

INFLUENCE OF MICELLIZATION BEHAVIOUR OF NON-IONIC SURFACTANT WITH ANTI-INFLAMMATORY DRUG.

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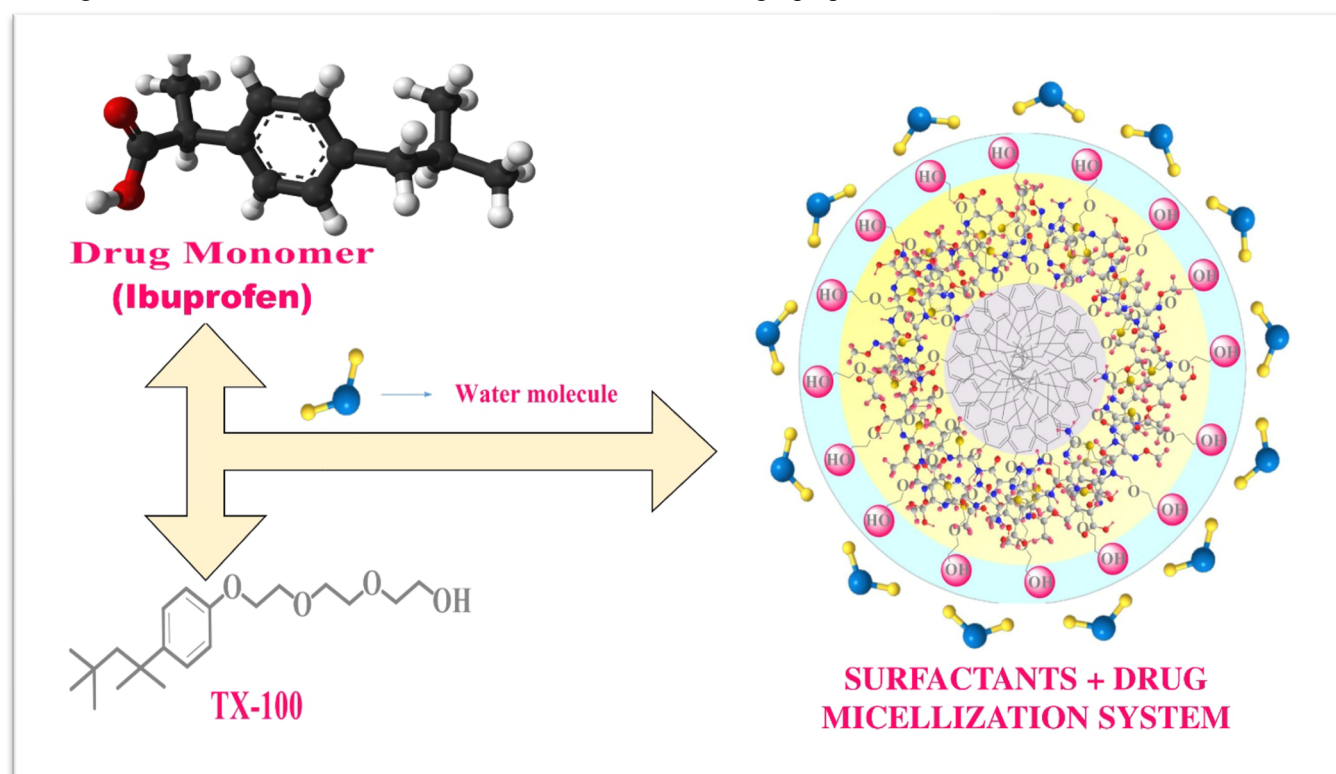
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Abstract

surfactants have shown promise in pharmaceuticals addressing stability, toxicity, and drug solubility issues. However, developing effective medication delivery mechanisms is crucial. Our current study focuses on understanding the micellization behavior of Triton X-100, a surface-active ionic liquid (SAIL) in the presence of anti-inflammatory drug (NSAIDs). The study employs surface tension measurements to assess the micellization nature of the surfactant and drug mixture at various concentrations. Viscosity properties of the mixed system are followed by viscosity techniques. Interaction between anionic surfactants and drugs. The results show a significant change in the CMC value with the rise in drug concentration, which depends on the nature of the drug. Triton X-100 (TX-100), a non-ionic surfactant, has been interacted with Ibuprofen drug characterized by ¹HNMR and FTIR spectroscopic techniques in the solid state. 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.



Keywords: Surfactants, Drug (NSAIDs), and Micellization

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1. Introduction

Door-to-door drug delivery and health care at home are rapidly growing due to rising demand for convenience, chronic disease management, telemedicine, and post-COVID care shifts [1]. Behind this, the society may face many issues in drug delivery due to their exceptional ability to enhance the solubility, stability, and bioavailability of pharmaceutical compounds, especially

poorly soluble drugs [2]. Their tunable chemical structure allows for customization in drug formulation to enable targeted delivery, controlled release, or increased membrane permeability [3]. To overcome these issues, our research is going to upgrade a mixed system known as TX-100 + Drug system, i.e., TX-100 + IBU [4][5]. In this evolving landscape, TX-100 can play a crucial role in enhancing the utility and effectiveness of drugs,

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especially in decentralized or home-based treatment models [6][7][8].

Surfactants are a class of salts featuring imidazolium cations paired with various anions, which are known for their low volatility and high thermal stability because of their antimicrobial properties, offering dual-functionally as both drug carriers and therapeutic agents [9] [10]. Furthermore, their environmental friendliness and recyclability contribute to their increasing use in green chemistry and sustainable technologies [11]. Ibuprofen is a common analgesic and anti-inflammatory drug, but its low water solubility makes it difficult to provide advanced treatment [12][13][14].

The self-aggregation characteristics of long alkyl chain surfactant are being studied to improve their applications. Current research focuses on surfactant in medication delivery to increase bioavailability and reduce the harmful effects of micellization [15][16][17]. The interactions between drugs and surfactant are crucial for improving drug delivery [18][19].

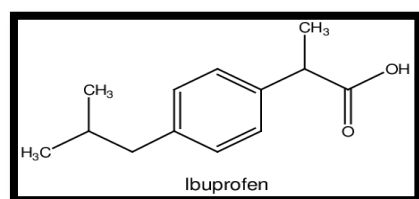
Research explores the use of surface-assisted ionic liquids (SAILs) in ibuprofen drugs to improve their anti-inflammatory activity against Probiotic strains, addressing drug resistance issues through micellization behaviour [20][21].

surfactants can act as solubilizing agents for ibuprofen, which might otherwise be poorly soluble in water [22][23]. This means they can form micelles in an aqueous solution, where the charged head groups interact with water, and the hydrophobic tails cluster together [24]. The use of SAILs in medicine delivery is currently the subject of extensive research in addition to all of these applications [25][26]. Many studies on the interactions between medications and surfactants have

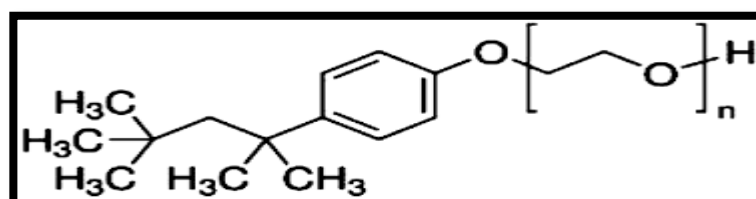
been conducted to increase bioavailability and reduce the harmful effects of drugs through the use of micellization [27][28].

Here we can review a few literature that are somewhat related to our research, such as Banjare *et al.*, who performed the physicochemical characteristics of nortriptyline hydrochloride with 1-ethyl-3-methylimidazolium bromide, 1-hexyl-3-methylimidazolium bromide, and 1-butyl-3-methylimidazolium bromide by using viscometry, volumetric analysis, and acoustic testing at 288.15 and 318.15 K. The result is that when the imidazolium ionic liquid's alkyl chain length grows, so does the strength of these interactions [29]. Similarly, Ghosh *et al.* used the surface tension, conductivity, fluorescence, and DLS to study the colloidal behaviour of 1-decyl-3-methylimidazolium tetrafluoroborate [Dmim][BF₄] and antidepressant medications, such as CPZ/PMZ, with tetradecyltrimethylammonium bromide (TTAB). Consequently, imidazolium-based ionic liquids would be preferable to conventional cationic surfactants in terms of medication delivery. The medication and surfactant systems are indicated by micellar and interfacial properties [25].

The study explores the impact of Triton X-100 with oral drugs like Ibuprofen. The researchers used surface tension, and conductivity to analyze critical micelle concentrations, interfacial notations, surface pressure, and adsorption efficiency. FTIR, and ¹H NMR characterised the mixture system. The current agreements will incorporate pharmaceutical sciences, cosmetics, medicinal delivery, and household goods [30][31][32].



Ibuprofen



Triton X-100

Figure 1: Structure of Ibuprofen, and Triton X-100.

2. Materials and methods

2.1. Materials

Ibuprofen [2-(p-isobutylphenyl)propionic acid] [(≥99% purity) (CAS No. 15687-27-1)] was acquired from Sigma Aldrich Pvt. Ltd. in India and Sigma-Aldrich in the United States provided the Triton X-100 [Polyethylene glycol p-(1,1,3,3-tetramethylbutyl)-phenyl ether, t-Octylphenoxy polyethoxyethanol] (8.0%), [(≥90% purity) (CAS No. 9002-93-1)] were acquired from Sigma Aldrich Pvt. Ltd. in India.

2.2 Methods

a) Stalagmometer

A Stalagmometer (ABGIL Borosilicate, India) was used to measure the surface tension of the TX-100 and NSAIDs (Ibuprofen) systems. Before the trials, the equipment of the platinum ring was carefully cleaned and flame-dried. By pouring a solution just beyond the designated threshold and counting the number of droplets passing through the capillary, the "drop volume count" viewpoint was ascertained [33]. The logarithm of surface tension vs surfactant concentration (M) of all systems was plotted on a graph.

b) Viscosity Method:

A viscometer (ABGIL Borosilicate, India) was used to determine the relative viscosity of surfactants and study

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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 [24].

c) 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 (TX-100), drugs (Ibuprofen), and their mixtures [34]. 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 [35].

d) ¹H NMR Spectroscopy:

Spectroscopy is used in analytical and physical chemistry to understand compound structure and location. ¹H-NMR spectra of pure IBU and TX-100-IBU were recorded using a Bruker Avance DRX 400 MHz NMR spectrometer. The chemical shift values of each sample were measured in ppm units after being

dissolved in 1ml of D₂O [36].

3. Result and discussion

3.1 Study the micellar behavior of surfactant [TX-100] with NSAIDs Drugs

The study examines the interaction between IBU drug with a non-ionic surfactant (TX-100) in an aqueous medium. Four sets of experiments were conducted to study molecular interactions and their connection. The results show that as the concentrations of these medications decrease, the CMC values for TX-100 shift [18].

a) Surface tension method

Surface tension is the change in free energy per unit area and is determined by the Langmuir principle, which links intermolecular interactions in bulk and interactions between molecules at the surface [25]. As the length of the alkyl chain in a cation increases, surface tension values tend to decrease. The CMC data of pure TX-100 were found to be 0.0009 M. Lower concentrations of polar hydrophilic medicines make them more soluble in water.

Methods	Concentration	Ibuprofen
Surface tension	Water	0.0009(0.00798) ^a ±0.0002
	0.03	0.00019±0.0004
	0.05	0.00017±0.0007
	0.07	0.00015±0.0004
	0.09	0.00013±0.0006

Table 1. The CMC of TX-100 in H₂O with IBU by using Tensiometric techniques.

Banjare et.al. [24]^a

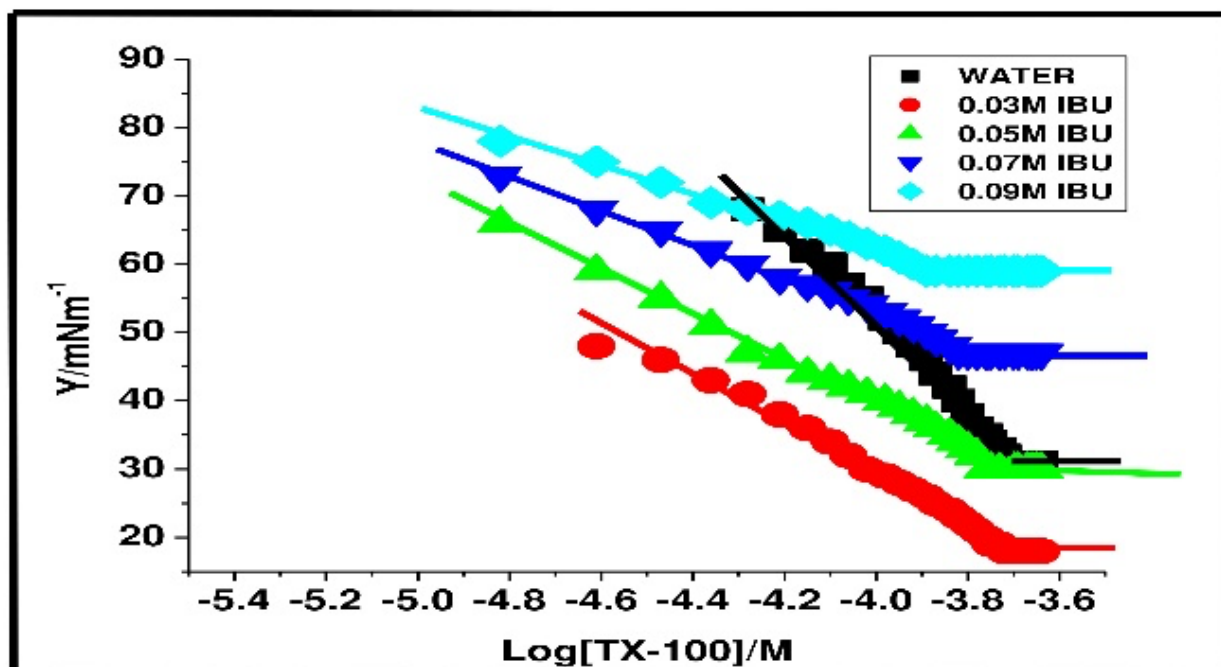


Figure 2: A graph plotted between the surface tension γ Vs log concentration. of TX-100 in the presence of different concentrations of Ibuprofen drug.

b) Study the Interfacial Parameter of TX-100 with Drug

Equations (1) through (4) were used sequentially to calculate different interfacial characteristics, surface tension at CMC, the minimum surface area per molecule (A_{min}), including the maximum surface excess concentration (Γ_{max}), the efficiency of adsorption (pC_{20}) and the surface pressure at CMC (π_{CMC}) of Surfactants and drugs (TX-100 + IBU) system. The metrics assess the drug and surfactants-adsorbed amphiphiles' orientation and position at the surface. The breakdown of H-bonds at the surface and the rise in amphiphile concentration are linked to this drop in surface tension in the hydrated phase. Figure 2 shows the variation in surface tension (γ) with the log of surfactants in aqueous pharmaceuticals at room temperature.

- **The Gibbs adsorption equation was used to quantify the excess surface concentration (Γ_{max}):**

$$\Gamma_{max} = 1/ 2.303 n RT. [dY/ d \log C]_{T,P}. \quad (1)$$

Where T is the temperature, NA is Avogadro's number, R is the universal gas constant (8.314 J mol⁻¹ K⁻¹), and n is the no. of the ionic category of the surfactant that varies with interface and bulk concentration [17]. Y is the surface tension expressed in mNm⁻¹. A_{min} is the minimum area per head group. According to the literature that is currently accessible, n = 1 was chosen for this investigation, and C stands for the concentration of surfactant in solution. The recorded interfacial parameters are displayed in Table 2. The Γ_{max} value has been recorded at various concentration, and observed values.

- **The minimum area per head group (A_{min}):**

Eq. (1) can be used to get the minimum area per head group (A_{min}), as

$$A_{min} = [1 / \Gamma_{max}]. NA \quad (2)$$

The A_{min} parameter indicates the minimum area per head group in the system. The value of CMC in the TX-100 + IBUPROFEN system leads to highly effective adsorption at the interface of surfactant because of positive-negative repulsion of this system is greater.

- **The surface pressure (π_{cmc}) at CMC:**

$$\pi_{cmc} = \gamma_0 - \gamma_{cmc} \quad (3)$$

For effective surface absorption of drug concentrations, the surface tension of distilled water and the relationship between surface active agents and the air-water surface are crucial. As can be seen from Table 2, suggesting a stronger interaction between the drug and surfactant.

- **The adsorption efficiency (pC_{20}):**

$$pC_{20} = - \log_{10} C_{20} \quad (4)$$

Finally, the standard surface phase state involves the surface being coated, including a single layer of surface-active agents at a surface pressure of zero. The pC_{20} parameter indicates that more efficient adsorption at the air/water interface, with a higher value leading to more adsorption and a lesser value leading to less adsorption resulting in the concentration of the drug (ibuprofen) with surfactant being indirectly proportional to the pC_{20} value. The value can be shown in Table 2.

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Ibuprofen (M)	A _{min} (m ² mol ⁻¹)	pC ₂₀	πCMC (mNm ⁻¹)	Γ _{max} (molm ⁻²)	γ _{cmc} (mNm ⁻¹)	CPP
Water	0.053± 0.002	4.27±0.03	42±0.1	3.12±0.01	30.12 ±0.03	
0.03	1.854±0.004	4.16±0.002	29.727 ±0.02	0.0032±0.0004	29.72 ±0.02	10.441±0.001
0.05	2.544±0.001	2.34±0.001	24.495 ±0.01	0.0033±0.0004	24.49±0.01	7.609±0.002
0.07	3.275±0.001	2.22± 0.002	24.253 ±0.06	0.0036±0.0004	24.25±0.02	5.911±0.002
0.09	5.331±0.002	1.77± 0.002	20.348± 0.01	0.0032±0.0002	20.34 ±0.01	3.631±0.002

Table 2. Displays all the characteristics of drug that were derived from surface tension measurements of TX-100. Banjare et.al. [24]^a

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 [24]. 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.

$$\eta_0 = \eta \dots\dots\dots(4)$$

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 TX-100-drug setup and, as a consequence, accepted the changes. In the same way, the (0.03M, 0.05M, 0.07M and 0.09M) concentrations of the TX-100-drug setup have been examined in a coming up break and trouble-free relative viscosity bend than at least concentrations [24].

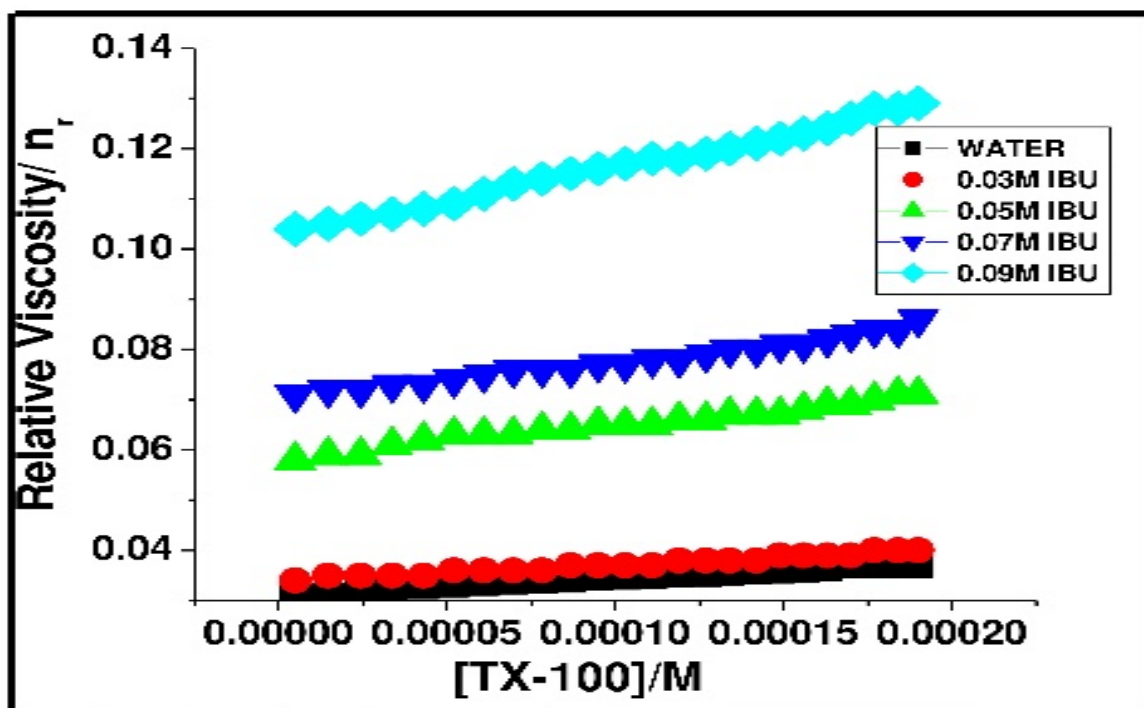


Figure 3 : Graph plots between relative viscosity versus concentration of surfactant (M) in the presence of IBU [0.03,0.05,0.07and 0.09M] at 298 K.

3.3 FTIR study of TX-100 and NSAIDs [IBU] 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 IBU	TX-100+IBU
O-H stretching vibration	3854	3783 3679 3092	3472.20
Phenols	3587.12	-	-
Bonded stretching of amines and amides	3373-3422	-	
C-H Symmetric and asymmetric stretching bands	2918.2-2954	2952	2867.51
Carboxyl acid	2500-3300	-	-
C-N	2322.8-2138.1	-	
Silicon compounds	2047.30	-	
Ketones	1733.59	1708	1609.71,
Alkanes	1405-1445	-	1458.67
C-O/C-H bending	1421-1415	1422	-
C-O	1382-1036	-	1357.56
Alkyl ketone	1215-1325	-	1100.56,
Alkyl amine	1020-1220		-
Vibration of the C-O in the alcohol hydroxyl group	1026	1006	
Alkyl halides	469	860	944.25, 830.55

Table 3. IR Absorption bands of conventional surfactant (TX-100) and IBU.

IR spectra of ibuprofen can be studied by using the FTIR Technique. The NSAIDs of pure ibuprofen spectra are shown in Table 3 and Fig. 4 (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 IBU. The sharp peak [O-H] bending is observed at

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1231 cm^{-1} for IBU. The carboxylic and ester group with [C-O] stretching bands are observed at 1708 cm^{-1} for IBU. The aromatic methylene ring containing [C-H] bending is observed at 1422 cm^{-1} for IBU. The [C-O] stretching band is observed at 1006 cm^{-1} for IBU. The aromatic ring [C-C] bending spectra are observed at 860 cm^{-1} for IBU.

IBRUFEN + TX-100 was observed, The carboxylic group containing [O-H] stretching (broad) bands is observed at 3472 cm^{-1} the broad band is the C-H BOND observed at 2867.51 cm^{-1} . Functional group Ketones were observed at 1609.71 cm^{-1} , C-O/C-H bending band is not observed, C-O band observed as 1357.56 cm^{-1} , Alkyl ketone band observed at 1100.56 cm^{-1} . The alkyl halides group observed at 944.25 and 830.55 cm^{-1}

a) IR spectra of the mixed TX-100 + IBUPROFEN system.

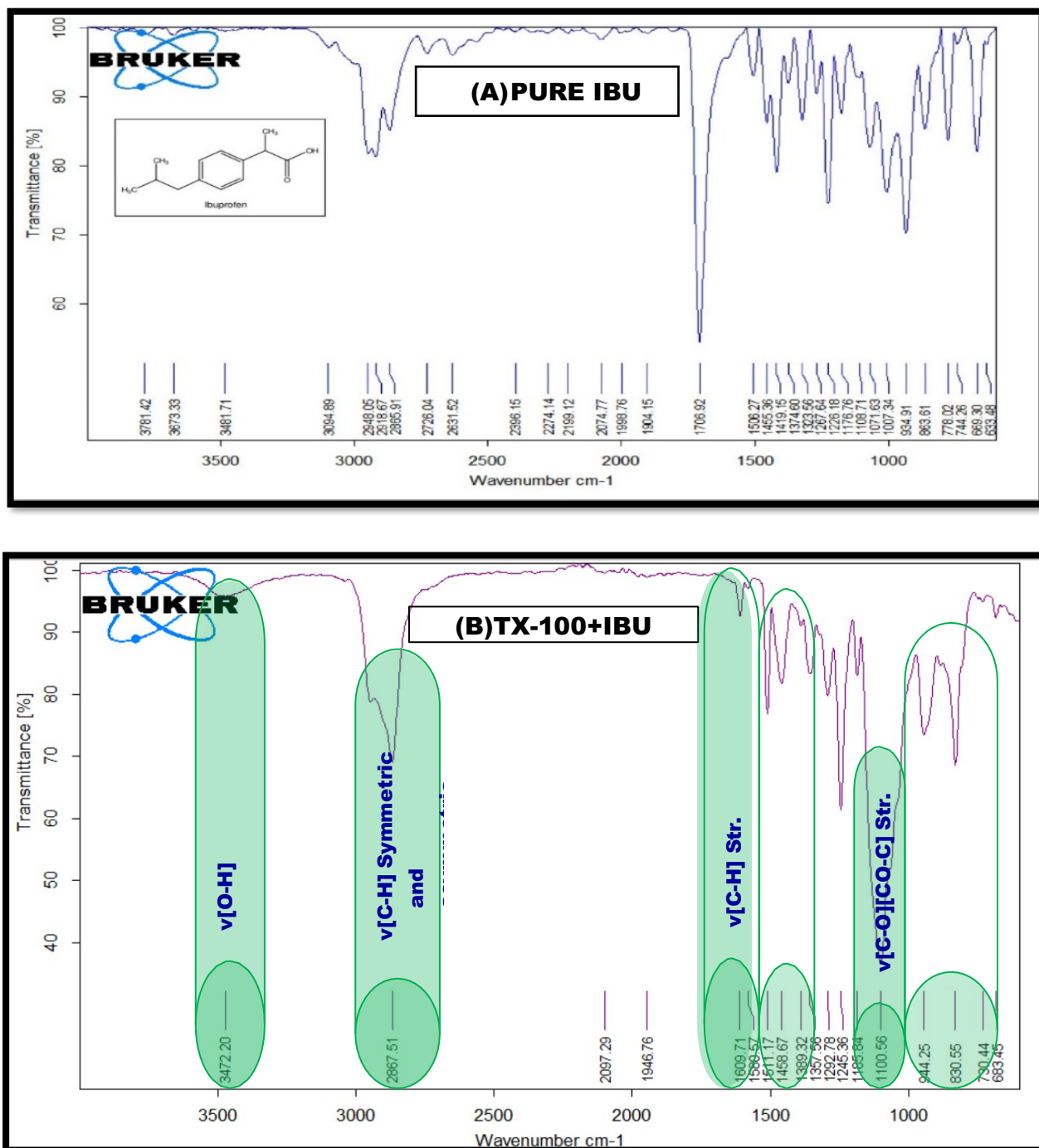


Figure 4: FTIR spectra of different conventional Pure IBU drugs and TX-100+IBU SYSTEM.

3.4 ¹H 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 (TX-100) as well as drug (IBU) undergo a chemical shift in ¹H-NMR spectra as an outcome of the molecular coupling between surfactant (TX-100) with Ibuprofen [24].

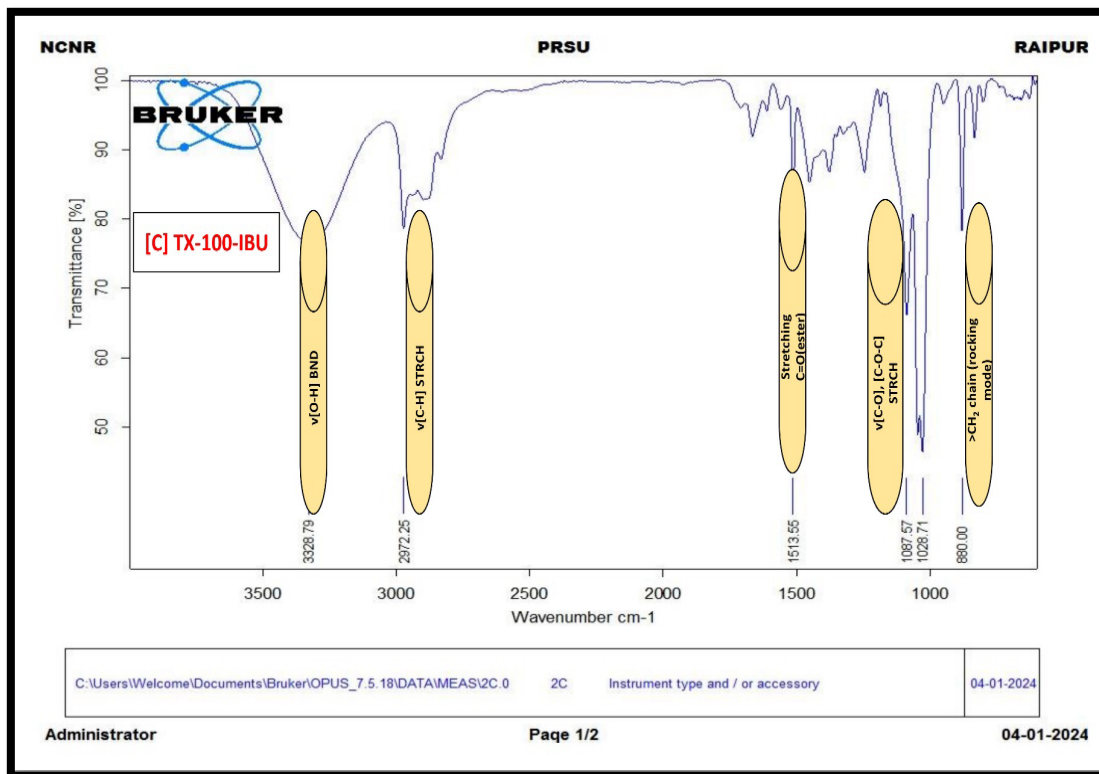


Figure 5: ¹H NMR spectra of the mixed system, i.e., TX-100-IBU.

Proton Signal (¹ H-NMR)	IBU	TX-100+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

Table 4. Transform in chemical shift (δ ppm) of the H of Ibuprofen molecule when complexed with surfactant, i.e., TX-100, molecules in DMSO solvent at 300K.

Therefore, NMR has been used extensively in pharmaceuticals to investigate the structure of surfactants, larger molecules, and complexation between globular proteins and metallic nanoparticles, medications, and other applications. The tiny hydrogen molecule near the IBU 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 IBU was carefully examined. The TX-100+IBU 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 IBU in water solution. The current work assessed the chemical shifts of IBU protons and examined the impact of surfactants on IBU complexation. The proton NMR spectra of 0.1M IBU in DMSO solution with surfactants are displayed in Figure 5, and Table 4 shows all of the ¹H-NMR data. The addition of three surfactants causes the Ha signals of the IBU's Hb to Hg to shift downfield and the Ha of the –CH₃ group on the IBU +ve ion to shift upfield.

4. Conclusions

The study examines the interactions of TX-100 with drugs at room temperature using various measurement tools. It reveals that micelle formation depends on the bonding nature of the drug. The drug adsorbs onto the outer surface of the micelle, reducing electrostatic forces and decreasing the CMC value as the drug concentration increases, depending on the drug's nature. Micelle formation is enhanced in aqueous media but delayed with changes in drug concentration. The free energy of micellization in TX-100-drug mixtures is perceived to be negative, indicating spontaneous processes. In most cases, micellization is exothermic, with exceptions at different concentrations at room temperature. The molecular interaction order and CMC value of the TX-100-drug system are tailored. The study reveals that adsorption is the primary phenomenon, with micellization being secondary. Hydrophobic/Hydrophilic interactions may exist between TX-100 and the drug. The relative viscosity graph is noted by using a viscometer. The FTIR data showed that the TX-100 + IBU system has good agreement and has been utilized as a cosolvent for the entire system, which can help bind TX-100 and NSAIDs. In agreement with the FTIR, it was shown that TX-100 with IBU causes compositional changes. Consequently, we may say that using an appropriate IBU 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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