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ORIGINAL ARTICLE

Qualitative Assay of Selected Brands of Ibuprofen Tablets Obtained from Pharmaceutical Stores in Adamawa State

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ABSTRACT
This assay of selected brands of Ibuprofen tablets obtained from pharmaceutical stores in Adamawa state is a qualitative assay, where samples were collected and analyzed using IR Spectrophotometers. The results indicated that all the ten sample brands of Ibuprofen tablets analyzed confirmed the presence of Ibuprofen as indicated by the various FTIR spectra. The data were expressed in tables and figures, and the figures were presented in the form of absorption spectra, with corresponding peaks and bonds which were assigned to various functional groups present on the samples, which were mirror images of the absorption spectrum of the standard Ibuprofen powder. It was concluded that all the ten sample brands satisfied the Pharmacopoeia specification for Ibuprofen tablets of labeled strength 400mg/tablet and therefore, the study concluded that none of the sample brands of Ibuprofen tablets analyzed could be classified as substandard or counterfeit.
Keywords*: Ibuprofen quality, Substandard drugs, Qualitative assay, Adamawa State, Nigeria, and Public health.*

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INTRODUCTION

Ibuprofen, a nonsteroidal anti-inflammatory drug (NSAID), is a cornerstone of pain management and fever reduction globally [17]. Its analgesic, antipyretic, and anti-inflammatory properties make it a first-line medication for various conditions, including musculoskeletal pain, headache, dysmenorrhea, and fever [1]. Due to its effectiveness, affordability, and ease of access, ibuprofen consumption is widespread, particularly in resource-limited settings like Nigeria [2]. Adamawa State, located in northeastern Nigeria, faces challenges in ensuring consistent access to quality-assured medications [10]. The influx of counterfeit and substandard drugs poses a significant public health threat [3]. These drugs often lack the declared active pharmaceutical ingredient (API) or contain incorrect quantities, potentially leading to treatment failure, adverse effects, and antibiotic resistance [5, 6].

Qualitative assays play a crucial role in safeguarding public health by verifying the presence and concentration of the labeled API in a drug product ((European Medicines Agency [EMA], 2023). This research article aims to conduct a qualitative assay of selected brands of ibuprofen tablets obtained from pharmaceutical stores within Adamawa State, Nigeria.

Ibuprofen's mechanism of action hinges on its ability to inhibit cyclooxygenase (COX), an enzyme responsible for prostaglandin synthesis [7]. Prostaglandins are lipid mediators involved in inflammation, pain perception, and fever regulation [8]. By inhibiting COX, ibuprofen reduces prostaglandin production, thereby mitigating pain, inflammation, and fever [5].

The World Health Organization (WHO) recognizes ibuprofen as an essential medicine, highlighting its importance in primary healthcare [16]. Its inclusion on the WHO Model List of Essential Medicines underscores its effectiveness, safety, and affordability in managing a broad spectrum of conditions [17].

Despite ibuprofen's established efficacy and global availability, concerns regarding the quality of circulating drugs persist in many developing countries, including Nigeria. A 2020 study by Onah et al. investigating the quality of anti-malarial medicines in Nigeria identified a significant prevalence of substandard and falsified products [11]. This trend likely extends to other medications, including ibuprofen. The circulation of substandard and falsified ibuprofen poses a double jeopardy to public health

in Nigeria. First, patients may not receive the intended therapeutic effect, leading to delayed recovery or worsening of their condition [12]. Second, these products may contain contaminants or incorrect dosages, potentially triggering adverse effects and even drug resistance [9, 17].

Several factors contribute to the proliferation of substandard and falsified medicines in Nigeria. Weak regulatory frameworks, inadequate enforcement mechanisms, porous borders, and a lack of public awareness all play a role [1, 4]. This necessitates robust quality control measures to ensure the availability of genuine and efficacious ibuprofen for the Nigerian population.

Qualitative assays serve as a critical line of defense in safeguarding public health by verifying the identity and concentration of the labeled API in a drug product [3]. These assays employ various analytical techniques to detect the presence of the claimed API and quantify its amount within an acceptable range as stipulated by pharmacopeias.

Common qualitative assay methods for ibuprofen tablets include thin-layer chromatography (TLC), high-performance liquid chromatography (HPLC), and Fourier-transform infrared (FTIR) spectroscopy [2, 14-16]. Each technique offers advantages and limitations. TLC provides a rapid and inexpensive method for preliminary identification of the API [5]. HPLC offers superior separation and quantification capabilities, making it a preferred method for confirmatory analysis [3]. FTIR spectroscopy allows for structural characterization of the API, aiding in the detection of counterfeits [2].

This study assessed the presence of the Ibuprofen 400mg tablets in some selected brands of Ibuprofen 400mg tablet formulations commercially available in the Nigerian market obtained in some pharmaceutical stores in Adamawa state, by using IR Spectroscopy. The findings can inform regulatory bodies, healthcare professionals, and the public about the presence or absence of substandard and falsified ibuprofen products within the state.

MATERIAL AND METHOD

Equipment and Reagents

The apparatus and equipment used are analytical balance, 250-g capacity (Resolution 0.0001g) glass wares, pestle and mortar, Fourier transform infrared spectrophotometer (FTIR Buck SCI Model M530), water bath, and measuring cylinder, as well as Samples brands and standard Ibuprofen crystals. The equipment and apparatus used in this study were calibrated to check their status before and in the middle of the experiments. Apparatus such as volumetric flasks, measuring cylinders, and other glass/plastic wares were thoroughly washed with detergents and tap water and then rinsed with deionized water. All glassware was cleaned with 10% concentrated Nitric acid (HNO_3) to clear out any heavy metal on their surfaces and then rinsed with distilled-deionized water, the reagents used are KBr, HNO_3.

Samples and Sampling Method

The standard Ibuprofen crystals were obtained from the chemistry department at, the American University of Nigeria Yola, all other chemicals were of analytical grade, and deionized water was used in all experiments. Ten commercially available sample brands of Ibuprofen tablets each with a label claim of 400mg were randomly selected and purchased from the selected retail pharmaceutical stores, in Adamawa state, the samples were blindly labeled as Sample A (A), Sample B (B), Sample C (C), Sample D (D) and Sample E (E), Sample F (F), Sample G (G), Sample H (H), Sample I (I), Sample J (J) and used as samples.

Table 1: Label Information of Sample Brands of Ibuprofen Tablets

S/N	Sample Code	Label Claim (mg)	Expiry Date
1	A	400mg	October 2026
2	B	400mg	August 2024
3	C	400mg	August 2024
4	D	400mg	March 2025
5	E	400mg	June 2026
6	F	400mg	January 2026
7	G	400mg	January 2025
8	H	400mg	March 2026
9	I	400mg	June 2026
10	J	400mg	April 2026

Sample Preparation for Fourier Transform Infrared

The powder was pressed into a pellet, and KBr was selected as the diluent (matrixes) for the mid-IR frequency range [13], the KBr Pellet was prepared by accurately weighing 10 tablets of Ibuprofen tablets and crushed uniformly with the help of a mortar and pestle.

The powdered samples and KBr were ground to reduce the particle size to less than 5mm in diameter (Otherwise, large particles scatter the infrared beam and cause a sloping baseline of the spectrum.), a spatula full of KBr was added into an agate mortar and ground to fine powder until the crystallites were no longer seen and it becomes somewhat "pasty" and sticks to the mortar, a small amount of the powdered samples (about of 0.1-2% of the KBr amount, or just enough to cover the tip of a spatula) was taken and mixed with the KBr powder, subsequently, the mixture was ground for 5 minutes.
The die-set was assembled and the powder was added into the 7mm collar, the die was put together with the powder into the Quick Handy-Press, and the powder was pressed for 2 minutes and a pellet was formed (A good KBr pellet is thin and transparent. Opaque pellets give poor spectra, because little infrared beam passes through them, a white spot in a pellet indicates that the powder is not ground well enough, or is not dispersed properly in the pellets) [14]. The die set was disassembled and the 7mm collar was taken out, the collar was put together with the pellet into the sample holder, the die was cleaned after each experiment, and the process was repeated for both the standard lab sample as well as each of the ten sample brands of the Ibuprofen tablets.

Qualitative assay using Fourier Transform Infrared Spectrophotometer

The samples were scanned and analyzed using an FTIR Spectrophotometer, after placing the sample in the sample compartment, the sample's absorbance of infrared light was measured at various wave numbers between 4000 and 200cm^{-1} [13].
The samples vibrated at different rates of vibration which gave rise to a closely packed absorption spectrum, the presence of various bands on the spectrum is characteristic of the functional groups and bonds in the samples. Thus, the IR spectrum of the samples presented a fingerprint for its identification. The absorption peaks on the spectra were assigned to the various functional groups which fall within the peak range for the specific sample and the process was repeated for each of the ten brands of Ibuprofen tablets and the standard sample.

Statistical Data Analysis

The data was generated and the results were evaluated by charts and tables, which were used to make comparisons for qualitative assay of the sample brands of the Ibuprofen tablets, a probability of $P < 0.05$ was considered significant.

RESULTS AND DISCUSSION

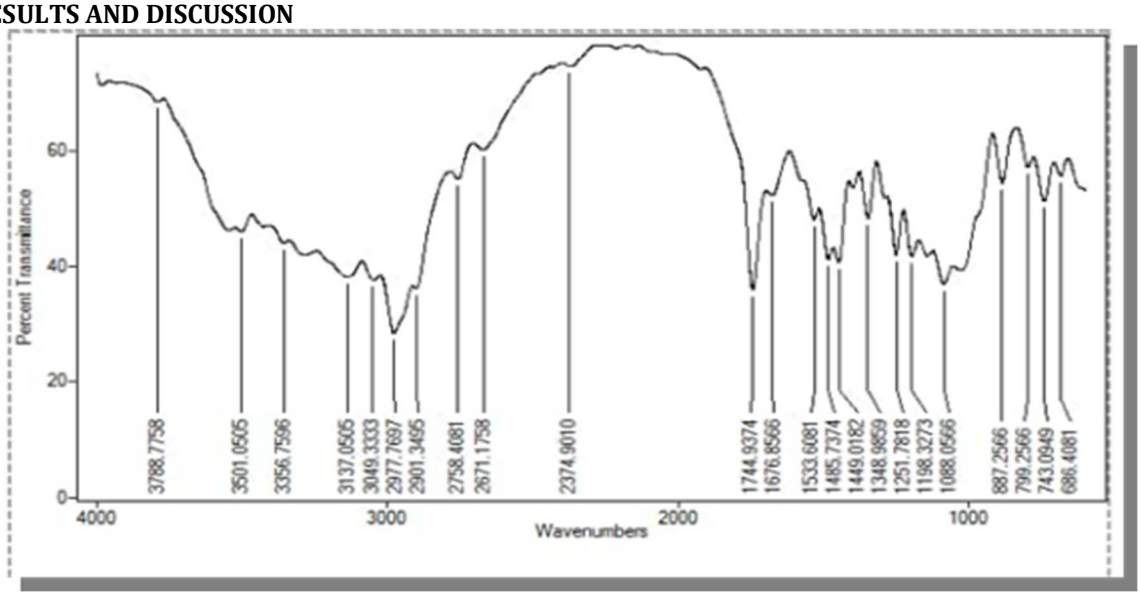

Figure 1. FTIR spectrum of the Standard Ibuprofen Tablet

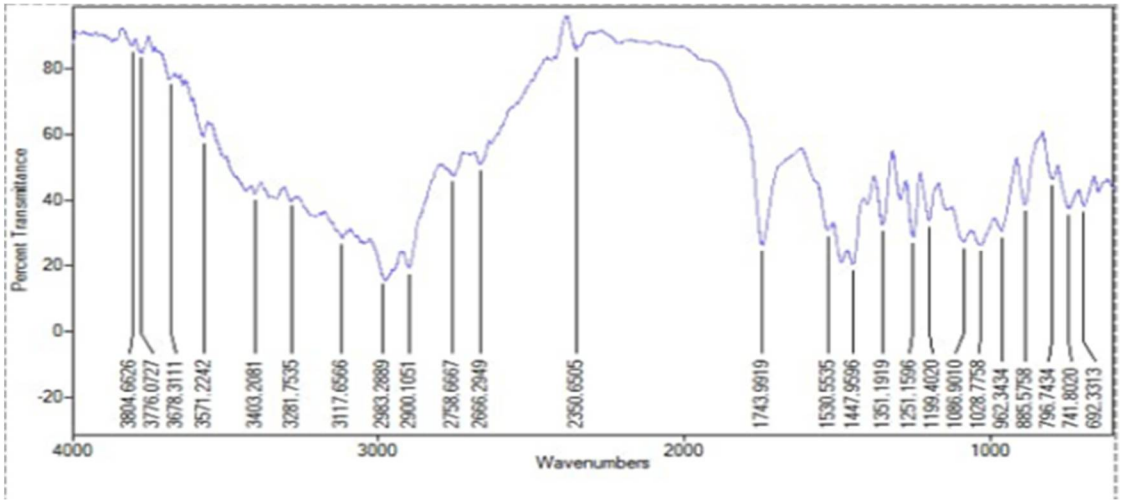

Figure 2. FTIR spectrum of the Sample Brand of Tablet Ibuprofen (Sample A)

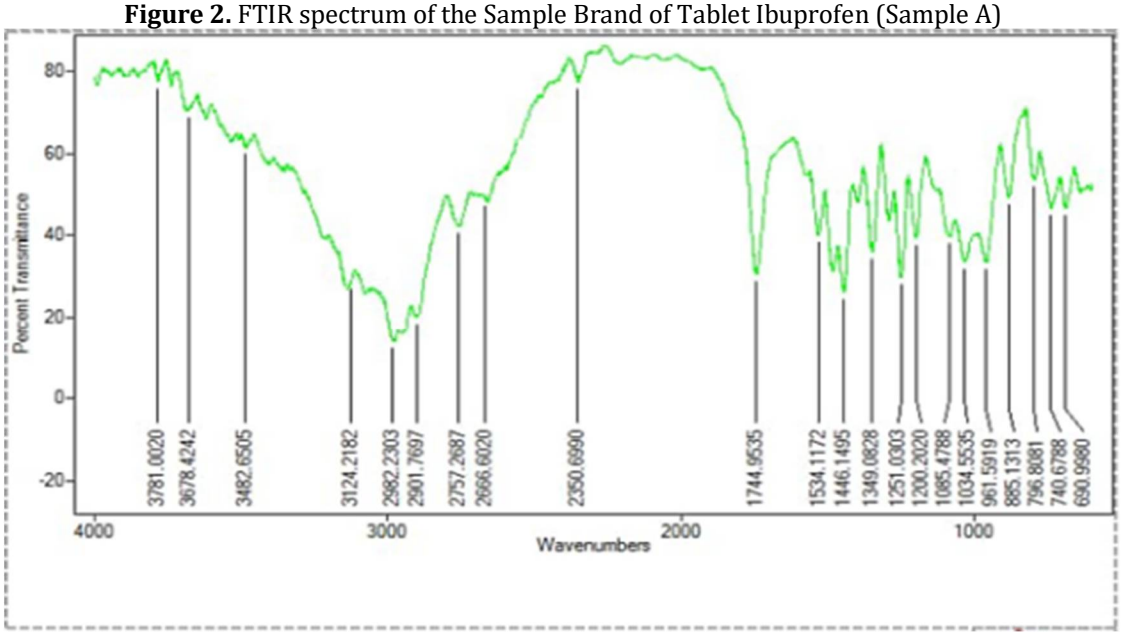

Figure 3. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample B)

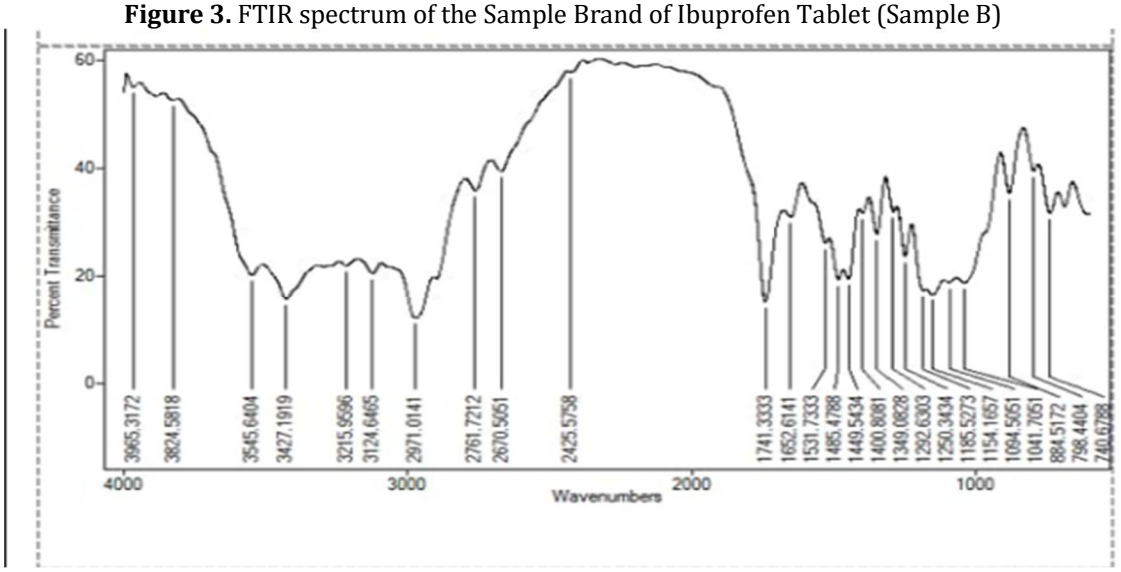

Figure 4. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample C)

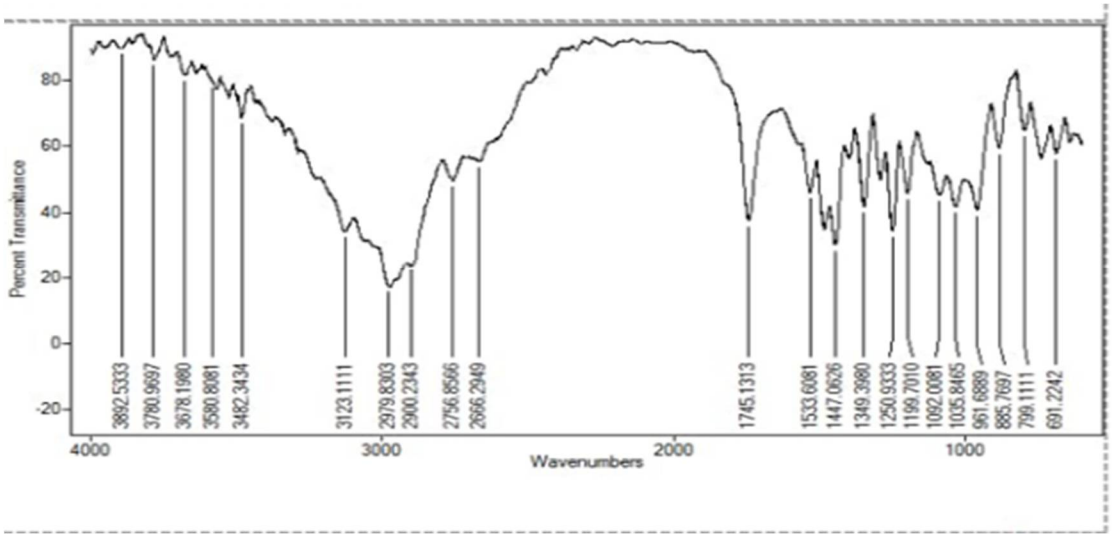

Figure 5. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample D)

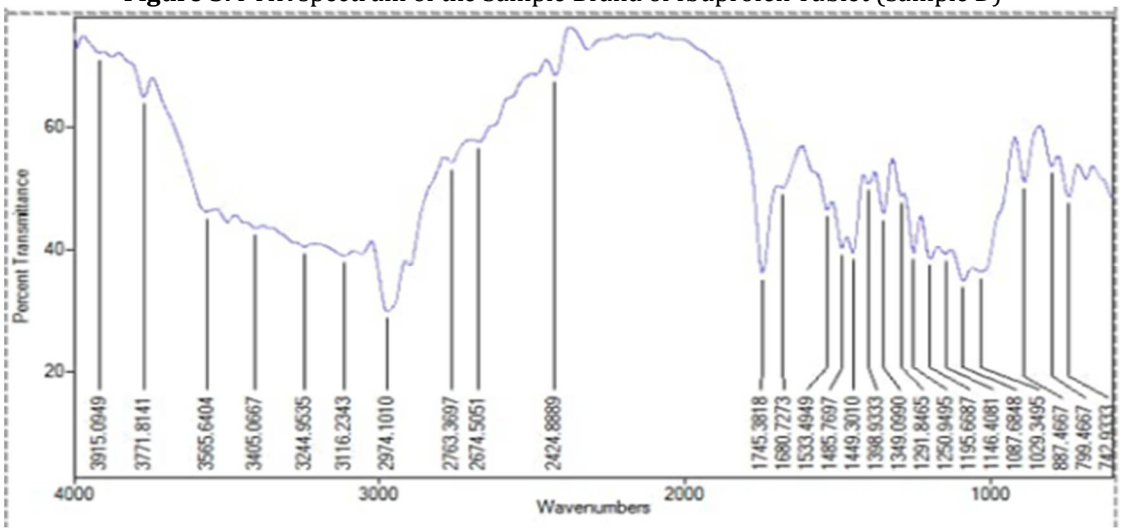

Figure 6. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample E)

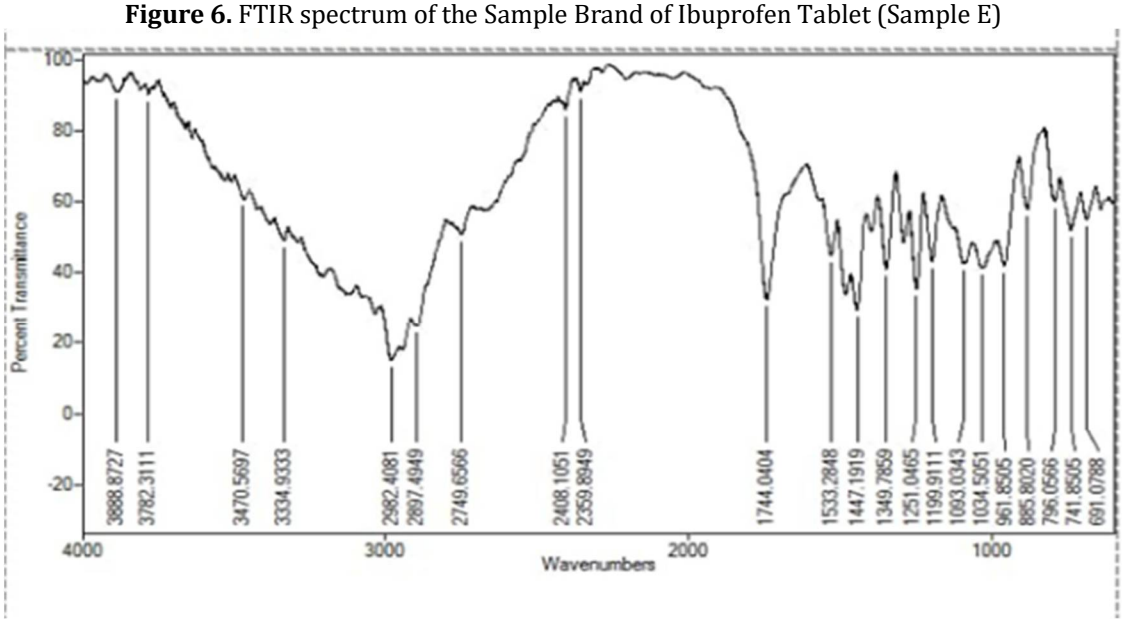

Figure 7. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample F)

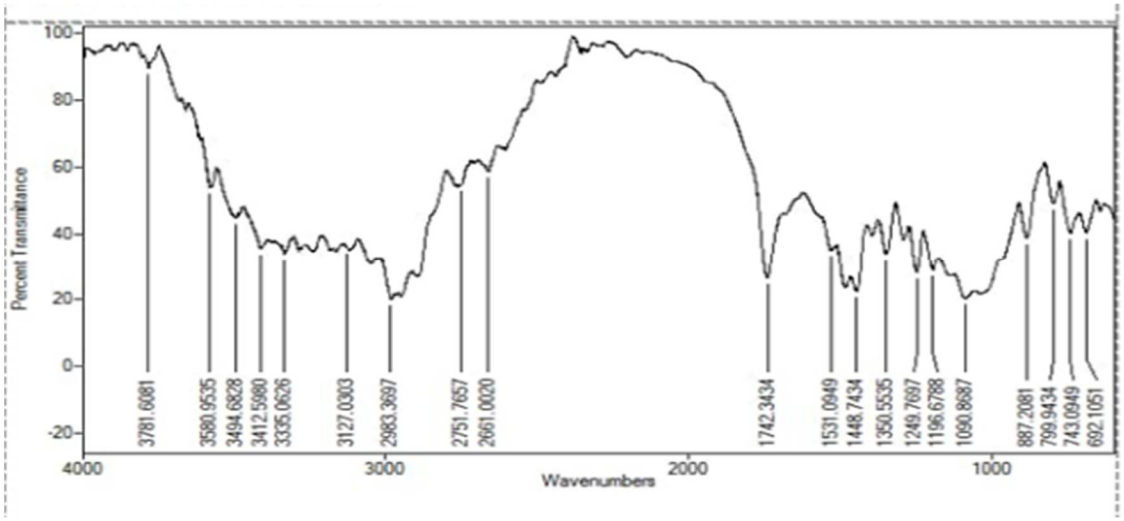

Figure 8. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample G)

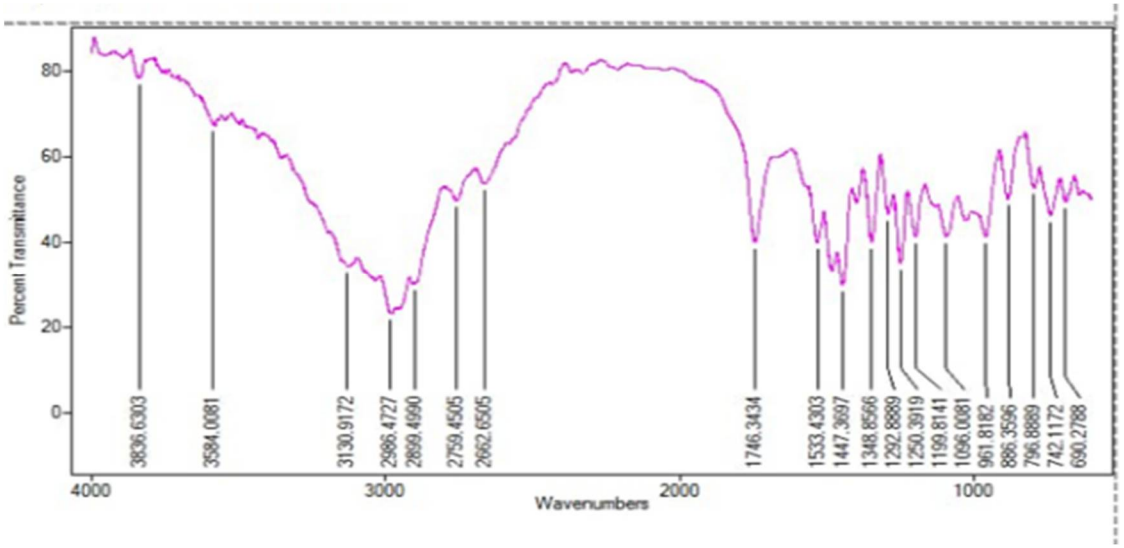

Figure 9. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample H)

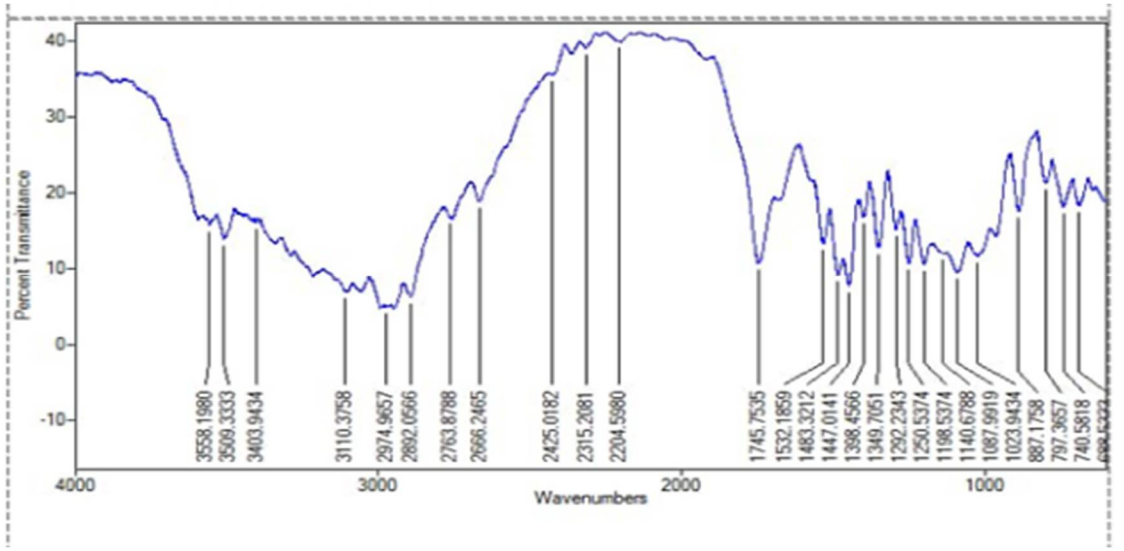

Figure 10. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample I)

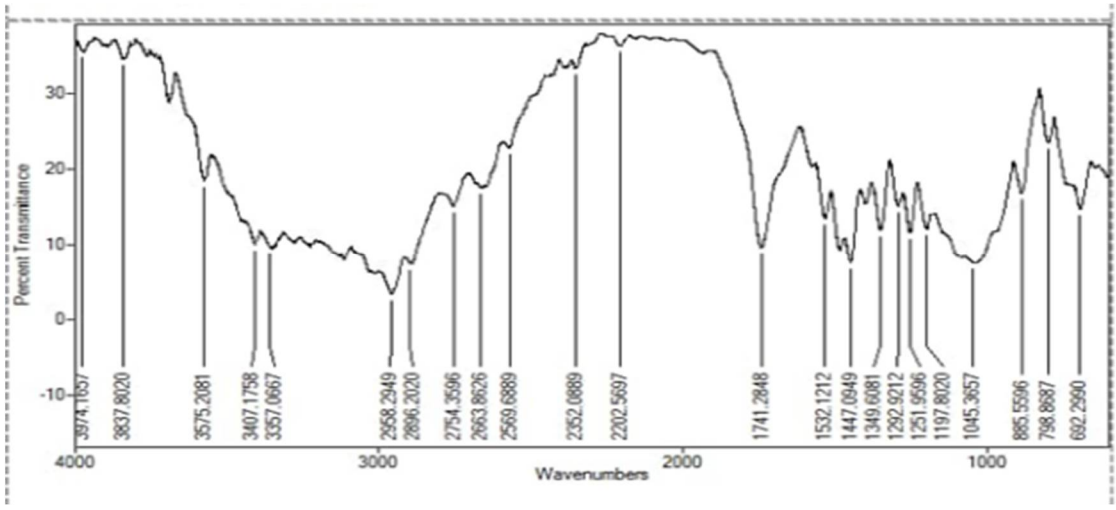

Figure 11. FTIR spectrum of the Sample Brand of Ibuprofen Tablet (Sample J)

Figure 1 above shows the absorption peaks assigned to the various functional groups on the spectrum of the standard Ibuprofen powder. The FTIR spectrum of the standard Ibuprofen powder shows, one prominent characteristic peak found at 2977.76 cm^{-1}, which was assigned to CH_3 Asymmetric stretching for O-H stretching vibration, and another band at 1744.93 cm^{-1} represented the carbonyl C=O stretching vibration from propionic acid.

The band at 743.09 cm^{-1} represented the CH_2 Rocking Vibration of the Isobutyl moiety aromatic benzene ring. The peak at 1449.01cm-1 was assigned aromatic (benzene) indicating C-C Stretching (in the ring). The band at 1088.05 cm^{-1} represented υC-O stretching which proved the presence of carboxylic acid, alcohol, esters, and ethers.

Figure 2 - 11 above also showed the absorption peak ranges assigned to the various functional groups on the spectra of each sample brand of Ibuprofen tablet (Sample A). The FTIR spectra of the ten sample brands of Ibuprofen tablets show, one prominent characteristic peak found between 2958.29 to 2986.47 cm^{-1}, which was assigned to CH_3 Asymmetric stretching for O-H stretching vibration, another band between 1741.01-1746.34 cm^{-1} represented the carbonyl C=O stretching vibration from propionic acid. The bands between 692.80 - 799.11cm^{-1} represented the CH2 Rocking Vibration of the Isobutyl moiety aromatic benzene ring. The peak between 1446.14 to 1449.84 cm^{-1} was assigned aromatic (benzene) indicating C-C Stretching (in the ring). The band at 1045.36 to 1251.04 cm^{-1} represented υC-O stretching which proved the presence of carboxylic acid, alcohol, esters, and ethers.

CONCLUSION

In this study, it was demonstrated that Ibuprofen as an active component was found to be present in all the ten sample brands of Ibuprofen tablets and that all the brands met the criteria specified in the official monographs for in vitro quality control test. It was concluded that all the brands satisfied the Pharmacopoeia specification for Ibuprofen tablets of labeled strength 400mg/tablet. Therefore, the study further concluded that none of the Ibuprofen tablet brands analyzed could be classified as substandard or counterfeit.

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