Asian Journal of Chemistry; Vol. 33, No. 6 (2021), 1403-1408

ASIAN JOURNAL OF CHEMISTRY

https://doi.org/10.14233/ajchem.2021.23146

Concentration and Temperature Dependence of the Thermodynamic Properties of Novel Biologically Active 3-Substituted Schiff Base of 4-Piperidyl *N*-(4-chlorophenyl)maleimide

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Received: 31 January 2021; *Accepted*: 26 April 2021; *Published online*: 5 June 2021; AJC-20375

In present work, the concentration and temperature dependence of the thermodynamic properties of 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide compound in 80% DMSO was estimated. Concentration (0.002-0.01 M) and temperature (298-313 K) dependent densitometric and viscometric measurement were employed to evaluate limiting molar volume (ϕ_v^0), semi-empirical parameter (Sv), Falkenhagen (A) and Jones-Dole (B) viscosity coefficient. The obtained results suggest the presence of weaker solute-solvent interactions and stronger solute-solute interactions. It was observed that these interactions strongly depend on the temperature of the system. Furthermore, the Gibbs free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) of the system were also evaluated. The negative values of ΔG and ΔH and positive values of ΔS indicating reaction was spontaneous and exothermic in nature.

Keywords: Density, Viscosity, Interactions, Thermodynamic property, Maleimide derivatives, Schiff base.

INTRODUCTION

The physical properties of the substances are the properties that can be measurable and observable without accompanying any change in the chemical composition of substance [1]. Some basic physical properties are colour, melting point, boiling point, density, *etc*. They are useful for both quantitative and qualitative analysis of the sample. The quantifiable properties are called as a physical quantity. The value of physical quantity represents the physical state of matter and change in its value gives information about the change in state of the substance. Based on the concentration dependency physical properties are mainly classified as extensive and intensive properties. Extensive properties are dependent on the concentration or amount of substance *viz.* mass, volume, entropy hence also termed as additive properties. However, intensive properties are independent of concentration or amount of substance as well as the size of the system. Some examples of intensive properties are density, viscosity, refractive index, physical constant and conductivity. An intensive property is an important class of properties and from a chemistry point of view measurement of intensive property is more advantageous than extensive property [2]. Among various intensive properties density and viscosity has much more importance. Furthermore, form their results, the thermodynamic parameters by using a simple mathematical equations can be estimated.

In literature, various reports are available where researchers measured the physical properties of a large number of organic compounds. Based on the experimental results they have investigated the nature and strength of molecular interactions and interpreted different useful properties of the compounds [3-8]. The measurement of the physical properties of heterocyclic organic compounds gives important information about its stability and reactivity. They are useful to investigate the strength and nature of the solute-solvent/solute-solute interactions. It can also provide information about the feasibility of chemical reaction and helps to estimate the equilibrium chemical composition of the reaction. Thermodynamic study with high precision can be useful to predict the relationship between thermodynamic parameters of organic compounds and their molecular structure [9].

Recently, we have reported the synthesis and antimicrobial activity of novel Schiff bases of 3 & 4-substituted *N*-aryl maleimides [10]. It was observed that Schiff bases of maleimide derivative acts as an efficient biological agent and may be due to the combined synergistic effect. Although, in literature, several reports are available on the thermodynamic properties of organic compounds [11-13]. But no reports are available on the thermodynamic study of Schiff bases of maleimide substituted derivatives. Therefore, in this work, the concentration and temperature dependence of the thermodynamic properties of novel biologically active 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide is conducted. The thermodynamic parameters such as Gibbs free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) of the proposed compound were evaluated from density (ρ) and viscosity (η) measurement in 80% DMSO. The densities and viscosities were also employed to evaluate apparent molar volume (ϕ_v), limiting apparent molar volume (ϕ_v^0), experimental slope (Sv) and viscosity coefficient (A and B). The evaluated values were used to explain the strength and nature of solute-solute and solute-solvent interactions and predict the feasibility of the reaction.

EXPERIMENTAL

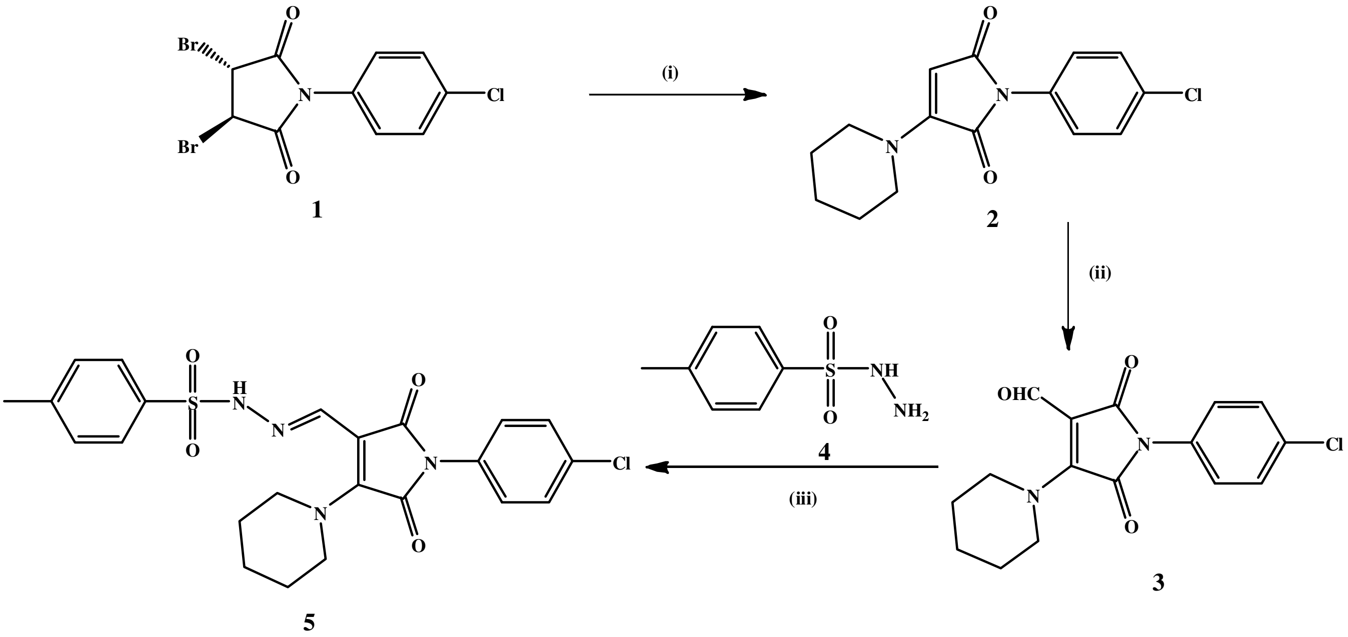
Ethyl alcohol, toluene and acetone were purchased from Sd-fine chemicals, India. Dimethyl sulphoxide (DMSO) and *N,N*-dimethylformamide (DMF) were purchased from Merck Chemicals, India. Before use, all the solvents were purified by distillation following the standard method [14].

Synthesis of 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide: 3-Substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide derivative was synthesized by the reported method [10]. In brief, 5 mL ethanolic solution of 3-substituted aldehyde of 4-piperidyl *N*-(4-chlorophenyl) maleimide (0.01 mol) was prepared with the addition of a small amount of acetic acid (0.005 mol). To the above solution 0.01 mol *p*-toluenesulfonyl hydrazide was added slowly under constant stirring at room temperature. The initial yellowish colour of the solution changes to transparent reddish. After ~20 min orange-red crystals were obtained. The product was separated by filtration at the suction pump using Büchner funnel. The product was washed with purified cold ethanol. The shiny orange crystals of the product were purified by recrystallization using ethanol. The synthetic procedure is schematically represented in **Scheme-I**.

Preparation of stock solutions of 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide: The solutions of purified 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide compounds were prepared before the measurement. A 0.05 M stock solution of synthesized compound was prepared by dissolving accurately weighed compound in 80% aqueous DMSO solution in a calibrated volumetric flask. From the stock solution, the solutions of required concentrations *viz.* 0.002-0.010 M for each compound was prepared.

Measurement of density: Bicapillary pycnometer having a bulb capacity of ~11 mL and the inner capillary diameter of ~1 mm was used. The temperature-dependent density measurement was carried in a transparent walled thermostatic water bath with temperature sensitivity ± 0.05 ºC.

To calculate apparent molar volume at 298, 303, 308 and 313 K graph of height (h) against the volume of the water (ν) at respective temperature was plotted. The apparent molar volume was derived by using slope and intercept values and formula volume (ν) = (height × slope) + intercept whereas density was measured by using formula $\rho = w/\nu$.



Scheme-I: Reagent and conditions; (i) Piperidine, DMF, 0-10 °C, 30 min; (ii) DMF, $POCl_3$, 0-5 °C, 30 min; (iii) Ethanol, acetic acid, 30 min

Measurement of viscosity: The viscosity measurement of the synthesized compound was performed by the capillary method using the Ubbelohde viscometer. The viscosity of the synthesized compounds was measured at different concentrations by varying temperature with 5 ºC interval. For temperature-dependent measurement, the temperature of the thermostat was increased and measurement was done after 30 min of attaining temperature.

By knowing flow time, the viscosity was calculated using the following equation (eqn. 1):

$$\eta = \rho\left(at - \frac{b}{t}\right) \tag{1}$$

Eqn. 1 can be rearranged as:

$$\eta \times \frac{t}{\rho} = at^2 - b \tag{2}$$

where, t = flow time (efflux time), ρ = density of the solution, a and b = constants or viscosity coefficient.

In the present study, for measurement of densities and viscosities of synthesized compounds in water, DMSO and 80% aq.s DMSO were used as solvents. Therefore, calibration was also checked for the same and the results are summarized in Table-1. It was noticed that both viscosity and density measurement show a good correlation in experimental and literature value.

TABLE-1
EXPERIMENTAL AND REPORTED VISCOSITIES (η_o) AND DENSITIES (ρ_o) OF WATER, PURE DMSO AND 80% DMSO AT 298-313 K

Temp. (K)	Viscosity (cP)		Density (g cm^{-3})	
	Experimental	Reported	Experimental	Reported
Water				
298	0.8909	0.8910	0.9970	0.9971 [16,17]
303	0.7897	0.7980	0.9956	0.99565 [16] 0.99568 [18]
308	0.7164	0.7190	0.9940	0.9940 [16] 0.9941 [18]
313	0.6544	0.6530	0.9922	0.9922 [16] 0.9923 [18]
Pure DMSO				
298	1.9909	1.9910	1.0956	1.0955 [16] 1.0956 [19]
303	1.8049	1.8060	1.0906	1.0904 [16] 1.0906 [20]
308	1.6933	1.6900	1.0855	1.0854 [16] 1.0855 [21]
313	1.5104	1.5110	1.0804	1.0803 [16]
80% DMSO				
298	3.3107	3.3124	1.0962	1.0960 [22]
303	2.9918	2.9921	1.0941	1.0940 [17] 1.0941 [23]
308	2.6747	2.6712	1.0895	1.0895 [17]
313	2.3480	2.3483	1.0851	1.0851 [17]

Determination of thermodynamic parameters: The measured densities and viscosities were used for the calculation of the thermodynamic properties *viz*. Gibbs free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) of the system. The correlation between relative viscosity of liquid and temperature is mathematically given by the Andrade equation [15] (eqn. 3).

$$\eta_r = A.e^{-\Delta G/RT} \tag{3}$$

where, η_r = relative viscosity, A = constant, ΔG = Gibbs free energy, T = temperature of the system, R = gas constant.

Eqn. 2 can be rewritten as:

$$\log \eta_r = -\frac{\Delta G}{2.303RT} + \ln A \tag{4}$$

Gibbs free energy and constant (A) is easily evaluated from the linear plot of log η_r *vs.* 1/T. Intercept gives constant whereas, Gibbs free energy calculated from slope using eqn. 5.

$$\Delta G = -2.303 \times R \times \text{Slope} \tag{5}$$

Furthermore, the relative viscosity is related to enthalpy by eqn. 6:

$$\frac{\log \eta_{r2}}{\log \eta_{r1}} = \frac{\Delta H}{2.303R} \times \frac{T_2 - T_1}{T_1 T_2} \tag{6}$$

Then, using the Gibbs-Helmholtz equation entropy of the system is calculated from eqn. 7:

$$\Delta S = \frac{(\Delta H - \Delta G)}{T} \tag{7}$$

where, ΔS = entropy of the system.

RESULTS AND DISCUSSION

Densities and viscosities of 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide: The densities and viscosities of the synthesized compound were measured at different concentrations by varying temperatures and the results are given in Table-2. Initially, the measurements were performed at constant temperature *i.e.* at room temperature (298 K) by changing concentration in the range of 0.002-0.01 mol L^{-1}. From the experimental data, it was observed that with increasing concentration both the densities and viscosities were also increased. By knowing the density of proposed compound apparent molar volumes (ϕ_v), were calculated from the Masson equation [24] and presented in Table-3. It was found that apparent molar volume was directly proportional to the concentration of the compounds.

Furthermore, the viscosities and densities were measured at different temperatures (298-313 K) for the concentration range (0.002-0.01 mol L^{-1}). The results revealed that with increasing temperature both densities and viscosities were decreases. Similarly, apparent molar volume was also calculated at different temperatures which were observed to be directly proportional to the temperature. Relative viscosity was calculated from observed viscosity at different concentrations, which showed an increasing trend. All these values *viz.* density (ρ), apparent molar volume (ϕ_v), viscosity (η) and relative viscosity (η_r) are tabulated in Table-2.

For a better understanding of the strength and nature of solute-solute and solute-solvent interaction limiting apparent molar volume and semi-empirical parameter (Sv) was evaluated. Limiting apparent molar volume explain solute-solvent interaction whereas semi-empirical parameter describes solute-solute

TABLE-2
DENSITY (ρ), APPARENT MOLAR VOLUME (ϕ_v), VISCOSITY (η) AND RELATIVE VISCOSITY (η_r) OF 3-SUBSTITUTED SCHIFF BASE OF 4-PIPERIDYL *N*-(4-CHLOROPHENYL)MALEIMIDE IN 80% AQUEOUS DMSO AT DIFFERENT TEMPERATURES

Temp. (K)	1/T (K^{-1})	Conc. (mol dm^{-3})	√C	Density (ρ) (g cm^{-3})	ϕ_v (cm^3. mol^{-1})	Viscosity (η) (cP)	Relative viscosity (η_r)	log η_r
298	0.003356	0.002	0.0447	1.1042	-3227.6289	3.4557	1.0438	0.0186
		0.004	0.0632	1.1044	-1421.3398	3.4623	1.0458	0.0195
		0.006	0.0775	1.1045	-819.2434	3.4689	1.0478	0.0203
		0.008	0.0894	1.1046	-519.3355	3.4755	1.0498	0.0211
		0.010	0.1000	1.1047	-337.5663	3.4820	1.0518	0.0219
303	0.00330	0.002	0.0447	1.1007	-2575.755	3.1178	1.0421	0.0179
		0.004	0.0632	1.1008	-1097.316	3.1243	1.0443	0.0188
		0.006	0.0775	1.1010	-610.5972	3.1310	1.0465	0.0197
		0.008	0.0894	1.1011	-362.6675	3.1376	1.0487	0.0207
		0.010	0.1000	1.1012	-211.1676	3.1441	1.0509	0.0216
308	0.003247	0.002	0.0447	1.0952	-2173.581	2.7832	1.0405	0.0172
		0.004	0.0632	1.0953	-893.1305	2.7896	1.0430	0.0183
		0.006	0.0775	1.0955	-473.9626	2.7962	1.0454	0.0193
		0.008	0.0894	1.0956	-259.7893	2.8027	1.0479	0.0203
		0.010	0.1000	1.0958	-134.0389	2.8093	1.0503	0.0213
313	0.003195	0.002	0.0447	1.0901	-1845.889	2.4344	1.0368	0.0157
		0.004	0.0632	1.0903	-730.8157	2.4408	1.0395	0.0168
		0.006	0.0775	1.0904	-362.1962	2.4473	1.0422	0.0180
		0.008	0.0894	1.0906	-176.7346	2.4537	1.0450	0.0191
		0.010	0.1000	1.0907	-66.3791	2.4602	1.0477	0.0202

interaction. In general, higher the value of limiting apparent molar volume and higher will be the solute-solvent interaction and *vice versa* [15]. Similarly, a higher value of semi-empirical parameter indicates stronger solute-solute interaction and *vice versa*. This parameter can be obtained easily from the plot of apparent molar volume (ϕ_v) *vs.* square root of concentration (√C) (Fig. 1).

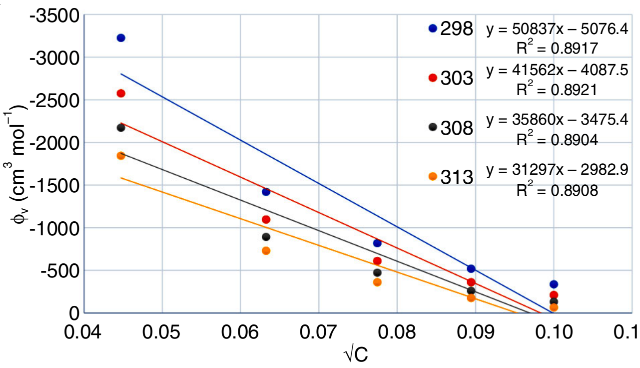


Fig. 1. Plot of ϕ_v *vs.* √C for the 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide at different temperatures (298-313 K)

The slope and intercept of the plot directly gives the semi-empirical parameter (Sv) and limiting apparent molar volume, respectively. The calculated parameters at 298-313 K are shown in Table-3. In present study, it is observed the negative values of limiting molar volume and positive values of semi-empirical parameter indicates weaker solute-solvent interactions and stronger solute-solute interactions. It was noticed that the semiempirical parameter decreases with increasing temperature of the system however limiting apparent molar volume increases. Thus, it is concluded that at a lower temperature (298 K) solute-solute interactions are stronger, however, with increasing temperature of the system this interaction becomes weaker.

TABLE-3
LIMITING APPARENT MOLAR VOLUME (ϕ_v^0), SEMI-EMPIRICAL PARAMETER (S_v) FALKENHAGEN CONSTANT (A) AND JONES-DOLE CONSTANT (B) FOR THE 3-SUBSTITUTED SCHIFF BASE OF 4-PIPERIDYL *N*-(4-CHLOROPHENYL) MALEIMIDE AT DIFFERENT TEMPERATURES

Temp. (K)	S_v	ϕ_v^0 (dm^3 mol^{-1})	A ($dm^{3/2}$ $mol^{-1/2}$)	B (dm^3 mol^{-1})
298	50837	-5076.4	1.295	-8.2193
303	41562	-4087.5	1.2362	-7.697
308	35860	-3475.4	1.1804	-7.1796
313	31297	-2982.9	1.0534	-6.123

Moreover, solute-solvent interactions are weaker at a lower temperature (298 K) and with temperature, it becomes stronger. Thus, the strength and nature of interactions are strongly dependent on the temperature of the system.

Moreover, viscosity data was also used to further confirm the nature and strength of solute-solute and solute-solvent interaction. Viscosity coefficient such as Falkenhagen constant (A) and Jones-Dole constant (B) depicts the solute-solute and solute-solvent interactions, respectively. These constants were calculated by extrapolating relative viscosity (η_r – 1)/C *vs.* square root of concentration (√C). The calculated values of these constants are summarized in Table-3. The plots of relative viscosities (η_r – 1)/C *vs.* square root of concentration (√C) at 298 K-313 K are represented in Fig. 2. The positive values of the Falkenhagen constant (A) further confirmed the presence of stronger solute-solute interaction however negative values of Jones-Dole constant (B) show weaker of solute-solvent interactions. It was found that with increasing temperature Falkenhagen constant (A) decreases and Jones-Dole constant (B) increases. It governs that at a lower temperature (298 K) degree of solute-solute interaction is more whereas solute-solvent interaction is less. Both density and viscosity measure-

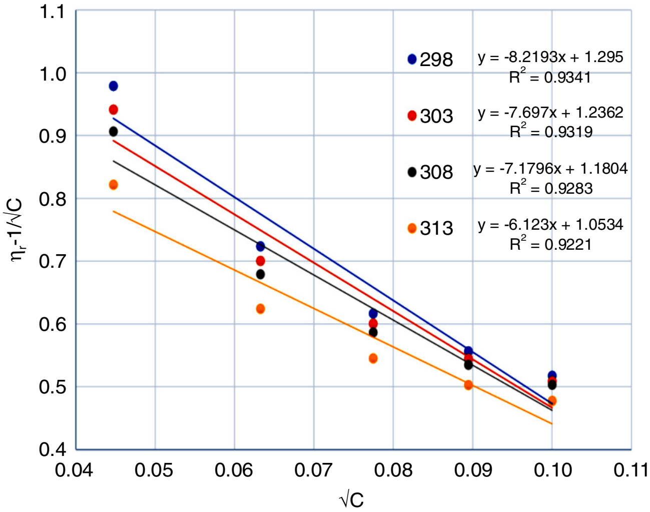


Fig. 2. Plot of η_r-1/√C *vs.* √C for the 3-substituted Schiff base of 4- piperidyl *N*-(4-chlorophenyl)maleimide at different temperatures

ments show a similar trend of solute-solute and solute-solvent interactions. The difference in strength of solute-solute and solute-solvent may be explained from the structure of the solute (synthesized compound) and solvent (DMSO).

Jone-dole coefficient (B) describes the degree of molecular disorder introduced into the proposed compound due to structural modification caused by solute-solvent interactions [24]. It was found that constant B was increased with temperature which reveals that structural modification occurs more at a higher temperature than at a lower temperature.

Thermodynamic properties of 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide: Thermodynamic parameters such as Gibbs free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) of the system were also reported. The negative values of ΔG, ΔH and positive values of ΔS suggest reaction is spontaneous and exothermic in nature (Table-4). With the concentration of solute, all these parameters were found to be decreased indicating the concentration of solute plays a crucial role in thermodynamic property. It was observed that increasing concentration entropy decreases indicating with the concentration system moves towards the more order form.

TABLE-4
CONCENTRATION-DEPENDENT THERMODYNAMIC PARAMETERS OF 3-SUBSTITUTED SCHIFF BASE OF 4-PIPERIDYL *N*-(4-CHLOROPHENYL)MALEIMIDE IN 80% AQUEOUS DMSO

Conc. (mol L^{-1})	ΔG (J mol^{-1} K^{-1})	ΔH (J mol^{-1} K^{-1})	ΔS (J K^{-1})
0.002	-335.975	-242.406	0.3139
0.004	-299.959	-205.463	0.3093
0.006	-264.709	-175.551	0.2918
0.008	-223.773	-138.492	0.2792
0.010	-188.867	-108.638	0.2626

Conclusion

In this work, the densities and viscosities of novel biologically active 3-substituted Schiff base of 4-piperidyl *N*-(4-chlorophenyl)maleimide in 80% DMSO were investigated. Measurements were performed by varying concentrations (0.002-0.01 M) of solute and temperature (298-313 K) of the system. The concentration-dependent study revealed that both viscosities and densities of solute increases with respect to concentration. However, temperature-dependent measurement shows the densities and viscosities of synthesized derivatives decreases with the temperature of the system. From the density measurement, the negative values of limiting molar volume and positive values of semi-empirical parameter (Sv) indicates a weaker solute-solvent interactions and stronger solute-solute interactions. Similarly, these interactions were also explained on the basis of the viscosity coefficient, the positive values of Falkenhagen constant (A) further confirmed the presence of stronger solute-solute interaction, however negative values of Jones-Dole constant (B) shows weaker of solute-solvent interactions. It was noticed that these parameters are found to be varied as a function of the temperature of the system. It was observed that at a lower temperature (298 K) solute-solute interactions are stronger whereas, with increasing temperature, it becomes weaker. However, solute-solvent interactions are weaker at lower temperatures and strengthen with the temperature of the system. Thus, the temperature of the system strongly affects the strength and nature of molecular interactions. The negative values of ΔG and ΔH and positive values of ΔS indicating reaction is spontaneous and exothermic. Thus, this investigation may be useful for the medicinal applications of the synthesized compound.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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