

VISCOSITY, UV AND FTIR STUDY OF TERNARY MIXTURES OF DCFC, DMSO AND ALKANOLS AT 303.15 K TEMPERATURE WITH SYNTHESIS AND CHARACTERIZATION OF DCFC

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ABSTRACT

In this work, we report a combined experimental viscosity, UV and FTIR of ternary mixtures of 6, 8-dichloro-3-formylchromone (DCFC)+ dimethyl sulfoxide (DMSO) + tertiary butyl alcohol (TBA) and 6, 8-dichloro-3-formylchromone (DCFC)+ dimethyl sulfoxide (DMSO) + ethyl alcohol (ET) at 303.15 K as well as synthesis and characterization of DCFC. Viscosity data over the entire composition range have been used to compute percentage (%) of alkanols. For DCFC + TBA + DMSO, minimum viscosity is about 90 % while maximum at 0 % and for DCFC + ET + DMSO; minimum viscosity is observed at 20 % while maximum at 100 %. FTIR study implies that ν_{OH} and $\nu_{S=O}$ for both mixtures are minimum at 90 % and maximum at 10 % indicating interaction becomes strong as the % of alkanols increases.

KEYWORDS: Viscosity, UV, FTIR, MS, NMR

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INTRODUCTION

We turned towards the synthesis of 3-formylchromones because of their synthetic and pharmaceutical importance and study their molecular interactions with some protic and aprotic solvent mixtures. Natural and synthetic 3-formylchromone derivatives exhibit a wide spectrum of biological activity including anti-pyretic, antiasthmatic, antiallergic, antimicrobial¹, cardiogenic, antiviral, and anticancer properties. Kawasaki et. al.² recorded the biological activity of 3-formylchromones and related compounds. Several 3-formylchromone derivatives have anti-tumor cell-cytotoxicity, anti-Helicobacter pylori, urease inhibitory and anti-HIV activity. 3-formylchromones are versatile antidiabetic, antiobesity³, anticonvulsant⁴ and antibacterial agents⁵.

The viscosity study provides information on the liquids which are required in the design of processes, which involves mass transfer, fluid flow etc ⁶. Thermodynamic properties of the composition are very useful tools to understand the nature and molecular aggregation due to intermolecular interaction between components ^{7, 8}. Viscosity is one of the striking characteristics of various liquid is the differences in their rates of flow. The derived parameters from these measurements are important for solubility measurements.

DMSO is less toxic than other members of this class, such as dimethyl formamide, dimethylacetamide, N-methyl-2-pyrrolidone. It is also extensively used as an extractant in biochemistry and cell biology 9. ET is a versatile solvent, miscible with water and with many organic solvents. It is a powerful psychoactive drug and one of the oldest recreational drugs. Ter-butyl alcohol (TBA) is a clear, noncorrosive liquid. It is miscible with water as well as most common organic solvents and forms azeotrope. Aprotic solvent, DMSO tend to have large dipole moments (separation of partial positive and partial negative charges within the same molecule) and solvate positively charged species via their negative dipole. Protic solvents like water or alkanols solvate anions (negatively charged solutes) strongly via hydrogen bonding.

It is well known that the energy of the H-bond depends on the H...Y distance and the Y...H-X angle, where X is a H-donor and Y is H-acceptor¹⁰. However, when the bond is longer than 1.6 Å, the strength of the bond depends less on the value of the Y...H-X angle¹¹. So our concentration was mainly on the length of H-bonds which are longer than 1.6 Å.

Spectroscopy is important technique by which the energy differences between the allowed states of a system are mapped and recorded by determining the frequencies of the corresponding light absorbed.

In the present paper, DCFC has synthesised and characterized by ¹HNMR, UV, IR and Mass spectroscopy. Similarly, ternary mixtures of DCFC+TBA+DMSO and DCFC+ET+DMSO have been studied with UV and FTIR methods.

Experimental Details

Synthesis of 6, 8-Dichloro -3-Formylchromone

11.47 g (121 ml) DMF was taken in dry R. B. flask, and then 7.513 g (0.49 mol.) of POCl₃ was added slowly with intensive stirring at about 50 °C. Heating and stirring were continued for two hrs at 50-60 C. The solution of 1-(3, 5-dichloro-2-hydroxyphenyl) ethanone 2.46 g (0.12 mol.) in DMF (20 ml) was then added drop by drop under stirring at about 50-60 C. The stirring continued for 2 hrs at 55-65 °C. The reaction mixture was then cooled and stirred over night at room temperature. Reaction mixture was diluted by adding 20 g crushed ice and cold water and stirred again for 6 hrs. The brown yellow solid of 6, 8-dichloro-4-oxo-4*H*-chromene-3-carbaldehyde was then filtered and recrystallized by ethyl acetate. Melting point was in the range 172-174 °C. The reaction was monitored by TLC and benzene was used as a solvent. The recrystallized product characterized by U.V., N.M.R. and Mass spectroscopy.

Reaction

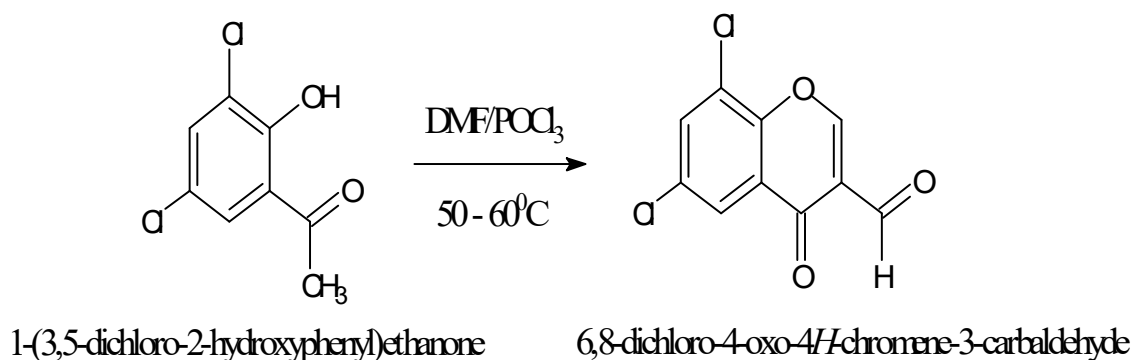


Figure 1

Spectral Data for 6, 8-Dichloro-4-oxo-4H-Chromene-3-Carbaldehyde Recorded

¹H NMR (CDCl₃): 10.35, 8.59, 8.17, 8.16, 7.80, 7.79, 7.26, and 1.58 δ.

MS: m/z 41, 53, 62, 74, 97, 109, 123, 132, 144, 160, 172, 188, 197, 213.95 (base peak), 242, 261, 287, 298.

UV λ_{max}, nm: 357 (0.446), 339 (0.420), 312 (0.320).

IR cm⁻¹: 3439.19 (O-H), 2997.48 and 2912.61 (C-H), 1666.55 (C=O), 1057.03 (S=O).

Ternary mixtures were prepared by mixing known masses of each liquid in airtight-stoppered glass bottles. The measurements were made on a digital balance (SHIMADZU, AUX 220) to an accuracy of ±1×10⁻⁴ mg. The more volatile component (ET) was filled directly into the bottle and the closed bottle was weighed. The other component was injected into the bottle through the stopper by means of a syringe. This method allowed negligible vapour loss and contamination. The possible error in mole fraction is calculated to be less than ±1×10⁻⁴. The preferential evaporation of solvents from the mixture was kept to a minimum as evidenced by reproducible measurements of the physical properties of these liquid mixtures over a time interval of 1 or 2 days.

A single capillary pycnometer (specific gravity bottle) was chosen for the present work^{12, 13}. The pycnometer was washed thoroughly with fresh chromic acid to remove impurities. Subsequently the pycnometer was washed with distilled water and then with acetone and dried with a stream of warm air from hot blower. It was then kept in an oven at about 60 °C for half an hour. The weights of empty pycnometer and pycnometer filled with experimental liquids were taken on SHIMADZU (AUX 220) balance having a sensitivity of 0.01 mg. The pycnometer was calibrated with freshly prepared triply distilled water.

The density measurements using mass/volume relationship for each experimental liquid were repeated at least three to four times and the results averaged. This procedure enabled us to get an uncertainty of ± 0.0005 g cm⁻³ in density measurements.

In the present study, the capillary flow method was employed to determine the viscosity of fluid at room temperature. The usual form of this simple, yet accurate, apparatus, an Ostwald viscometer was used for calculating viscosities of different liquids.

The viscosity values were determined using the relation:

$$\eta = \rho (at - b/t)$$

Where η is the viscosity, ρ is density of the liquid, t is flow time, a and b are the constants for a given viscometer.

The viscometer was calibrated with triply distilled water. The constants a and b were obtained by measuring the flow times of triply distilled water at experimental temperatures.

THEORY

FTIR Measurements

FTIR spectra were recorded on a FTIR spectrometer (SIMADZU 8400S PC) in the region 400 -4000 cm⁻¹ with 4.0 cm⁻¹ resolution.

Ultraviolet Measurements

U.V. spectra were recorded on a U.V. 2400 PC in the wavelength range 200-900 nm. The scan speed is fast and sampling interval is about 2.0. The U.V. measurements are reported directly in nm. On passing electromagnetic radiation in the U.V. regions through a compound with multiple bands, a portion of the radiation is normally absorbed by the compound. The amount of absorption depends on the wavelength of the radiation and the structure of the compound. Ultraviolet spectrum recorded the wavelength of an absorption maximum i.e. λ_{\max} .

RESULTS AND DISCUSSION

Experimental densities (ρ) and viscosity (η) values of pure liquids are given in Table 1. The viscosities for the ternary mixtures of DCFC+DMSO+TBA and DCFC+DMSO+ET at 0.001m and 0.01m concentrations and at atmospheric pressure are listed in Table 2. Polynomial equations of these ternary mixtures with correlation coefficient (R^2) are listed in the Table 3. FTIR frequencies i.e. ν_{OH} and $\nu_{\text{S=O}}$ are given in the Table 4.

Viscosity Study

DCFC + TBA + DMSO (0.001 & 0.01m; Figures 2 and 3) viscosity up to 50 % suddenly decreased and then no drastic change is seen. Minimum viscosity is about 90 % while maximum at 0 %. The observed decreased viscosity up to 50 % indicating solute (DCFC) weakens dipole-dipole forces or dispersion or breaks strong H-bonding between solvent molecules and then DCFC forms H-bonds with these solvent molecules hence no drastic change is observed after 50 % in viscosity.

While in DCFC + ET + DMSO (0.001m and 0.01m; Figs. 1 and 2) systems, nature of the curve is obtained as a power type and change in viscosity is very small upto 40 %, thereafter exponentially increased upto 100 % suggesting H-bonding becomes strong between solute and solvent molecules with increase in % ET. Minimum viscosity is observed at 30 % while maximum at 100 %.

It is also revealed that among these systems maximum interaction follows the order as: DCFC + TBA + DMSO (0.01m) > DCFC + TBA + DMSO (0.001m) > DCFC + ET + DMSO (0.001m) > DCFC + ET + DMSO (0.01m).

Table 1: Experimental Densities (ρ) and Viscosities (η) of Pure Liquids at 298.15, 303.15 and 308.15 K

Liquids	Temperature	$\rho \times 10^{-3} \text{ (kg m}^{-3}\text{)}$		$\eta \times 10^3 \text{ (kg m}^{-1}\text{s}^{-1}\text{)}$	
		Experimental	Literature	Experimental	Literature
ET	298.15	0.78522	0.785 22 ^a	1.109	1.109 ^l
	303.15	0.78054	0.78050 ^b	0.986	0.986 ^j
	308.15	0.76387	0.76391 ^c	0.768	0.767 ^c
TBA	298.15	0.78123	0.78120 ^d	4.3839	4.372 ^d
	303.15	0.77541	0.77540 ^e	3.3787	3.378 ^g
	308.15	0.7702	0.7702 ⁱ	-	-
DMSO	298.15	1.0955	1.0955 ^g	1.9971	1.997 ^l
	303.15	1.0901	1.0896 ^h	1.8303	1.830 ^k
	308.15	1.0856	1.08560 ⁱ	1.6138	1.614 ^e

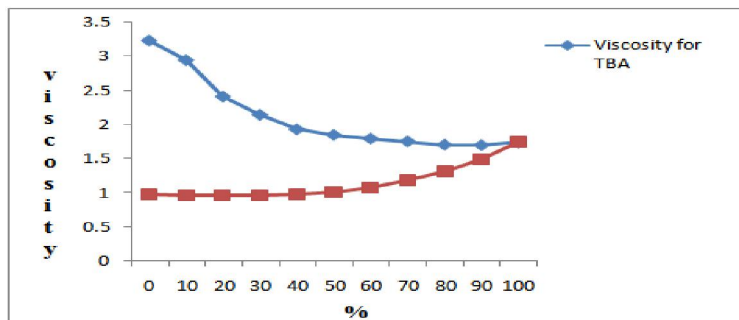


Figure 2: % Vs η for DCFC + % TBA+DMSO and DCFC+ % ET+DMSO (0.001 m).

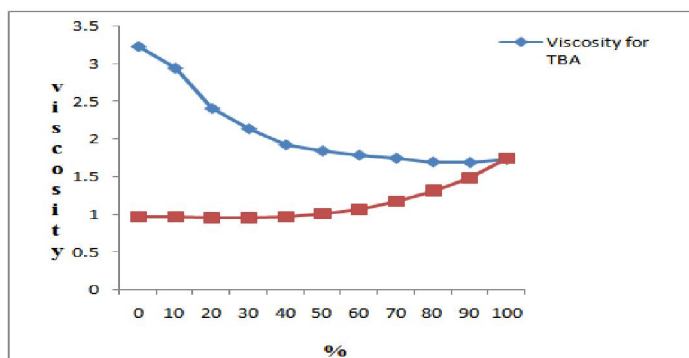


Figure 3: % Vs η for DCFC + % TBA+DMSO and DCFC+ % ET+DMSO (0.01 m).

Table 2: % Alkanols and Viscosities (η) of Ternary Mixtures at Temperature 303.15 K.

η for DCFC+ % TBA+DMSO (0.001m)		η for DCFC+ % TBA+DMSO (0.01m)	
% TBA	η	% TBA	η
0	3.213	0	3.231
10	2.906	10	2.945
20	2.398	20	2.409
30	2.096	30	2.141
40	1.884	40	1.928
50	1.797	50	1.846
60	1.760	60	1.792
70	1.739	70	1.751
80	1.720	80	1.701
90	1.724	90	1.695
100	1.752	100	1.735
η for DCFC+ % ET+DMSO (0.001m)		η for DCFC+ % ET+DMSO (0.01m)	
% ET	η	% ET	η
0	0.984	0	0.967
10	0.938	10	0.965
20	0.935	20	0.959
30	0.938	30	0.957
40	0.973	40	0.976
50	0.992	50	1.007
60	1.081	60	1.069
70	1.161	70	1.179
80	1.291	80	1.319
90	1.476	90	1.490
100	1.756	100	1.750

FTIR Study

In IR spectroscopy, the main preoccupation of an organic chemist is the region 4000-650 cm^{-1} . Absorption of an IR light can occur only if the dipole moment of the molecule is different in the two vibration levels. The stretching frequency of chemical bond depends upon bond strength and reduced masses of the atoms forming bond. Any factor which will increase bond strength will increase stretching frequency of the bond, and if the mass of atoms forming the chemical bond is increased, the reduced mass will increase and stretching frequency will decrease. FTIR of alcohols have characteristic absorptions associated with both the $\nu\text{O-H}$ and the $\nu\text{S=O}$ vibrations. These appears in the regions 3500-3200 cm^{-1} (a very intense, broad band) and 1100-1000 cm^{-1} , respectively.

Table 4 includes νOH for two ternary complexes. The OH stretching vibrations of TBA and ET occur around 3416 and 3410 cm^{-1} , respectively. There is a decrease in the normal νOH from 3416 to 3377.47 cm^{-1} up to 90 % of TBA in DCFC+TBA+DMSO and from 3410 to 3346.61 cm^{-1} up to 90 % of ET in DCFC+ET + DMSO systems.

For DCFC + TBA + DMSO, $\nu\text{O-H}$ steadily decreased. Minima at 90 % while maximum at 10 % are noted. For DCFC+ET+DMSO, $\nu\text{O-H}$ exhibited nonparticular trend minimum at 90 % (3346 cm^{-1}) and maximum at 10 % (3398 cm^{-1}).

In this work, strong absorption bands are seen for DCFC+TBA+DMSO as compared to DCFC+ET+DMSO. This throws light on stronger H-bonding present in DCFC+ET+DMSO than DCFC+TBA+DMSO system. The reason may be due to bulky nature of TBA. It also supports to the viscosity study of these two ternary complexes.

$\nu\text{S=O}$: For both systems, no particular trend of this frequency was observed. For DCFC + TBA + DMSO, $\nu\text{S=O}$ absorbed between 1045-1058 cm^{-1} (Table 4) with no particular trend over entire composition (maxima at 20 and 30 % while minimum at 90 %). For DCFC+ET+DMSO, $\nu\text{S=O}$ appeared between 1049-1089 cm^{-1} (Table 4), indicating strong H-bonding between oxygen of S=O and hydrogen of OH than that of DCFC + TBA + DMSO.

Table 3: Polynomial Equation Results for Ternary Mixtures with Correlation Coefficient (R^2).

Sr. No.	System	Polynomial Equation	R^2
1	DCFC+ % TBA+DMSO (0.001m)	$y = -5\text{E-}11x^6 + 2\text{E-}08x^5 - 2\text{E-}06x^4 + 0.000x^3 - 0.003x^2 - 0.008x + 3.215$	0.999
2	DCFC+ % TBA+DMSO (0.01m)	$y = -6\text{E-}11x^6 + 2\text{E-}08x^5 - 3\text{E-}06x^4 + 0.000x^3 - 0.004x^2 - 0.004x + 3.234$	0.998
3	DCFC+ % ET+DMSO (0.001m)	$y = 8\text{E-}12x^6 - 2\text{E-}09x^5 + 3\text{E-}07x^4 - 2\text{E-}05x^3 + 0.000x^2 - 0.008x + 0.984$	0.999
4	DCFC+ % ET+DMSO (0.01m)	$y = 9\text{E-}12x^6 - 2\text{E-}09x^5 + 3\text{E-}07x^4 - 1\text{E-}05x^3 + 0.000x^2 - 0.001x + 0.968$	0.999

Table 4: νOH and $\nu\text{S=O}$ (cm^{-1}) for DCFC + % alkanols + DMSO (0.001m) systems.

% DCFC + % TBA + DMSO	νOH		$\nu\text{S=O}$	
	DCFC + % TBA + DMSO	DCFC + % ET + DMSO	DCFC + % TBA + DMSO	DCFC + % ET + DMSO
10	3419.10 (vw)	3398.69 (vw)	1055.10 (s)	1057.03 (s)
20	3406.40 (w)	3387.11 (w)	1058.96 (s)	1055.10 (s)
30	3406.40 (m)	3392.90 (w)	1058.96 (s)	1051.24 (s)
40	3410.26 (m)	3389.04 (m)	1055.10 (s)	1087.89 (vw), 1049.31 (s)
50	3408.33 (m)	3389.04 (m)	1051.24 (m)	1087.89 (vw), 1049.31 (s)
60	3408.33 (s)	3377.47 (m)	1049.31 (m), 1026.16 (w)	1087.89 (vw), 1049.31 (s)
70	3402.54 (s)	3360.11 (s)	1047.38 (m), 1028.09 (m)	1087.89 (w), 1049.31 (s)
80	3389.04 (vs)	3352.39 (vs)	1047.38 (w), 1022.31 (m)	1089.82 (m), 1049.31 (s)
90	3377.47 (vs)	3346.61 (vs)	1045.45 (w), 1022.31 (m)	1089.82 (m), 1049.31 (s)
100	3375.54 (vs)	3342.75 (vs)	-	-



Figure 4: Experimental FTIR νOH (cm^{-1}) in: A) DCFC+10 % TBA+DMSO; B) DCFC+50 % TBA+DMSO; C) DCFC+90 % TBA+DMSO; D) DCFC+10 % ET+DMSO; E) DCFC+50 % ET+DMSO; F) DCFC+90 % ET+DMSO; FTIR $\nu\text{S}=\text{O}$ (cm^{-1}) in: G) DCFC+10 % TBA+DMSO; H) DCFC+50 % TBA+DMSO; I) DCFC+90 % TBA+DMSO; J) DCFC+10 % ET+DMSO; K) DCFC+50 % ET+DMSO; L) DCFC+90 % ET+DMSO.

UV Study

UV absorption spectra of a molecule are highly dependent on the polarity of the solvent used. The intensity, position, and shape of the absorption bands often change with the polarity of the solvent. This is because, with changing the polarity of the medium, the intermolecular interactions of the chromophores are altered, leading to a change in the energy difference between the ground state and the excited state. This solvent effect on the spectra of the compound, resulting in a hypsochromic (or blue) shift with increasing solvent polarity, is usually called negative solvatochromism. The corresponding bathochromic (or red) shift is termed positive solvatochromism. From a literature survey, the UV-visible study provides a new opportunity to investigate how a solute can be preferentially solvated through specific and non-specific interactions. Preferential solvation is due to the different solute-solvent and solvent-solvent interactions. Hydrogen bonding is the major factor for preferential solvation. There are three parameters: solvent polarity-polarizability, H-bond donation ability, and H-bond acceptor capability, which differ in solute-solvent interaction.

In the present paper, in DCFC + TBA + DMSO (Table 5), at 10%, 50%, and 90% ($\lambda_{\text{max}} = 347.5, 346.5$ and 345.5 nm and absorbance - 0.859, 1.066 and 1.099 respectively). For DCFC + TBA and DCFC + DMSO mixtures, λ_{max} values are observed at 344.5 and 345.5 nm, while absorbance is 1.273 and 1.425, respectively.

For DCFC+ET+DMSO (Table 6), 344 nm (10%), 321 nm (50%) and 308.5 nm (90%) obtained with their absorbances are 1.532, 0.337 and 1.211. In DCFC + ET and DCFC + DMSO, $\lambda_{\text{max}} = 344$ nm ($A = 0.581$) and $\lambda_{\text{max}} = 348$ nm ($A = 0.825$).

In the present investigation, the maximum wavelength is found in DCFC+TBA+DMSO than that of DCFC+ET+DMSO, suggesting that $n \rightarrow \pi^*$ transition requires less energy for DCFC+TBA+DMSO as compared to that of DCFC+ET+DMSO, representing that strong hydrogen bonding in DCFC+ET+DMSO as compared to that of DCFC+TBA+DMSO. This supports the viscosity and FTIR studies.

Table 5: The Values of λ_{max} and Absorbance of DCFC + % TBA + DMSO

%	λ_{max}	Absorbance
DCFC + 10 % TBA+ 90 % DMSO	347.5	0.859
	317.5	0.933
	232.5	3.763
DCFC +50 % TBA+ 50 % DMSO	346.5	1.066
	317.0	1.136
	232.5	3.913
DCFC + 90 % TBA+10 % DMSO	345.5	1.099
	306.5	1.435
	221.5	4.000
DCFC + 100 % TBA	344.5	1.273
	305.5	2.039
	230.5	3.675
DCFC +100% DMSO	345.5	1.425
	305.0	2.060

Table 6: The Values of λ_{max} and Absorbance of DCFC+ % ET+DMSO

%	λ_{max}	Absorbance
DCFC +10 % ET +90 % DMSO	344.0	1.532
	305.0	2.104
	239.0	3.913
DCFC +50 % ET + 50 % DMSO	321.0	0.337
	251.0	0.441
DCFC + 90 % ET +10 % DMSO	308.5	1.211
	232.0	3.612
DCFC + 100 % ET	344.0	0.581
	318.5	0.825
	309.5	0.831
DCFC + 100 % DMSO	348.0	0.825
	318.0	0.852
	224.0	3.913

CONCLUSIONS

Finally, it has been concluded that viscometric and spectroscopic investigations indicate that the presence of dispersion forces, dipole-induced dipole and dipole-dipole interactions between the components of the system. Presence of hydrogen bonding between OH of ET/TBA with oxygen of C=O group of DCFC and between hydrogen of DCFC with oxygen of S=O group of DMSO.

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