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A green approach for quantification of Naproxen in bulk and oral dosage form by FT-IR method

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ABSTRACT

A very simple, non-destructive, inexpensive and green strategy applied for the quantification of Naproxen using transmission Fourier Transform Infrared (FTIR) spectroscopy in bulk & tablet dosage form and the Beer's concentration range found to be 5-25µg. For the present investigation of Naproxen, O-H, 3000 - 3500cm^{-1} was selected for the analysis. The correlation coefficient for the method found to be 0.9965 and the developed method analyzed for specificity, limit of detection (LOD), limit of quantification (LOQ), linearity of response, precision and accuracy. This work clearly shows the capability of transmission FTIR spectroscopy for assessment of exact quantity of API to control the quality of finished products as well as during processing in pharmaceutical industries. Therefore, as compared to other spectroscopic or chromatographic method, costly chemicals and toxic solvents totally avoided in this direct, inexpensive and green approach. Such types of FTIR applications have a strong potential to replace classical methods in quality assurance /quality control (QA/QC) for the analysis of active contents in pharmaceutical preparations.

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INTRODUCTION

In recent decade, it is very essential for the quality control laboratories to ensure the quality, purity and potency of the new drugs or combination of new drugs are coming in to the market, it is happening only with the different analytical techniques. The techniques like HPLC, GC, HPTLC, UV, Colorimetric, FT-IR are effective methods for qualitative and quantitative analysis in the industries. From the literature review, UV, (Dharmalingam S *et al.,* 2013; Vinay Wamorkar *et al.,* 2011) HPLC (B. Tsvetkova., 2015; Vasundhara H *et al.,* 2013; Manidipa Debnath *et al.,* 2015) and LC/MS (NK Sahoo *et al.,* 2014) analytical methods are available for Naproxen, but there is no quantification study on FT-IR method. So, the present investigation is to develop a method based on FT-IR and its validation as per regulatory requirements.

Naproxen [2-(6-methoxy-2-naphthyl) propionic acid] is a non-steroidal anti-inflammatory drug (NSAID) often preferred to acetylsalicylic acid for the activities like anti-inflammatory, analgesic and antipyretic, because of its better bioavailability through oral administration and fewer adverse effects. Its anti-inflammatory activity generally thought to be inhibition of cyclo-oxygenase and by decrease in the levels of prostaglandin in different fluids and tissues. (John M. Beale *et al.,* 2002; Roberts Z *et al.,* 2001) Mostly Naproxen is formulated as tablet form, widely applied in the treatment of rheumatoid arthritis and other rheumatic or musculoskeletal disorders and acute gout. (Bansal V *et al.,* 2001).

KBr Pellet Method

Potassium bromide, it is an alkali halide when subjected to pressure forms a plastic like sheet that is transparent in the IR region. Potassium bromide (KBr) is the widely used alkali halide for the preparation of pellets. Before forming KBr powder as pellets, it is pulverized in 200 mesh, dried at 110°C for 2 hours. Rapid heating may cause oxidization of KBr in to brown coloration of Potassium bromate ($KBrO_3$). Then dried powder is stored in a desiccator and used immediately to prepare the pellets.

MATERIALS AND METHODS

All the chemicals used throughout the experiment were of highest purity of IR grade. Potassium bromide (KBr), Naproxen (Bulk Material) was obtained as a gift sample from a reputed company. Naproxen tablets (Dosage form) was purchased from local market.

Instrumentation

All spectral measurements were made on Bruker instrument (model no: MB 3000) with KBr press (model no: M 15).

Selection of Wavenumber

The selection of wavenumber is the important parameter for the Quantitative estimation by FT-IR method. For the present investigation of Naproxen, O-H, 3000 - 3500cm^{-1} was selected for the analysis.

Preparation of stock solutions of Naproxen

Initially 100mg of Naproxen weighed, and it is grinded with 100mg of Potassium Bromide in a mortar until uniform and fine powder was obtained. So, that each pellet prepared will contains uniformly distributed drug. By this mixing it will give 1mg/1mg of KBr i.e., 1000µg of naproxen (Primary stock).

Take 10mg of above primary stock into a mortar and it is grinded with 90mg of Potassium Bromide until uniform and fine powder was obtained. By this mixing it will give 100µg concentration of Naproxen (Secondary stock). Finally working samples are prepared by mixing 5, 10, 15, 20 & 25mg of secondary stock with KBr 95, 90, 85, 80, 75 mg respectively to produce 100mg final weight of pellets having 5, 10, 15, 20 & 25µg concentrations.

Procedure for FT-IR Spectroscopic method

The working samples prepared are made into a pellets (100mg) by KBr Pressed pellet technique. These pellets (5-25µg) were quantitatively analyzed in FT-IR absorbance mode at the wavenumber of O-H 3000 - 3500cm^{-1}.

Preparation of sample solution

10 tablets of Naproxen (Naprosyn 500mg) were triturated after taking their average weight. The tablet powder equivalent to 1 tablet was transferred to the volumetric flask and dissolved in chloroform. The supernatant liquid was passed through Watt Mann filter paper no. 41. The filtrate was evaporated, the residue obtained 10mg was accurately weighed, made up to 100mg with dried KBr and triturated well. Then the dilution is further performed from this stock mixture to prepare the pellet of desired concentrations as mentioned in the previous section.

Method Validation

The developed method was validated with the parameters like specificity, linearity, precision, LOD, LOQ and accuracy as per the regulatory requirements for validation of analytical procedures.

RESULTS

A Simple and sensitive Fourier Transform Infrared (FT-IR) spectrophotometric method have been developed for the estimation of naproxen in raw material and in tablet dosage form and the Beer's concentration range was found to be 5-25µg. The method is based in the measurement of absorption of radiation at absorption band of Naproxen O-H at 3000 - 3500cm^{-1}. The proposed method was validated as per regulatory requirements.

Specificity

Specificity of the Naproxen tablet formulation was studied for the examination of various excipients, the results revealed that they did not interfere in the assay method.

Linearity

The linearity of the Naproxen is assessed by linear regression method with the help of calibration curve method by taking the concentration range 5-25µg of Naproxen. The calibration curve was constructed by taking concentration of Naproxen on X-axis and absorbance values on Y – axis. The results obtained for linearity was shown in Figure 1 & 2 and Table 1.

Precision

The intra-day and inter-day precision of the method was checked by repeated scanning and measurement of the absorbance at 3000 – 3500 cm^{-1} (n = 5) of 10 µg of Naproxen per mg of KBr without changing the parameters. The repeatability and inter-day precision was expressed in terms of relative standard deviation (RSD) and reported in Table 2.

Accuracy

To validate the accuracy of the test method, recovery experiments were conducted at 80, 100 and 120% concentrations. Each test preparation was

Table 1: Summarized data of absorbance and linearity of Naproxen

S. No.	Concentration (µg)	Absorbance
1.	5	0.194
2.	10	0.412
3.	15	0.601
4.	20	0.863
5.	25	1.029
6.	Slope	0.0424
7.	Y-Intercept	0.0165
8.	Correlation coefficient	0.9965

Table 2: Intra & Inter day Precision of Naproxen

Concentration in µg	Intra-day Precision (Absorbance)			Inter-day Precision (Absorbance)		
	Morning	Afternoon	Evening	Day-1	Day-2	Day-3
10	0.413	0.415	0.418	0.413	0.420	0.416
10	0.412	0.414	0.415	0.412	0.418	0.414
10	0.414	0.417	0.417	0.414	0.415	0.417
10	0.414	0.415	0.418	0.414	0.416	0.412
10	0.412	0.413	0.420	0.412	0.411	0.410
Mean	0.413	0.4148	0.4176	0.413	0.416	0.413
SD	0.0010	0.0015	0.0018	0.0010	0.0034	0.0029
% RSD	0.24	0.36	0.44	0.24	0.82	0.69

Table 3: Accuracy Data of Naproxen

% Spiking level of API	Sample Amount added*	Total Amount of Spiking level*	Absorbance	Amount recovered from Spiking level	Amount found*	Recovered Amount Found*	% recovery
80%	10	18	0.716	18.04	9.9882	8.0518	100.65
80%	10	18	0.719	18.07	9.9882	8.0818	101.02
80%	10	18	0.712	17.98	9.9882	7.9918	99.90
100%	10	20	0.867	20.07	9.9882	10.0818	100.82
100%	10	20	0.864	20.10	9.9882	10.1118	101.12
100%	10	20	0.862	19.96	9.9882	9.9718	99.72
120%	10	22	0.953	22.11	9.9882	12.1218	101.01
120%	10	22	0.952	21.96	9.9882	11.9718	99.76
120%	10	22	0.955	22.07	9.9882	12.0818	100.68

* Results are in µg/mL

Table 4: Tablet formulation analysis result of Naproxen by FT-IR

Brand Name	Labeled amount (mg/tablet)	Absorbance	Estimated amount (mg/tablet)	% Amount	Mean	SD	% RSD
Naproxen	10	0.44	9.98821	99.88	99.49	1.801	1.81
	10	0.43	9.75236	97.52			
	10	0.445	10.10613	101.06			

Table 5: Summary of Results for FT-IR method of Tetrabenazine

Parameter	FT-IR Method
Wave number (cm^{-1})	3000 - 3500
Beer's law range (µg/mg)	5-25
Molar absorptivity	0.04175
Correlation coefficient, r^2	0.9965
Slope (b)	0.0424
Intercept	0.0165
Precision (% RSD)	< 2
Accuracy	99.72 % to 101.12 %
LOD	0.07783 µg
LOQ	0.235849 µg
Assay	99.49%

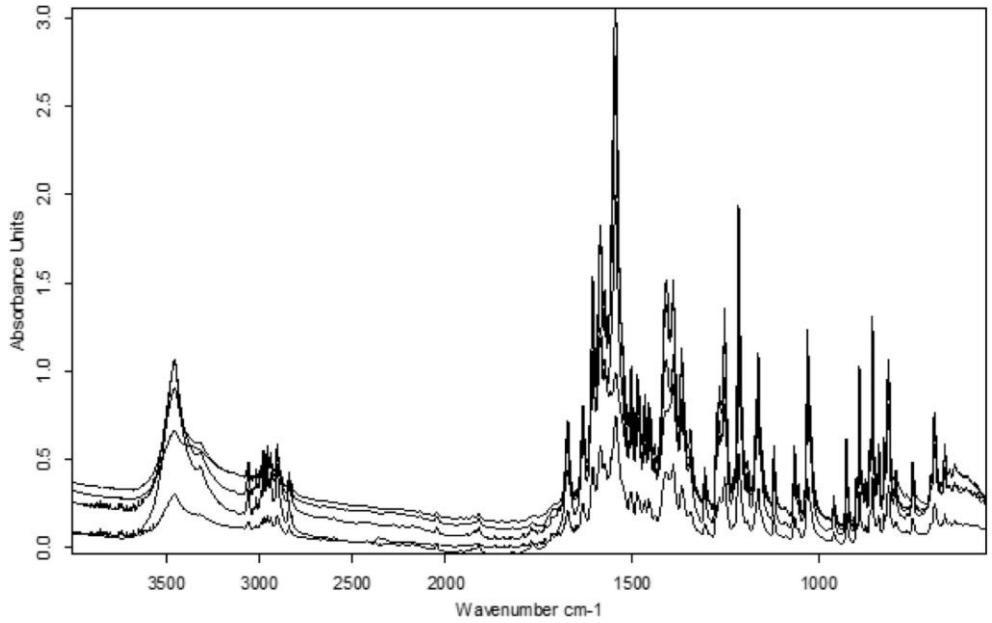

Figure 1: FT-IR overlay Spectra of Naproxen (5-25 µg)

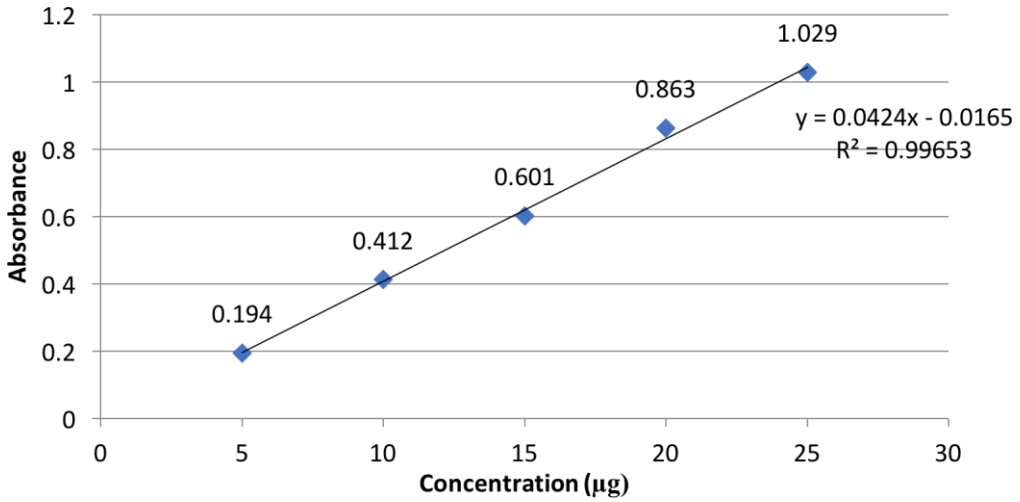

Figure 2: FT-IR Calibration Plot (linearity) of Naproxen (5-25 µg)

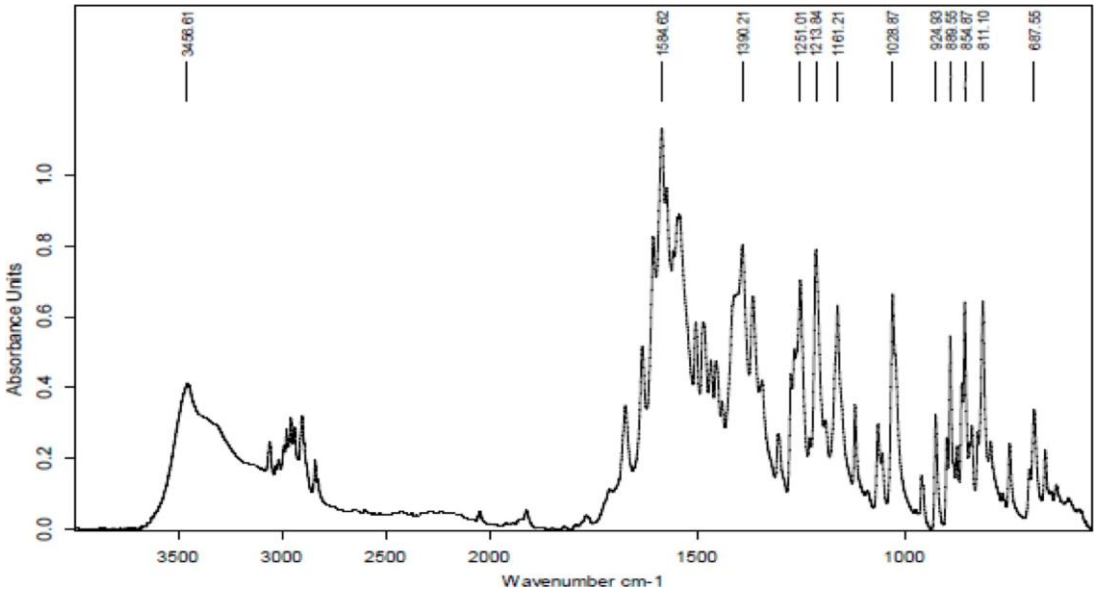

Figure 3: FT-IR tablet formulation assay spectra of Naproxen

prepared in triplicate and the analysis was performed in triplicate. The assay value at the beginning of validation was considered as the true value (100%) for recovery calculations. From the analyzed data, % assay and % recovery were calculated and reported in Table 3.

Limit of Detection and Limit of Quantification

The LOD and LOQ of Naproxen calculated by using standard deviation method LOD = 3.3 SD / Slope and LOQ = 10 SD / Slope and are 0.07783 and 0.235849 respectively.

Assay of Naproxen

10 tablets of Naproxen were accurately weighed, triturated well and a weight equivalent to 10 mg of Naproxen was weighed, diluted to 100mg with potassium bromide. It was mixed properly until obtaining a homogeneous powder. Then this powder mixture was crushed in a mechanical die press to form translucent pellet. Dilutions with potassium bromide were made to give final concentration 10µg. The analysis was carried out using the sample which were analyzed in three replicates represented in Table 4 and Figure 3.

DISCUSSION

Naproxen obeys Beer's law in the concentration range of 5-25µg, it showed good linearity as indicated by correlation coefficient value 0.9965 and the recovery studies indicates that there is no interference of other ingredients present in the formulation. The percentage recovered was found in the range of 99.72 – 101.12% and the optical characteristics molar absorptivity, the regression characteristics like slope, intercept, precision, LOD and LOQ were calculated and the results were summarized in Table 5.

CONCLUSION

The present study reported simple, rapid and green methods for the analysis of Naproxen and its tablet dosage formulation. Proposed methods are suitable for QC process in pharma industry. Therefore, as compared to other spectroscopic or chromatographic method, this is a direct, inexpensive and green approach in which costly chemicals and toxic solvents are totally avoided. Such types of FT-IR applications have a strong potential to replace classical methods in quality assurance / quality control (QA/QC) for the analysis of active contents in pharmaceutical formulations.

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