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Molecular Interaction between CTAB with NSAIDs (IBFN) by using Tensiometry, Viscometry, FTIR, and 1H NMR Techniques

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Abstract
Cetyltrimethylammonium bromide (CTAB), a cationic surfactant, has been interacted with Ibuprofen drug characterized by ^{1}HNMR and FTIR spectroscopictechniques in the solid state. The surface tension method was used to calculate the values of the critical micelle concentration (CMC). Viscosity properties of the mixed system are followed by viscosity techniques. Interaction between anionic surfactants and drugs. These artificially produced surfactants are effective in raising the solubility and bioavailability of drug molecules. Additional screening was done on surfactants to check for antibacterial and antifungal properties. Parameters of the interface, like the maximum concentration of surface excess (Γ_{max}), A molecule's smallest area (A_{min}), π_{cmc}, and the adsorption efficiency (pC_{20}), were determined.

Key words: Nonsteroidal anti-inflammatory [IBFN], CMC, surfactant, FTIR, and ^{1}H NMR.

I. Introduction

Ibuprofen and other nonsteroidal anti-inflammatory drugs (NSAIDs) are the most often used pharmaceutical ingredients for the treatment of inflammation [1][2][3][4]. Their primary mechanism of action involves inhibiting cyclooxygenases, which reduces the synthesis of prostaglandins, which are involved in the reactions of pain and inflammation [5][6]. Nevertheless, a significant disadvantage when considering the integration of most NSAIDs into hydrophilic matrices for drug release and bioavailability is their low water solubility [7][8][9]. NSAIDs' poor water solubility [10]. Amphipathic compounds with polar and non-polar moieties in their head and tail, respectively, are surface-activeagents[11][12]. These surfactants can be categorized as anionic, cationic, amphoteric, or non-ionic based on the charge that is present on their head moieties [13].

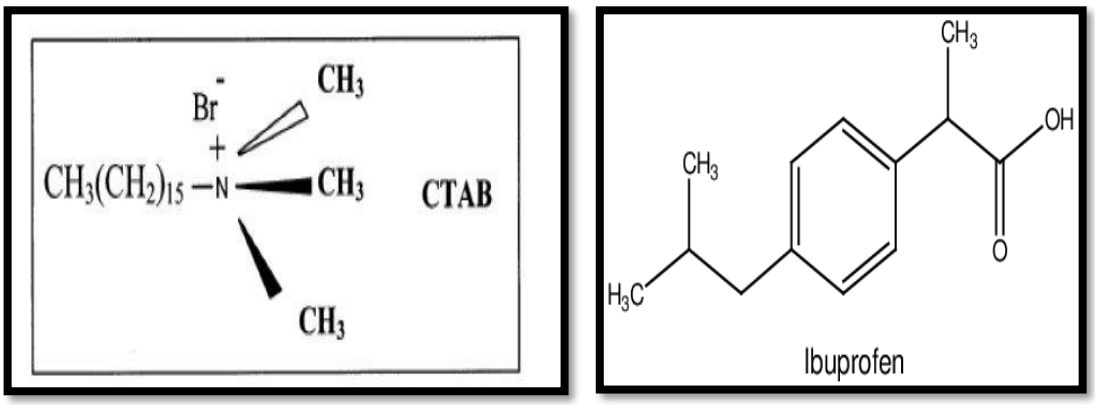

Figure 1. Chemical structure of a) Cetyltrimethylammonium bromide, b) Ibuprofen.

Because of a small hydrophobic group, such as an aromatic ring system, certain amphiphilic medicines can self-assemble into tiny aggregates in aqueous solutions that mimic surfactant micelles [14][15][16]. Surfactants find extensive usage in the chemical, pharmaceutical, material science, and energy sectors as emulsifiers, solubilizers, and detergents in many applications [17][18][19][20]. Utilizing the inherent feature of micelles and other aggregates so generated, surfactants are used in many applications to solubilize materials that would otherwise be intractable in solvent systems [21]. Because surfactants may solubilize a wide range of water-insoluble drugs, they are used in pharmacology as effective agents for delivering drug molecules to be delivered to target organs. Among their many benefits as medication carriers are the following: The capacity to dissolve intractable substances and their retention in the body, particularly in the blood, permit a gradual accumulation in the target organ. It can be required to synthesize novel or inventive surfactants or use mixed micelles of already-existing surfactants in order to reduce the amount and cost of surfactants used, enhance the effectiveness of medication delivery systems, and diminish pollution [22]. Because it is often essential to lower the surface tension of water—something that cannot be achieved with a single surfactant—a range of amphiphilic combinations is utilized [23]. Over the last three decades, several comprehensive studies have been

conducted to examine the efficacy of combinations of amphiphilic polymer-polymer, surfactant-drug, drug-drug, etc., as agents for drug administration. Creating controlled release mechanisms is a compelling application for this subject [24] [25]. Targets should be able to be reached, and the medicine should be released in a regulated way using an ideal controlled release system. In this regard, there are a number of noteworthy benefits to using surfactant micelles as opposed to alternative drug delivery systems like polymers and liposomes.

Banjare et al. have conducted a comprehensive discussion of the molecular and electrostatic, hydrophobic, cation-π, and π–π interactions. Quantitative evaluation of the process of interaction between an anti-inflammatory drug, ibuprofen (Ibu), and 1-dodecyl-3-methylimidazolium chloride (C_{12}mimCl) complexes through various techniques such as surface tension, steady-state fluorescence, UV-visible absorption, dynamic light scattering, and ^{1}H-NMR measurements has been indicated to provide the best knowledge of the C_{12}mimCl – Ibu interaction in a synergistic manner [10]. Banipal et al., have studied the physiochemical interaction parameter, partition coefficient (*Kc), binding* constant (Kb), and thermodynamic parameters of micellization of drug-surfactant mixtures as sulfamethoxazole (SMZ) and trimethoprim (TMP) with cationic surfactant (CTAB) and anionic surfactant (SDS) using UV–visible spectroscopy, electrical conductivity, and cyclic voltammetric techniques [26].In the current study, we measured the interaction between IBFN and Cetyltrimethylammonium bromide (CTAB) using the Surface tension method, viscosity, FTIR, and H NMRmethods. In addition to being well-known solubilization enhancers in the detergent and pharmaceutical sectors, hydrotropes can have a significant effect on the surface tension of solutions. The presence of the additives in any given situation affects the drug-surfactant combinations' micellar and solution properties (interfacial characteristics), as well as the relationship between the surfactant molecules and the medication [27] [28].The interpretation of these findings revolves around the modifications of hydrophobic and electrostatic effects, their impact on the energetics of micellization and solubilization, and the potential implications for the use of these systems as formulations for controlled release [29].

2. Materials and Methodology

2.1 Material:

Ibuprofen [2-(p-isobutylphenyl)propionic acid] [(≥99% purity) (CAS No. 15687-27-1)] was acquired from Sigma Aldrich Pvt. Ltd. in India. Cetyltrimethylammonium bromide (CTAB) [(>99.0% w/w) (CAS No. 57-09-0)] was bought from Merck and utilized without being further refined. To make the 0.001 M stock solution of CTAB and the 0.5 M stock solution of IBFN were made with double-distilled and deionized water. The chemical structures are displayed in Scheme 1.

2.2 Methods:

2.2.1 Surface Tension Method:

A Stalagmometer (ABGIL Borosilicate, India) was used to determine the surface tension of surfactants and study their effect on NSAIDs, calibrated using double-distilled water and using the "drop counts method". It was used to calculate the surface tension and CMC of the surfactant-drug mixed system at a temperature of 299 K.

2.2.2 Viscosity Method:

A viscometer (ABGIL Borosilicate, India) was used to determine the relative viscosity of surfactants and study their effect on NSAIDs, calibrated using double-distilled water and using the time flow method. It was used to calculate the viscosity of a surfactant-drug mixed system at a temperature of 299 K.

2.2.3 FTIR Spectroscopy:

Bruker, Billerica, Massachusetts, USA, manufactured the DRS-FTIR (diffused reflectance Fourier transform infrared spectroscopy) instrument, which was used to record the FTIR spectra of the pure surfactants (CTAB), drugs (ibuprofen), and their mixtures. Model: Alpha-II. Averaging 24 scans at 4 cm-1 resolution across the 600–4000 cm-1 spectral range yielded all of the spectra, with platinum ATR diamond accounting for wavelength dependence [30].

2.2.4 1H NMR Spectroscopy:

Spectroscopyisusedinanalyticalandphysicalchemistrytounderstandcompoundstructureandlocation.^{1}H-NMRspectraofpureIBFNandSURFACTANT-IBFNwererecordedusingaBrukerAvanceDRX400MHzNMRspectrometer.Thechemicalshiftvaluesof eachsampleweremeasuredinppmunitsafter being dissolved in1ml of D_2O.

3. Result and Discussion

3.1 Study of micellarbehavior of CTAB with NSAIDs

Surface tension was used to examine the micellarbehavior of surfactant and ibuprofen drug solution system made from distilled water. We have noticed that the CMC of drug-surfactant systems using surface tension techniques at 299 0.5K.

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Table 1 for the CMC values of pure CTAB-NSAIDs (ibuprofen) systems. The CMC values are significantly reduced in CTAB-Ibuprofen in comparison with water. The CTAB-Ibuprofen system has the best significance on CTAB**.**

S.No.	Ibuprofen drug (M)	CMC value of CTAB
1.	**Water**	0.001(0.001)[a]±0.0001
2.	0.05 M	0.0008±0.0002

Banjareetal[10][a]
Table 1. The calculated CMC values of CTAB with various concentrations (M) of Ibuprofen.

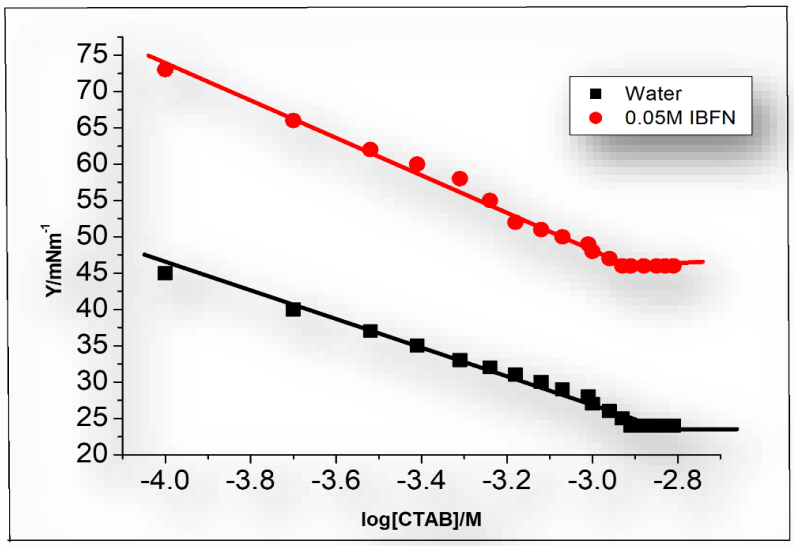

Figure 2. A graph plotting the logarithm of CTAB against IBFN drug.

3.1.1 Surface tension measurements
The surface activity of surfactant CTAB was determined in an aqueous solution with 0.05M concentration of ibuprofen in aqueous solution by using a stalagmometer measurement at 299 K temperature to provide the CMC value for each Surfactant-IBFN system. The CMC value of the CTAB-IBFN system increased, resulting in higher solubility and bond formation between IBFN and CTAB interfaces [10]. Surface tension decreased with IBFN concentration.

3.1.2 Effect of CTAB on the interfacial parameter of the micellar system
Some effective parameters of the interface, like the maximum concentration of surface excess (Γ_{max}), A molecule's smallest area (A_{min}), surface pressure at CMC (π_{cmc}), and the adsorption efficiency (pC_{20}), were determined.

a) Maximum surface excess concentration (Γ_{max})
It is calculated from the slope of (dγ/ dlog10C) using Eq. 1:

$$\Gamma_{max} = 1/\ 1\ 2.303nRT\ x\ (d\ \gamma\ /d\ logC\ )_{T.P.} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

The values of Γmax were calculated using Eq. 1 and 0.05 M concentration of IBFN with CTAB at a temperature of 298 K. The Γmax value decreases with increasing concentration of the drug. This results in the improved hydrophobic character[22]. As a result of the high surface activity of CTAB give rise to a buildup tendency to aggregates around the air-water interfaces of surfactant molecules.

b)Minimum area per molecule (A_{min})
The air–water interface was calculated by using Eq. 2:

$$A_{min} = 1/\Gamma_{max}NA \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$

The order obtained for the minimum area per molecule is molecule-increased. The minimum a molecules is inversely proportional to the maximum surface excess concentration. In the CTAB-IBFN system can be shown that the Γ_{max}value

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decreases and the A_{min} value increases, resulting in the molecules being less tightly packed for being flexible at the air-water contact. It can be indicated that the effect of CTAB reduces the surface area of surfactant molecules perfectly [26].

c) Efficiency of adsorption

It is calculated from Eq. 3. It has been determined that the pC_{20} values decrease with an increase in 0.05 M concentration of Ibuprofen drug in the binary systems.

pC_{20} = –log10 C20……..(3)

S.No.	Conc.	γ_{CMC}	Γ_{max} (molm	A_{min}	π_{CMC}	pC_{20}
1.	Water	29(23)[a]±0.0001	1.37±0.0006	0.120 ±0.0005	43.0±0.0 002	4.42±0.0001
2.	0.05	46.286±0.0002	0.0031±0.0004	1.833±0.0002	25.71±0. 0002	0.030±0.0001

Table 2. The γ_{CMC} is the surface tension of Ibuprofen, surface excess concentration (Γmax), minimum surface area per molecule (Amin), surface pressure at CMC (π_{CMC}), and the efficiency of absorption (pC_{20}) of CTAB mixed media at 298 K.

Banjare et al. (10)[a]

d) surface pressure at the CMC (π_{CMC})

The surface tension of distilled water and the connection among surface active agents and the air-water surface for effective surface assimilation of 0.05 M concentration of Ibuprofen. The maximum value of π_{CMC} indicates that the CTAB and Ibuprofen system has more effective adsorption due to its ionic molecules as well as its larger water-affine hard part. The sequence of π_{CMC} is shown in Table 2, indicating a higher interaction between the surfactant and drug.

3.2 Viscosity Study

The ratio of the suspension setup to the solvent viscosities is characterized as the Relative viscosity.

The Viscometer technique was magnified significantly to examine rheological properties and conformational properties changes of solvents that exposed viscosity properties. Numerous surfactant-Ibuprofen setup was characterized by estimating their relative viscosity, which was considered by Eq. (4), and the histogram schemed between the relative viscosity vs the concentration (M) of various setups is shown in Figure 3.

$\eta_{0} = /\eta$ ..... (4)

Where η_0=the viscosity of the Pure solvent, η_r=the relative viscosity of the setup, and η_s=the viscosity of the suspension. A suspension of a concentration was utilized for the attributes pinpointing of the surface-active agents-drug setups. The viscosity activity was initiated when the inaugural twist occurred at different concentrations of the CTAB-drug setup and, as a consequence, accepted the changes. In the same way, the 0.05M concentrations of the CTAB-drug setup have been examined in a coming up break and trouble-free relative viscosity bend than at least concentrations.

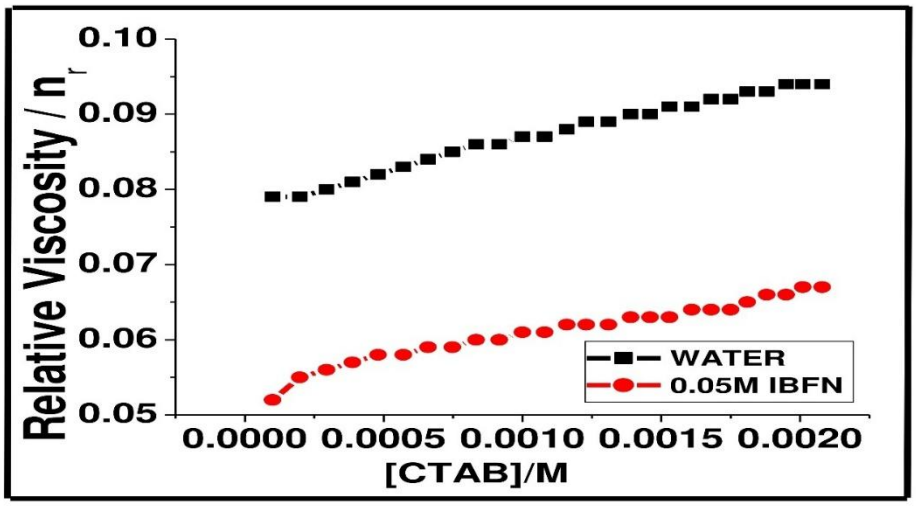

Figure 3. A Graph plots the relative viscosity of CTAB in the presence of 0.05 M concentration of IBFN at 298 K.

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3.3 FTIR study of CTAB and NSAID [IBFN] drug

It is the most powerful for classifying organic or inorganic chemicals, detecting intermolecular interactions in surfactant molecules technique of surfactant deduces insightful information about the molecular structure and molecular interaction prevailing within the chemical system. In the present study, FT-IR spectra of the various surfactants with aqueous paracetamol have been measured by using a Bruker, Billerica, Massachusetts, USA, manufactured DRS-FTIR (diffused reflectance Fourier transform infrared spectroscopy) instrument, summarized in Table 3 and Figures 3 and 4.

Functional group	Frequency Range (cm^{-1})	Pure IBFN	IBFN -CTAB
O-H stretching vibration	3854	3783 3679 3092	-
Phenols	3587.12	-	-
Bonded stretching of amines and amides	3373-3422	-	-
C-H Symmetric and asymmetric stretching bands	2918.2-2954	2952	2923.86
Carboxyl acid	2500-3300	-	-
C-N	2322.8-2138.1	-	-
Silicon compounds	2047.30	-	-
Ketones	1733.59	1708	1659.12
Alkanes	1405-1445	-	-
C-O/C-H bending	1421-1415	1422	1511.80
C-O	1382-1036	-	1373.41
Alkyl ketone	1215-1325	-	1216.67
Alkyl amine	1020-1220		-
Vibration of the C-O in the alcohol hydroxyl group	1026	1006	
Alkyl halides	469	860	835.82

Table 3. IR Absorption bands of conventional surfactant CTAB and IBRUFEN.

3.3.1 IR spectra of Pure IBUPROFEN.

IR spectra of ibuprofen can be studied by using the FTIR Technique. The NSAIDs of pure ibuprofen spectra are shown in Table 2 and Fig. 2 (a) and (b). The carboxylic group containing [O–H] stretching (broad) bands is observed at 3783 cm^{-1}, 3679 cm^{-1}, and 3092 cm^{-1} for IBU. The symmetric and asymmetric [C–H] stretching bands are observed at 2952 cm^{-1}, 2921 $cm^{-1,}$ and 870 cm^{-1} for IBFN. The sharp peak [O–H] bending is observed at 1231 cm^{-1} for IBFN. The carboxylic and ester group with [C-O] stretching bands are observed at 1708 cm^{-1} for IBFN. The aromatic methylene ring containing [C–H] bending is observed at 1422 cm^{-1} for IBFN. The [C–O] stretching band is observed at 1006 cm^{-1} for IBFN. The aromatic ring [C-C] bending spectra are observed at 860 cm^{-1} and 777 cm^{-1} for IBFN.

3.3.2 IR spectra of the mixed CTAB + IBUPROFEN system.

IBRUFEN -CTAB was observed, the broad band is the C-H BOND observed at 2923.86 cm^{-1}.Functional group Ketones were observed at 1659.12 cm^{-1}, C-O/C-H bending 1511.80 cm^{-1}, C-O band observed as 1373.41 cm^{-1}, Alkyl ketone band observed at 1216.67 cm^{-1}. The alkyl halides group observed at 835.82 cm^{-1}

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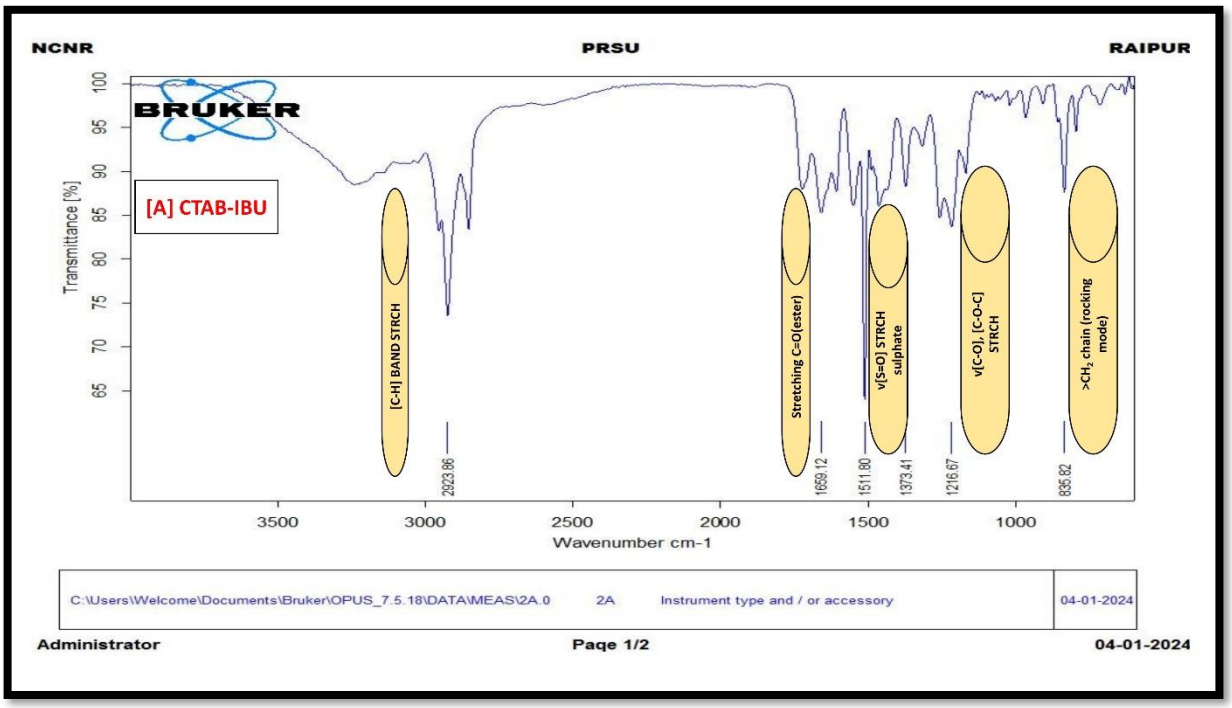

Figure 3.FTIR spectra of different conventional CTAB- IBFNdrugs.

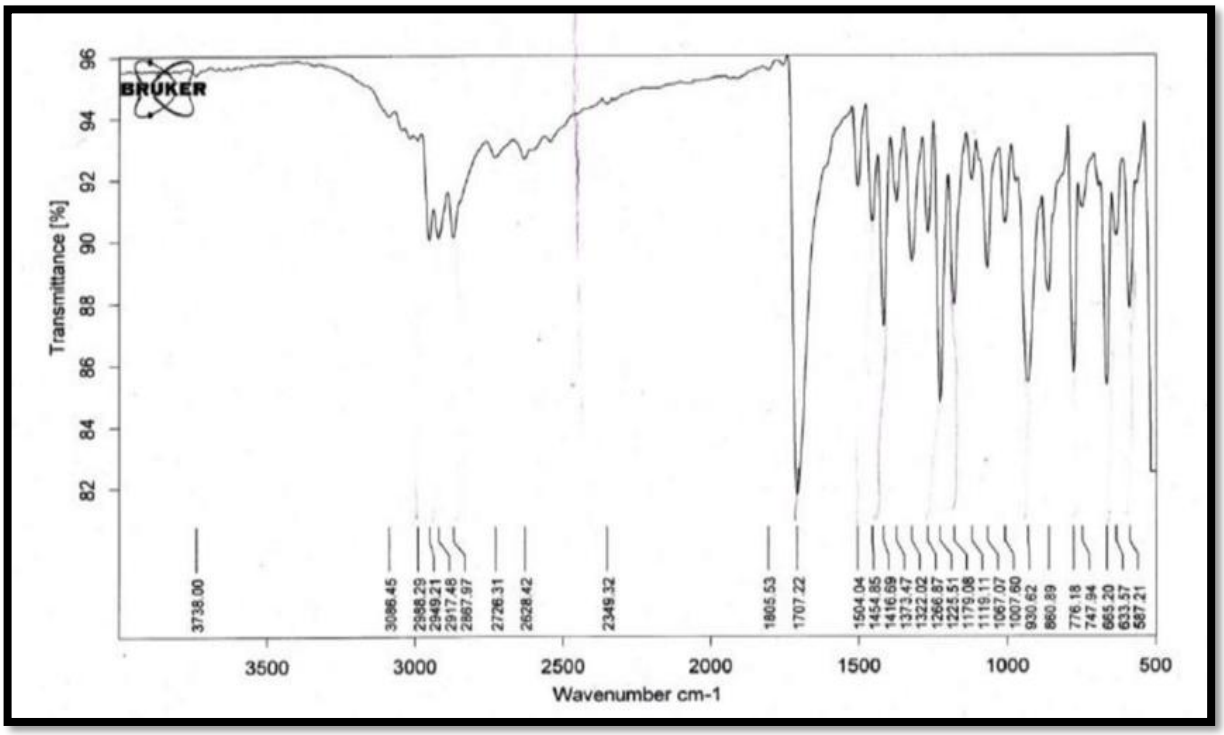

Figure 4. FTIR spectra of different conventional Pure IBFN drugs.

Randomly, surfactant interaction with IBFN can break down and modify the hydrogen bond structure. Numerous hydrogen bonds are produced in IBFN has OH− and –COO− groups. The interaction between CTAB-IBFN. These possible molecular interactions are shown in Figure 3. The C–O stretching bands in the case of IBFN have a bandwidth at 1710 cm−1 following irradiation, but the first feature is a strong peak at 1708 cm^{-1} [33]. These findings suggest that when the two samples were exposed to radiation, the hydrogen bond structure dissolved and reorganized [34]. The fingerprint region changed the peak locations of many bands, as well as a shift in the relative intensity. The alkyl halide shown in the IR frequency is 835 cm^{-1} and 860 cm^{-1} regions and is shifted to 835.82 cm^{-1}.

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3.4 ^{1}H NMR Study

NMR is a highly sensitive method for determining if inclusion occurs and how it does. Due to their mutual screening throughout space, the interacting protons of the surfactants (CTAB) and IBU undergo a chemical shift in 1H-NMR spectra as an outcome of the molecular coupling betweensurfactant CTAB with Ibuprofen [29].

Table 5. Transform in chemical shift (δ ppm) of the H of Ibuprofen molecule when complexed with surfactant, i.e., CTAB, molecules in DMSO solvent at 300K.

Proton Signal (^{1}H-NMR)	**IBU**	**CTAB-IBU**
	Chemical Shift (δ)	
Ha	7.15, 7.13	
Hb	7.27, 7.25	
Hc	3.74	4.531
Hd	1.90	1.981
He	1.85	1.311
Hf	1.55	1.042
Hg	0.94, 0.93	0.812

Therefore, NMR has been used extensively in pharmaceutics to investigate the structure of surfactants, larger molecules, and complexationbetween globular proteins and metallic nanoparticles, medications, and other applications. The tiny hydrogen molecule near the IBFN saw the strongest chemical change and NMR signals during the interaction. The NMR phenomena are visible due to the good solubility of Ibuprofen; the mixture of surfactants in IBFN was carefully examined. The CTAB-IBFN blend produced the best outcomes. The recognized outcomes can offer appropriate proof of the impact of surfactants' additional hydrophobic/hydrophilic component on forming complexes with IBFN in water solution. The current work assessed the chemical shifts of IBFN protons and examined the impact of surfactants on IBFNcomplexation. The proton NMR spectra of 0.1M IBFN in DMSO solution with surfactants are displayed in Figure 5, and Table 5 shows all of the 1H-NMR data. The addition of three surfactants causes the Ha signals of the IBFN's Hb to Hg to shift downfield and the Ha of the –CH3 group on the IBFN +ve ion to shift upfield.

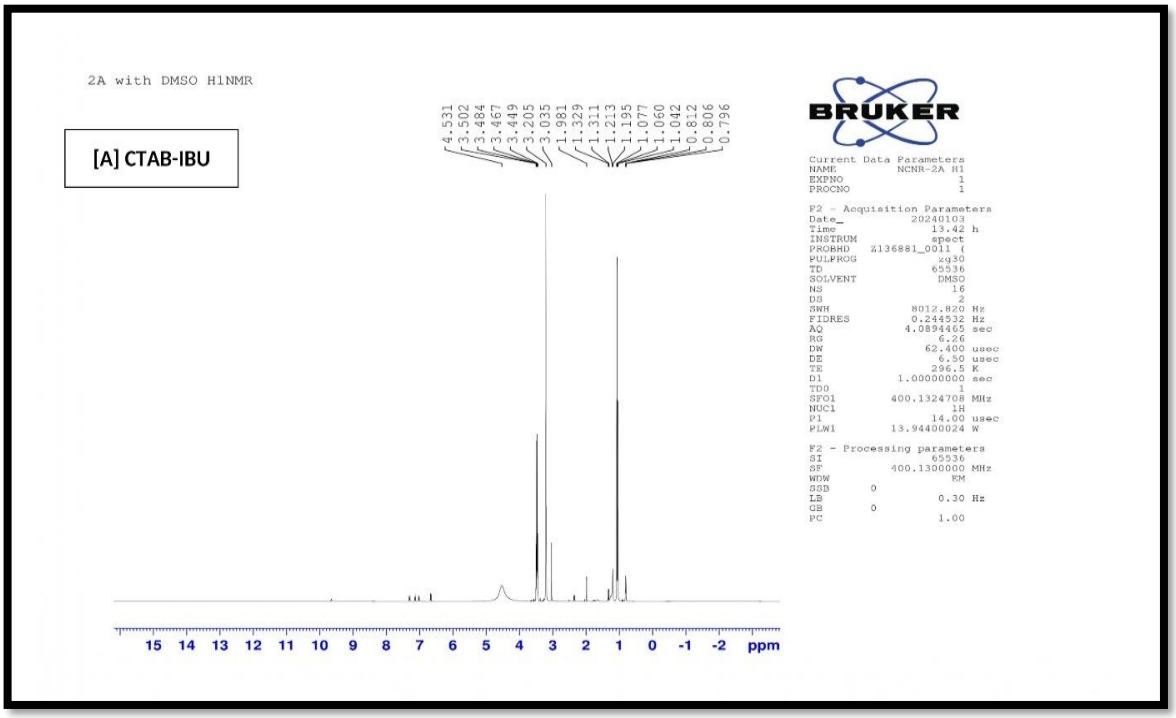

Figure 5. ^{1}HNMR spectra of the mixed system, i.e., CTAB-IBU.

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CONCLUSION

The molecular interaction of Cetyltrimethylammonium bromide (CTAB) and NSAIDs, i.e., ibuprofen (IBFN), was studied by calculating the CMC value and their interfacial parameter using the surface tension method. The relative viscosity graph is noted by using a viscometer. The FTIR data showed that the CTAB + IBFN system has good agreement and has been utilized as a cosolvent for the entire system, which can help bind CTAB and NSAIDs. In agreement with the FTIR, it was shown that CTAB with IBFN causes compositional changes. Consequently, we may say that using an appropriate CTAB will help have a positive impact on drug delivery and reduce the deficiencies of drugs. The current research will have effects on drug delivery, molecular biology, and pharmaceutical sciences.

Author contributions

The manuscript was written through the contributions of all authors. All authors have approved the original version of the manuscript.

Conflicts of interest

There are no conflicts to declare.

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