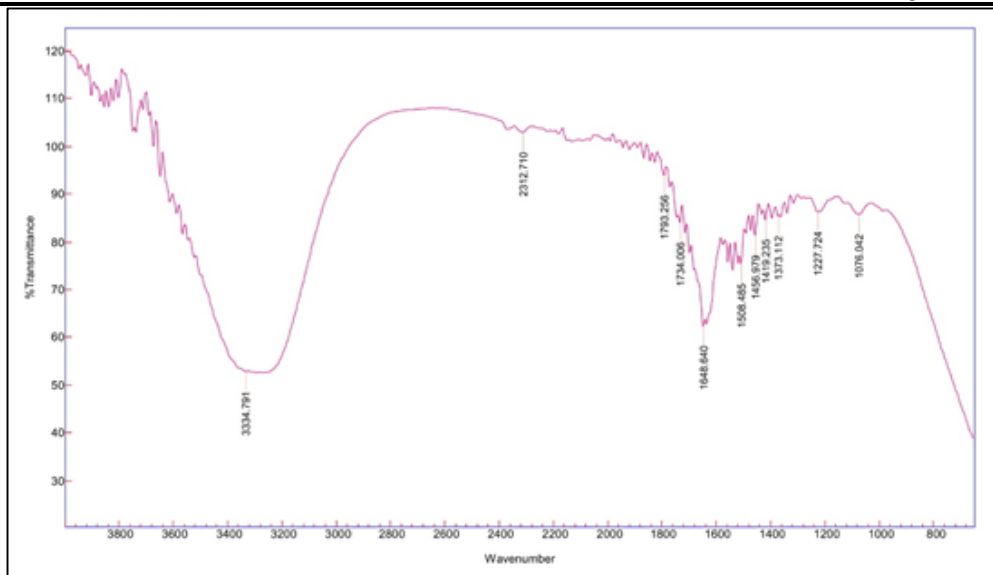Figure.1: FTIR Spectra of Human holo transferrin.

Figure.2: FTIR Spectra of Human holo transferrin + Ciprofloxacin.
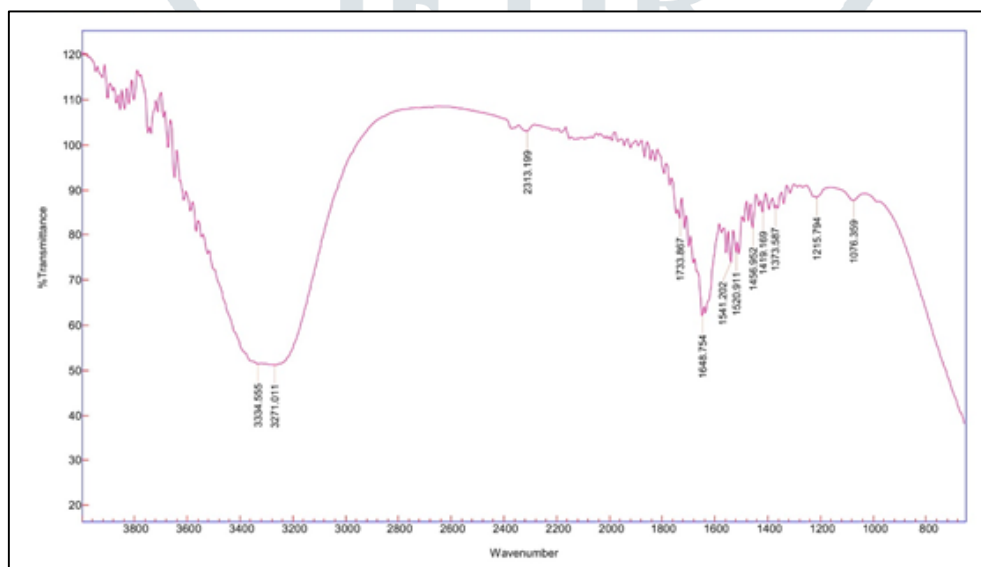

Figure.3: FTIR Spectra of Human holo transferrin + Tetracycline.

Table 1 FTIR and RAMAN peak assignments of Human holo transferrin (HHT), HHT+ Ciprofloxacin (CIP) and HHT + Tetracycline (TEC) complex

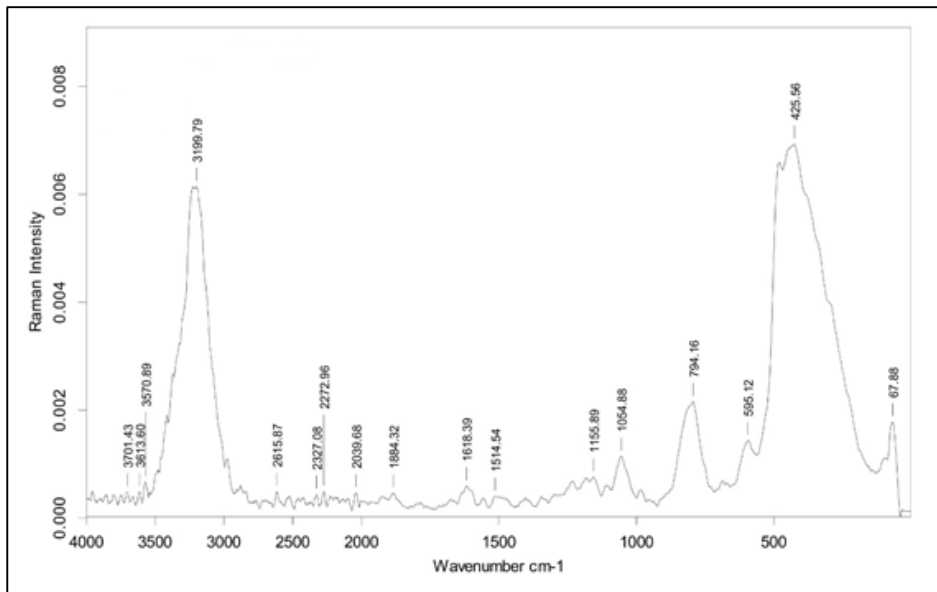| HHT (cm^{-1}) FTIR	HHT (cm^{-1}) RAMAN	Peak Assignment	HHT+CIP (cm^{-1}) FTIR	HHT+CIP (cm^{-1}) RAMAN	Peak Assignment	HHT+TEC (cm^{-1}) FTIR	HHT+TEC (cm^{-1}) RAMAN	Peak Assignment
	3613.6	O-H-O Frequency Of Hydrogen Bond				3334.5		O-H Stretching
	3570.8	O-H Stretching						
3333.8		C-H Stretching	3334.7		O-H Stretching	3271.0		Liquid aliphatic primary amines
3245.7		O-H Stretching	2312.1		Alkyl & Aryl Phosphines	2313.1		Alkyl & Aryl
	3199.7	O-H Stretching		2287.5		1733.8		Aliphatic aldehydes & C=O Stretching
	2615.8	O-H In Plane Mode Stretching		1919.1			1619.0	C=O in plane mode stretching
2317.7	2327.0	2-Carbonyl And Ammonium	1793.2		C=O Stretching	1648.7		C=O Stretching
1991.7		Cumulated Double Bond	1734.0					
1733.7		C=O Stretching	1648.6			1541.1		C=O Stretching, Aromatics & Heteroaromatics
				1591.5	Ring C-C Stretching Vibration	1520.9		C=O Stretching
1623.4	1618.3	C=O Stretching	1508.4		C=O Stretching, Aromatics & Heteroaromatics	1456.9		Aromatics & Heteroaromatics
1592.6		C=O Stretching	1456.9		Aromatics & Heteroaromatics	1419.1		Aromatics & Heteroaromatics
				1477.1	Ring C-C Stretching Vibration	1373.5		Aromatics & Heteroaromatics
1509.0	1514.0	Asymmetrical And Symmetrical NH3 Bending	1419.2		Aromatics & Heteroaromatics	1215.7		Hydrocarbons
1489.3		Strong Symmetrical Bending	1373.1		Aromatics & Heteroaromatics		1062.6	C-O-C symmetric stretching
1398.6		Symmetrical Stretching	1227.7		C-N Stretching	1076.5		C-N Stretching
1362.7		Sulfonamides	1076.0		C-N Stretching		792.4	N-H out of plane wagging and C=O Out of plane ring deformation
				1057.1	C-O-C Symmetric Stretching		72.0	Lattice vibration
1284.2		C-O Stretching Weak &C-H Bending Vibration		792.9	C-H Out Of Plane Bending			
1228.8		C-O Stretching Weak &C-H Bending Vibration		597.1				
1180.4		C-O Stretching		483.8	Strong Bending C-C Aliphatic Chain			
1129.9		C-O Stretching		424.5				
	1155.0	C-C-H Symmetrical Plane Bending		69.5	Lattice Vibration			
	1054.8	C-H In Plane Deformation						
1068.0		Symmetrical Stretching						
1008.0		Monofluoro-Alkane						
811.0		Asymmetrical Ring Stretching						
	67.8	Lattice Vibrations						

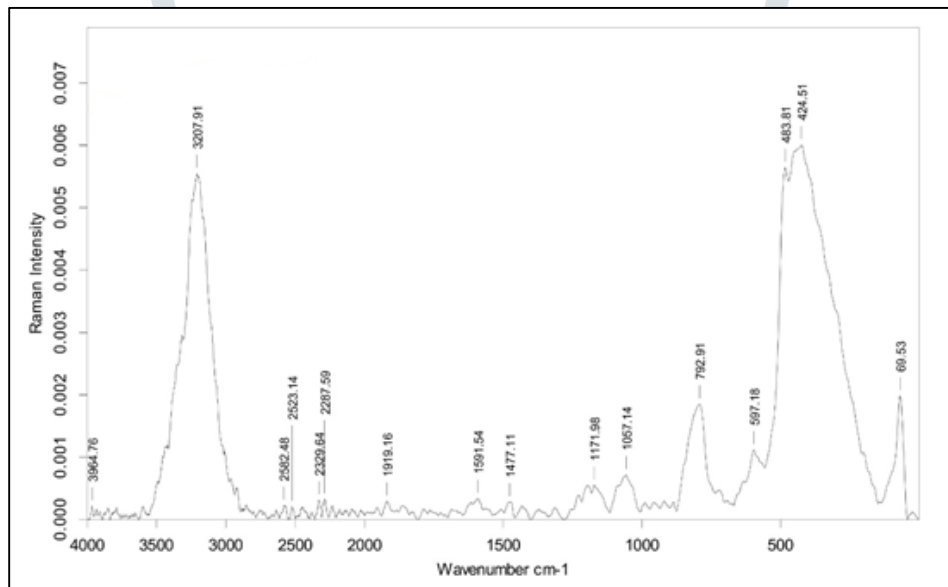Figure.4: Raman Spectra of Human holo transferrin

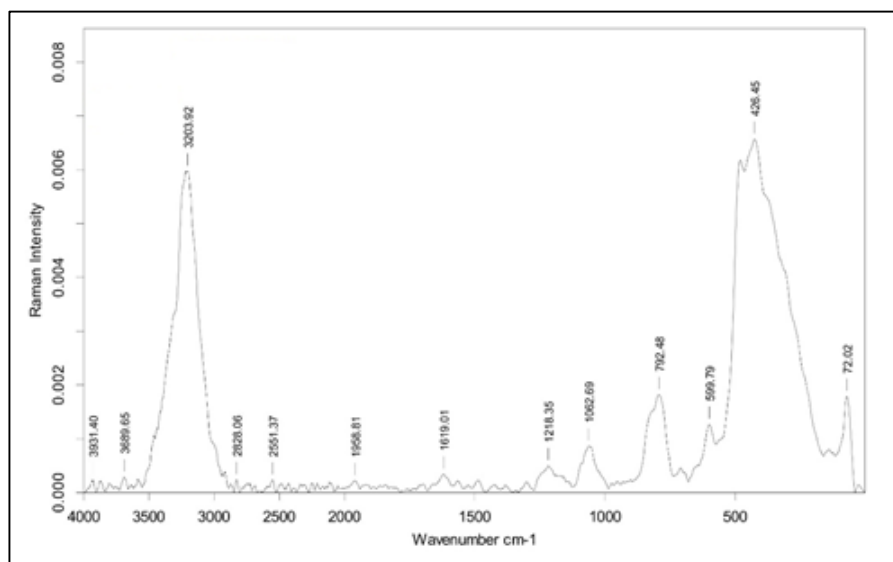Figure.5: Raman Spectra of Human holo transferrin + Ciprofloxacin.

Figure.6: Raman Spectra of Human holo transferrin + Tetracycline.

The observed prominent Raman shifts were given in (Figs. 4,5 & 6). The Raman shifts at 484.22 cm^{-1} show strong bending vibration of C – C of the aliphatic chain of cyclopropyl group and C – N stretching vibration of piperazinyl group (Xu J et al., 1997). While the band at 771.47 cm^{-1} revealed the symmetric stretching vibration of C – F group (CM Sharts and V.S. Gorelik 2002), the peak at 1411.63 cm^{-1} was because of symmetric stretching vibration of O – C – O group of carboxylic acid and methylene deformation mode of the piperazinyl group (Bright A. et al., 2010). A band at 1655.11 cm^{-1} (table.1) was for symmetric stretching of the carbonyl group of the pyridone moiety (Skoulika S.G and C.A.Georgiou 2001).
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IV. Conclusion:

FTIR and FT- Raman spectroscopic methods were employed further qualitative analysis of the antibacterial drugs Ciprofloxacin and Tetracycline with Human Holo Transferrin. An acceptable vibrational assignment of the drugs has been done from the FTIR and FT-Raman spectra or the drugs. They confirm the fundamental functional group present in the compound.

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