International Journal of Pharmaceutical Research and Applications
Volume 7, Issue 4 July-Aug 2022, pp: 1133-1140 www.ijprajournal.com ISSN: 2456-4494

Applications of GC-FTIR in Pharmaceutical Analysis: Review

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Submitted: 25-07-2022 Accepted: 05-08-2022

ABSTRACT:
A hyphenated technique is combination or coupling of two different analytical techniques with the help of proper interface. Mainly chromatographic techniques are combined with spectroscopic techniques. The term hyphenated techniques refer to separation, identification & better analysis of component.GC-FTIR (gas chromatography/Fourier transform infrared spectroscopy) is a combined technology in which the GC stage separates the samples and the IR stage identifies them. For increased chromatographic efficiency, a capillary GC column is frequently employed, and the IR stage typically incorporates an interferometric (Fourier transform or FT) spectrometer. Although GC-FTIR is typically used to provide qualitative information about various types of analytes, quantitative results can also be obtained. The aim of this article is to present overview of Gas Chromatography & Fourier Transform Infrared Spectroscopy (GC-FTIR) and its application in the analysis of various pharmaceutical, phytopharmaceutical including flavourand fragrances,fatty acids, perfumes and pesticide.
KEY WORDS: GC-FTIR, Hyphenated techniques, Pharmaceutical analysis

I. INTRODUCTION:

A hyphenated technique is the combining of two distinct analytical techniques with the use of an appropriate interface. Most frequently, chromatographic and spectroscopic techniques are combined. [5] Hyphenated procedures include separation-separation, separation-identification, and identification-identification strategies. The phrase "hyphenation" first appeared Hirschfeld modified it in 1980 to describe a combination of two or more possibilities a single run of analytical techniques (Hirschfeld, 1980). Obviously, the purpose of this coupling is to obtain a detection with a lot of information for both compared to that, identification and quantification using just one analytical method. According to the detection mechanism information-rich detectors can be divided into three categories: detection based on molecular mass spectrometry: b) detection based on molecular spectroscopy, such as Fourier-transform infrared (FTIR) and nuclear magnetic resonance (NMR) spectroscopy; and c) detection based on atomic spectroscopy (elemental analysis) by coupling with such as inductively coupled plasma (ICP)-MS, atomic absorption spectroscopy (AAS) and atomic emission spectroscopy (AES), respectively [1,8]

II. TYPES OF HYPENATED TECHNIQUES:

1. Double hyphenated techniques.
2. Triple hyphenated techniques.
1. Double hyphenated techniques
- GC-IR
- LC-NMR
- LC-IR
- LC-MS
- CE-MS
- GC-MS
- HPLC-DAD
- GC-FTIR

2. Triple hyphenated techniques
- LC-API-MS
- APCI-MS-MS
- ESI-MS-MS
- LVI-GC-MS
- LC-ESI-MS
- LC-UV-NMR-MS-ESI
- LC-MS-TSPLC-UV-NMR-MS
- LC-NMR-MS
- LC-DAD-API-MS
- LC-PDA-MS

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Volume 7, Issue 4 July-Aug 2022, pp: 1133-1140 www.ijprajournal.com ISSN: 2456-4494

- LC-PDA-NMR-MS
- SPE-LC-MS[10,5]

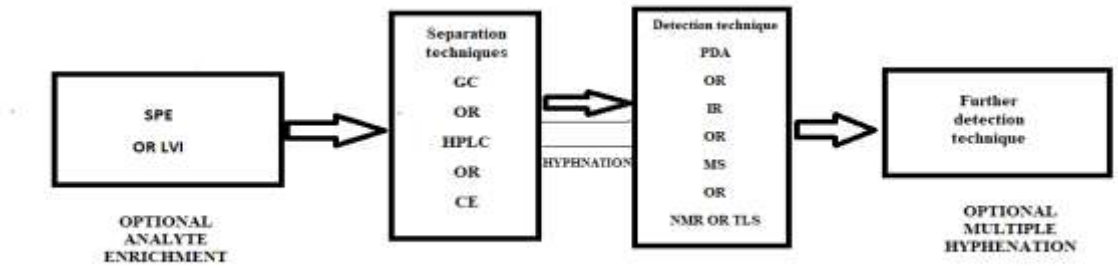

Figure1: Schematic presentation of hyphenation of chromatographic and spectrometric techniques [1]

III. GC-FTIR SPECTROSCOPY:

GC-FTIR (gas chromatography/Fourier transform infrared spectroscopy) is a combined technology in which the GC stage separates the samples and the IR stage identifies them. For increased chromatographic efficiency, a capillary GC column is frequently employed, and the IR stage typically incorporates an interferometric (Fourier transform or FT) spectrometer.[3]Although GC-FTIR is typically used to provide qualitative information about various types of analytes, quantitative results can also be obtained. [2]

Complex mixtures of volatile compounds can be separated using modern GC-FTIR apparatus, which also allows for the molecular characterization of the components.IR spectroscopy can identify their functional groups. There is spectral library and search software available. Fraction might be recognized automatically. Apparatus forth is type provides fast, dependable outcomes without any comprehensive sample preparation in the past. It has previously been used in a number of fields. [6]

A potent method for identifying the constituents of complex mixtures, such as natural products, pesticide residues, environmental contaminants, and drug metabolites, is the combination of gas chromatography and FTIR. The most popular way for connecting a gas chromatograph to an FT-IR is the traditional light-pipe interface. [14, 15]

Instrumentation and Working:

1. Interface
1.1. Light pipe(LP)
1.2. Matrix Isolation Technique (MIT)
1.3. Direct Deposition Interface (DDI)
2. Detector

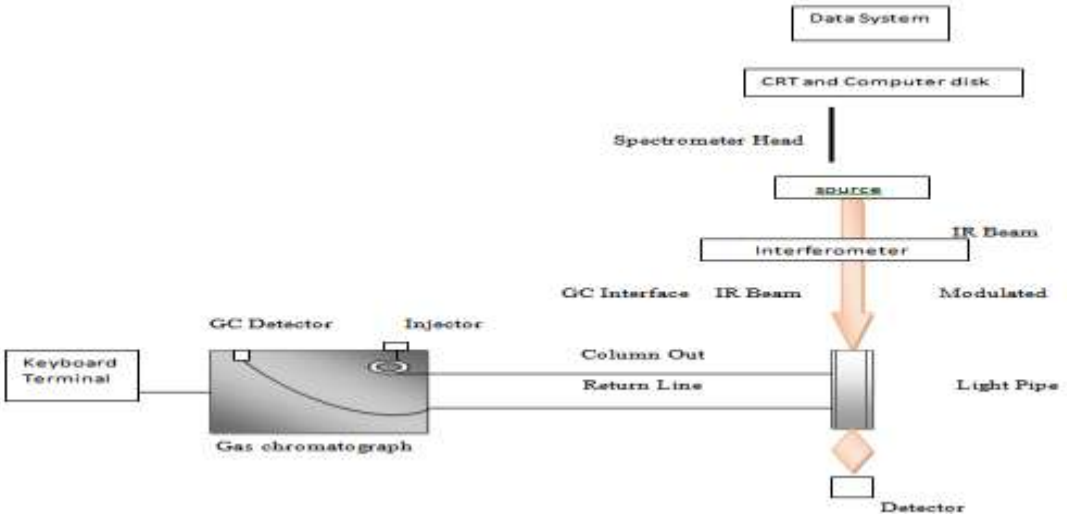

Figure 2:Block diagram of GC-FTIR system[3]

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LIGHT PIPE:

The LP is a gold-coated glass tube. The gold coating enhances the cell's overall optical throughput. By allowing the IR beam to reflect several times. This material is thought to be chemically inert, exceptionally robust, and stable, which is a significant benefit for a material that must be heated and must be chromatographically inert. The LP's high temperature, on the other hand, is an issue because as the temperature rises, the signal intensity drops dramatically. This phenomenon is caused by large amounts of unmodulated IR radiation released by the hot LP, which is an IR radiator in and of itself, radiation that saturates the detector orpreamplifier. The emission from the heated LP is eliminated by placing a cold barrier at the end of the LP. [9, 4]

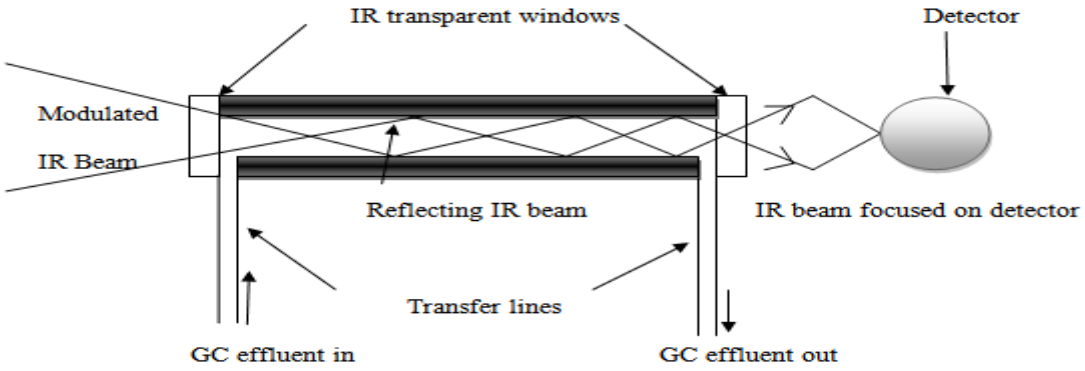

Figure 3: Schematic light pipe interface [4]

INTERFEROGRAM:

An FTIR apparatus used for connection to GC is essentially a single-beam FTIR spectrometer, with a rapid-scanning two-beam delivering the modulated IR beam. Generally an interferometer based on the original Michelson design.[19]The instrument known as the Michelson interferometer can use a to split an IR beam of energy into two pathways. The two beams within a beam-splitter, and then after a route difference, a single modulated beam moving mirror had introduced it. The consequently, the interferometer's interference pattern, the interferogram reflects the changes in intensity of these a function of route difference, beam and can be measured by an IR sensor. It is the cosine Fourier interferogram. Spectrum of the incident radiation transformed The IR spectrum is calculated by computing the cosine Fourier transform, which is mathematically reciprocal to the Fourier transform. Interferogram's transform every single rapid-scanning interferometer typically works by successive scans that average the signal. To average signals precisely, each interferogram needs to have a precise sampling rate. The same lag time between each scan. Therefore it is necessary to match the moveable mirror with extreme precision. to regulate the fluctuation of the optical path difference. [4]

DETECTOR:

Since the majority of typical GC detectors (flame ionisation, electron capture, mass spectroscopy, alkali flame, and others) have detection limits one to five orders of magnitude lower than FTIR, sensitivity is one of the main problems in the GC/FTIR experiment. Therefore, the mercury cadmium telluride (MCT) detector is the preferred detector. Compared to the typical triglycine sulphate (TGS) detector, this one delivers an increase in S/N of around a factor of 5 to 50, depending on the spectral range. [12]

WORKING:

In an on-line GC/FTIR coupling device, the effluent from the GC runs through a heated transfer-line, together with the makeup gas, into the light-pipe. The interferograms are scanned during a run. continually (more than one scan per second). This allows for real-time reconstruction of a chromatogram the Gram-Schmidt vector approach, which measures time approach, and a single beam

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IR Spectra is calculated. by the Fourier transformation. This is measured in comparison to a spectrum collected before the sample was put into the GC. After the actual run is complete, the spectra of each GC peak are matched, co-added, and separated by the piece of software.

The best IR spectra are produced when one GC peak's substance completely fills the light pipe. Therefore, it must be assured that during a very little period of time One of the peaks flows through the light pipe without overlaps with another This denotes the gas flow rate. through the GC column and the make-up gas pressure must be in good alignment with the light-pipe volume. In our case for a wall-coated capillary at a pressure of 1.6 bars before the column, a make-up gas It requires pressure between 0.8-0.9 bars.

The temperature of the light-pipe has a big impact on sensitivity. Heat the make-up gas and the transfer line at least to the final the GC temperature program's but the sensitivity of spectroscopy declines as temperature of the light-pipe hence it should be held as low as is practical. However, high-altitude condensation in any event, boiling fractions must be avoided. Increasing the temperature by roughly 100 K only yields half the sensitivity is determined by integrating the absorbance. The signal-to-noise ratios of the devices, like the sensitivity, Temperature causes spectra to narrow. Hence, both the spectra's quality and their assignment will be influenced. [6]

IV. APPLICATIONS:

1. ENVIRONMENTAL:

There have been significant efforts to use GC/IR for qualitative and quantitative environmental analysis of common environmental pollutants. [20, 21] The claimed benefits were specificity and selectivity, the ability of IR to distinguish between isomers, and the complementary nature of GC/FTIRS and GC/MS was quickly recognised. Environmental concerns drove several efforts to improve the performance of GC/FTIR interfaces, particularly the LP interface. For standards, soil samples, and a herbicide still-bottom, this technique finally produced minimum detectable levels in the 10–50 ng range. [22]For priority contaminants, the quantitative capabilities of GC/FTIRS were also well demonstrated [23]

2. FLAVOR AND FRAGRANCE:

The main use of GC/IR is for the identification of taste and aroma compounds. Its complimentary the characteristics of GC/IR for structural analysis were discovered quite early for volatile flavouring substances. focusing in particular on IR's special ability Spectroscopy can be used to tell between individual isomers.(24)Early writing up until the end of 1985 has been applications to the constitution of the aroma covered, tropical fruits, and the volatiles emitted from whole Coffea arabica fruits. The technique has proven to be particularly effective for the analysis of essential oils, especially for the detection of volatile research of the structures of terpenes and sesquiterpene where MS alone cannot clearly identify is very typically challenging, and when FTIR produces spectra with clarity Fingerprints. Recent investigations made use of the DD interface for greater sensitivity and specificity IR spectra corresponded to the spectra in the published literature. KBr is acquired as a liquid or pellets. The benefit of analysis of taste extracts using the MI method hasalso been demonstrate [4]

3. FATTY ACIDS:

To discriminate between Cis and Trans isomers, IR spectroscopy has long been employed as a dedicated technique. Additionally, to calculate the total amount of trans fatty acids in oil and fats. The infrared examination of each isomer in Unsaturated fatty acid complex combinations were created GC/FTIR technology combination makes this possible. The first case reported in the literature gave the analysis of isolated unsaturated cyclic fatty acid mixtures with the use of an LP interface, from heated oils.[25]This interface type was frequently used to define geometrical Various polyunsaturated fatty acid (PUFA) isomers applications.[26] However, it soon became clear that equipment sensitivity compatible with HRGC is higher to identify the small components discovered when small components, such as trans PUFA levels, are less than 1% in mixes with severely imbalanced dynamic ranges of concentration. The MI in this regard Mossoba et al. [27] used the interface successfully. Examples of typical spectra have already been shown exhibited above. It has been proven more recently; the DD interface is also ideally suited for the investigation of elaborate fatty acid combinations. [28]

4. PHEROMONES:

The pheromonal compound's hydroxyl group was validated by the IR spectrum the OH stretching frequency (3668 cm^{-1}) was in the secondary alcohols An additional stretching frequency (3605 cm^{-1})shown intra molecular hydrogen bonding, most likely resulting from a

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carbon-carbon unsaturation in the molecule interacting with This is a system with intra molecular hydrogen bonds .The hydroxyl group's position in relation to other molecules is fixed by a property that is specific to vapor-phase spectra, as already mentioned. A double bond between carbon and carbon, and is sovery instructive in terms of structure. A CH out-of-plane was blamed for the medium absorption at 960 cm^{-1}.A Tran's disubstituted ethylenic bond being deformed. [4]

5. PESTICIDE:

Our research on the chemistry of pesticides has benefited from the use of GC/FTIR as a tool to track long-term responses. For many sample preparations, the reaction must progress for several days or weeks before producing the desired chemicals. The GC/FTIR data of aliquots taken from the reaction material at regular intervals can be used to identify the location where the concentrations of the target products are at their optimum. One of the by-products of GC/FTIR monitoring of one of the preparations for the 8,10-dihydrogen mirex isomers was the discovery of a novel mirex derivative.[7]

6. QUANTITATIVE ANALYSIS OF CAFFEINE:

Caffeine concentrations are first calibrated using the ratio of caffeine to diphenylamine using real-time spectral window chromatograms. The peak area for caffeine is measured between 1650 and 1700 cm^{-1}, whereas the peak area for diphenylamine is between 1475 and 1525 cm^{-1}. The correlation coefficient is 0.997 for 6 concentrations ranging from 0.1556 to 5 µg/µl The absorbance spectra that were acquired while the two chemicals were being eluted have all been calculated a second time. Then, a further chromatographic trace is computed in accordance with the sharp spectra. With reference to real-time measurements, the correlation between the ratio of peak areas observed in the earlier spectral windows and the concentration is enhanced (r=0.9998)

With the aid of this quantitative methodology, we were able to determine the amount of caffeine present in both coffee and a sample of typical pee. The outcomes are:

URINE [caffeine] =0.1 µg/ml

COFFEE =13 µg/ml [caffeine].

These findings are consistent with the analytical information often provided by traditional chromatographic techniques. They show that, with some care taken in terms of separation and calibration, GC-FTIR is capable of being used for quantitative applications. [11]

7. COMPOSITIONAL STUDIES OF HIGH-TEMPERATURE COAL TAR:

A by-product of cooking processes, coal tar is a crucial raw material for both industrial and environmental purposes. Additional and more thorough investigations of coal tar's composition are desirable due to the substance's importance to the environment and the usage of coal tar fractions as chemical feedstock. Despite being widely used, coal tar has remained poorly understood its composition is unclear because different coals are used and there are varying cooking conditions. Over the years, it has drawn a lot of attention from researchers. Capillary GC/FTIR can be used to separate and identify substances and is typically more effective at identifying isomers that are closely linked to one another. However, the use of retention indices (RI) for GC effluent identification is becoming more widespread. As a result, identifying effluents by combining capillary GC/FTIR with RIs can completely utilise each technology's benefits and boost the accuracy of identification. The goal of the current work was to separate, identify, and quantify the composition of pounds in high-temperature coal tar. [13]

8. ANALYSIS OF PERFUMES:

Natural or synthetic perfumes are complex mixtures made up of a variety of substances. In addition to the GC-MS, the GC-FTIR is also utilised in the analysis of perfumes. The majority of recent investigations have been carried out using GC-VFIR systems that are based on traditional light pipes (LP).

A reference (synthetic) mixture containing 16 major and several minor ingredients was utilised to compare the performance of the LP and tracer of GC-FTIR systems. The mixture's constituents are substances that are frequently found in branded perfumes. Under the same chromatographic circumstances, the reference mixture's GC-FTIR spectra from an LP and a tracer system were obtained. The higher sensitivity level of the 'Tracer system is in fact demonstrated by a comparison of the two sets of data so produced. Additionally, the comparison reveals that some of the key elements picked up by the 'Tracer system wasn't present in the LP data. When these compounds come into touch with the hot gold surface, they decompose thermally, as can be seen by a closer inspection. For three samples of commercialized perfume, GC-FTIR measurements

International Journal of Pharmaceutical Research and Applications
Volume 7, Issue 4 July-Aug 2022, pp: 1133-1140 www.ijprajournal.com ISSN: 2456-4494

were collected. By comparing the spectra of these samples to a digitised spectral library made using the tracer data from the reference mixture, it was simple to identify the main components of these samples. [15]

9. ODOUR ANALYSIS OF ESSENTIAL OILS:

Based on GC-FTIR analysis of odour components in East Indian Sandalwood Oil (Santalum album L.) and Patchouli Oil (PogostemoncablinBenth), the compounds α-standalone, α-scintilla, β-scintilla, epic-β-scintilla, α-bergamot and spirosantalol in Sandalwood Oil and of patchoulol, α-guaiene, α-patchoulene, seychellene, α-bulnesene, norpatchoulenol and pogostol as the strongest odour components of patchouli oil.[16]

10. ANALYSIS OF UNKNOWN ILLICIT DRUGS:

Due to the combination of extremely effective separation and highly precise identification, the combination of high-resolution gas chromatography (HRGC) with Fourier Transform Infrared Spectroscopy (FTIR) offers a unique instrument to resolve this concern. Based on the retention behaviour of the analytes and the IR absorption bands, GC-FTIR enables fast identification of functional groups in unknown compounds. In order to structurally identify volatile and semi-volatile compounds, IR may well complement the information provided by mass spectrometry (MS) by relying on certain chemical features. Additionally, FTIR spectroscopy helps to overcome one of the limitations of MS detection, which is the inability to distinguish between Regio isomeric molecules, by measuring minor energy variations based on rotational and vibrational amplitudes between individual molecular bonds. [17]

11. ANALYSIS OF SPIRIT:

Spirits are distilled alcoholic drinks meant for human consumption, and the ethanol they contain must come from agricultural sources. They consist of wine, distillates made from pomaceous fruit and stone, roots, and plants that provide starch and sugar. Directives governing their production and labelling have been enacted by the EC (European Community)

The use of various raw materials changes a product's composition and determines its key attributes, while various fermentation and distillation procedures change its organoleptic qualities. The flavour is also impacted by variations in raw material origins, production methods, and manufacturer-dependent product requirements. Gas chromatography, which is useful for the beginning, intermediate, and final products' quality control, can be used to authenticate the identity of the majority of these products using compound-specific detectors like IR and MS. Any altered organoleptic impression, adulteration, or contamination brought on by foreign substances, substandard raw materials, or defective raw materials can be found using a straightforward qualitative comparison. [18]

V. CONCLUSIONS:

This review elucidate that GC-FTIR spectroscopic hyphenated technique applicable in analysis of different unknown drugs, organic compounds, pharmaceuticals and pesticides with reproducible result and rapid cost and time saving. This review on GC-FTIR hyphenated techniques is helps to analysis of spirit, coal tar, perfumes, odour analysis of essential oil, which help in understanding of available applications.

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