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Ion-Solvent Interactions of Chromium (III) and Iron (II) Sulphates in Aqueous Solutions

*Abdul Emmanuel O.¹, Owoyomi Olanrewaju¹, Akanni Michael Sola¹

Department of Chemistry, Obafemi Awolowo University, Ile-Ife, Nigeria.

ABSTRACT: The viscosities of the aqueous solutions of iron (II) and chromium (III) sulphates were measured in the temperature range, 298 – 323 K using an Oswald type viscometer. The objective was to evaluate the specific volumes of the solutes and examine the extent of the interaction of the solute with the solvent. The viscosities of the salt solutions increased with concentration and decreased with increase in temperature. The specific volumes of the solutes were fairly constant with the rise in concentration but decreased with increase in temperature. The viscosity data were fitted to the Jones-Dole and Mahiuddin equations. The B values obtained from the two methods were positive and decreased as the temperature decreased but the B coefficients from the latter method were generally higher than from the former. The first derivatives of B, from both techniques, with respect to temperature, were negative. The combination of the B values and their first derivative suggests that both solutes were structure makers with chromium sulphate perturbing the solvent structure more and veering to the large structure maker class. The activation energies for the viscous flows of the chromium salt solutions were slightly higher than those of the iron salt solutions of equal concentration.

KEYWORDS: Viscosity, Ions, Specific Volume, B-Coefficient, Structure Making, Activation Energy.

I. INTRODUCTION

It has been observed that small and highly charged cations are net structure makers of the solvent [1, 2]. Thus, for the alkali metals, Li^+ and Na^+ ions are classified as net structure makers, K^+ ion is slightly a structure breaker, and the breaking tendency increases through Rb^+ to Cs^+ ions. Among the halides, F^- is a structure maker while the other halide ions are structure breakers. Composite bulky acid radicals such as NO_3^- and ClO_4^- are strong structure breakers and a relatively less bulky ones like OH^- ion is a structure maker.

The mechanism involved in the making-breaking phenomena of the structure of solvents by ions implicated the spherical symmetrical electric field of an ion to tear water (solvent) dipoles out of the water lattice and make them point with the appropriate charge end towards the solute ions. The ion is thus viewed as a point charge and the solvent molecules as electric dipoles. The ion-dipole forces act as the principal basis of ion-solvent interaction. Thus, ions in solution will influence the shear stress and make viscosity a suitable technique for measuring the extent of ion-solvent interaction [3]. Poiseuille, as noted by Covington and Dickinson [4], like other researchers had observed that some salts can increase the viscosity of water whereas others can decrease it [5-7]. Jones and Dole expatiated on Poiseuille's observation and explained that the electrostatic forces tend to maintain aggregation of ions in solution to form an ionic atmosphere of oppositely charged ions and such aggregations would stiffen the solution or increase its viscosity [3]. Miller as well as others [8-11], have confirmed the observation of Onsager that the proportionality of such electrostatic forces to the square root of the concentration of dilute solutions can be used with confidence even if not with unquestionable faith while Jones and Dole [3] extended this relationship to obtain an expression for the relative viscosity of solutions in the form:

$$\eta_{rel} = \frac{\eta}{\eta_0} = 1 + A\sqrt{c} + Bc \quad (1)$$

where η is the viscosity of solution, η_0 is the viscosity of the pure solvent, c is the concentration in mol dm^{-3} , A is a constant that gives the extent of ion-ion interaction and B is a coefficient that represents the magnitude of ion-solvent interaction. The B -coefficient measures the solvation effects, hydrodynamic effects and ionic influence on the solvent structure. The smaller the value of B with respect to the variation of temperature, the greater is ion-solvent interaction [12-14]. B is also a measure of the order or disorder (entropy) introduced by ions into the solvent structure and at a particular temperature, positive B indicates a structure making effect while negative B shows a structure

breaking effect. B is known to have additivity characteristics, that is, B can be partitioned into B^+ and B^- as contributions from the cations and anions, respectively [15, 16].

Varma and Kumar, while reporting the viscosity measurement of some magnesium soap solutions in non-aqueous solvents, observed that the viscosity and the activation energy for the viscous flow increase with increasing carbon chain length suggesting that the carboxylate anion plays a critical role in its macroscopic flow [17]. Guveli [18], studied the effect of aliphatic alcohols on the B-coefficients of decyltrimethyl ammonium bromide at 298K. He concluded that the decreased dielectric constant led to a decrease of the charge density and in turn caused a decrease of the surface ionic field and consequently enhanced the structure breaking effect of the solute.

In our previous study [19], the A and B coefficients of some concentrations of aqueous sodium periodate, NaIO_4 (1.02×10^{-3} – 9.00×10^{-2} mol dm^{-3}) were determined to be 0.0061 and 0.0298 respectively. The values compared well with previously published values of $A = 0.0072$ and $B = 0.0215$ for the aqueous solution of NaIO_4 by Nightingale and Benck [20]. This close agreement between our values and those of these workers enhanced our confidence in the accuracy of the use of the viscosity method to obtaining the A and B coefficients as well as the specific volumes of solutes in solutions.

The present study measured the viscosities of some concentrations of chromium (III) and iron (II) sulphates in water. The objective was to examine the extent to which the size or the charge of the cations will disturb the structure of the solvent and affect the specific volumes of the solutes.

II. MATERIALS AND METHODS

The hydrated chromium (III) and iron (II) sulphates ($\geq 99\%$) were analytical grades purchased from Sigma Aldrich and were used without further purification. Electrolyte solutions were prepared with high purity deionized water produced by reverse osmosis.

The calibration of the Oswald type viscometer used, and the viscosity measurements were as described elsewhere in our previous article [21]. The viscometer constant was 1.516×10^{-2} with a calculated standard deviation in this value being less than 3%.

III. EXPERIMENTAL RESULTS

Viscosity of liquids generally decreases with increase in temperature in a rapid but non-linear pattern and is independent of pressure up to several atmospheres. It is thus sufficient to measure the viscosity of liquids without paying attention to the little variation of the normal atmosphere while the more critical factor of the temperature was addressed using a regulated thermostated water bath. The viscosities of 0.10 to 1.00 mol dm^{-3} $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and 0.10 to 0.50 mol dm^{-3} $\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$ were measured at different temperatures and the viscosity data were fitted to the Einstein equation [22, 23]:

$$\eta = \frac{1+0.5\phi}{(1+\phi)^4} \quad (2)$$

The quantity, η is the relative viscosity and ϕ is the volume fraction occupied by the solute expressed as a percentage or in cm^3 of 100 cm^3 of solution. Thus, ϕ divided by the weight of the solute per 100 cm^3 of solution gives the specific volume (volume occupied by one gram) of the solute. The data obtained at 303K for the salt solutions are presented in tables 1 and 2.

Table 1: Viscosities and Specific Volumes of Varying Concentrations of Iron (II) Tetraoxosulphate (VI) heptahydrate in Water at 303K

Concentration (M)	Viscosity (mPa.s)	Relative Viscosity	Specific volume
0.10	1.06	1.09	0.71
0.15	1.12	1.15	0.74
0.20	1.18	1.22	0.77
0.25	1.22	1.26	0.72
0.30	1.29	1.33	0.73
0.40	1.42	1.47	0.74
0.50	1.59	1.64	0.76

Table 2: Viscosities and Specific Volumes of Varying Concentrations of Chromium (III) Tetraoxosulphate (VI) pentadecahydrate in Water at 303K.

Concentration (M)	Viscosity (mPa.s)	Relative Viscosity	Specific volume
0.10	1.24	1.28	0.80
0.15	1.40	1.44	0.79
0.20	1.53	1.58	0.74
0.25	1.72	1.77	0.73
0.30	2.00	2.06	0.76
0.40	2.62	2.71	0.76
0.50	3.19	3.29	0.72

The tables show that the viscosities of the salt solutions increase gradually with increase in concentration. This can be attributed to the increase in both the solute-solvent and solute-solute interactions at high concentrations. However, the specific volumes are, within the limit of experimental error, fairly constant with varying concentrations at constant temperature. The trend agrees with Kunitz[23] who observed that the specific volume should be constant at a given temperature for the various concentrations of a solute or suspension unless the solute or suspension is solvated to a significant extent.

The effect of the variation of temperature on the viscosities of the salt solutions or specific volumes of the solute was examined by measuring the viscosity of 0.25 mol dm⁻³ solution of Cr₂(SO₄).15H₂O in the 298 - 325K temperature range. The data obtained is presented in table 3.

Table 3: Viscosity and Specific Volume of 0.25 mol dm⁻³ Chromium (III) Tetraoxosulphate (VI) Pentadecahydrate at Various Temperature

Temperature(K)	Viscosity (mPa.s)	Relative Viscosity	Specific volume
298	1.896	1.957	0.846
303	1.716	1.771	0.778
308	1.557	1.606	0.611
313	1.337	1.379	0.423
318	1.205	1.243	0.284
323	1.106	1.141	0.186

Table 3 shows that both the viscosity and specific volume decrease with increase in temperature. The decreasing viscosity is due to the increased kinetic motion at higher temperatures which promotes the breaking of the intermolecular bonds between adjacent layers of the streamline flow. The association equilibria in solution is much reduced and the increased loss of water molecules around the ions at higher temperatures coupled with the rearrangement of the units of viscous flow affect the specific volume of the solute and cause it to decrease with the rise in temperature[24].

The viscosity data was fit to the Jones-Dole equation and the A and B coefficients from the intercepts and slopes respectively of the plots of $\frac{\eta_{rel}-1}{\sqrt{c}}$ against $\sqrt{c}$ were obtained. The values of A and B coefficients obtained at different temperatures are shown in table 4.

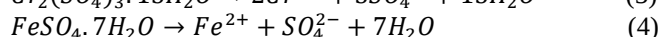
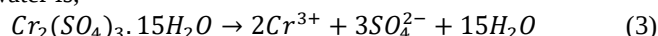
Table 4: Values of A ($\text{dm}^{1/2} \text{mol}^{-1/2}$) and B ($\text{dm}^3 \text{mol}^{-1}$) Coefficients from the Jones-Dole Plots for $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$ at Different Temperatures

Temperature(K)	Iron (II) sulphate		Chromium (III) sulphate	
	A	B	A	B
298	- 0.316	2.215	- 0.981	5.912
303	- 0.442	2.107	- 1.131	5.773
308	- 0.593	1.721	- 1.426	5.561
313	- 0.707	1.400	- 1.588	3.366
318	- 0.832	1.347	- 1.692	3.348
323	- 1.192	1.327	- 1.750	3.311

The A coefficient which indicates the extent of the ion-ion interactions decreases with increasing temperature. The trend is in consonance with the fact that as the kinetic energy increases, the influence of the ions on each other is greatly reduced. Similarly, the B coefficient decreases with the rise in temperature and the decreasing B is indicative of the greater ion-solvent interactions. This assertion is in agreement with the observation of Merza[15] that the smaller the B value due to rise in temperature, the greater the ion-solvent interaction. The rationalization provided is that at low temperatures, ions in solution are close to each other while at higher temperatures ions are more dispersed to reduce influence of ion-ion interaction and thus enhance the ion-solvent interactions.

The values of B are positive for both salts showing that the solutes (ions) are structure makers and thus induce order into the solvent structure. The aggregation of ions at low temperatures gradually gives a way to the ordering of solvent molecules around the ions as temperature rises. It must be noted however, that our temperature range is narrow as we presume that at temperatures above the upper limit within which the present study was carried out, the kinetic energy or kinetic collisions of both the solute ions and the solvent molecules will be sufficiently high such that both the ion-ion and ion-solvent influence will become marginal for the order induced by the ions into the solvent structure to break down.

Both the A and B coefficients are higher for $\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$ in absolute magnitude than for $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. The ionization of the salts in water is,



The higher charge on the cation as well as the greater number of ions in the chromium sulphate solution than that of iron sulphate solution are responsible for the higher values of the A and B coefficients in the former solution.

The Jones-Dole equation has been known to give good results at low concentrations ($\leq 0.30 \text{ M}$). An improvement of the Jones-Dole equation applicable at higher concentrations was developed by Mahuiddin[25] from the Vogel-Tamman-Fulcher equation in the form:

$$\eta_{rel} = a_o + \exp(b_o M + c_o M^2) \quad (5)$$

where η is the viscosity of the solution, a_o , b_o and c_o are constants for a solution at a particular temperature and M is the concentration in mol dm^{-3} . In the logarithmic form,

$$\ln(\eta_{rel}) = \ln a_o + (b_o M + c_o M^2) \quad (6)$$

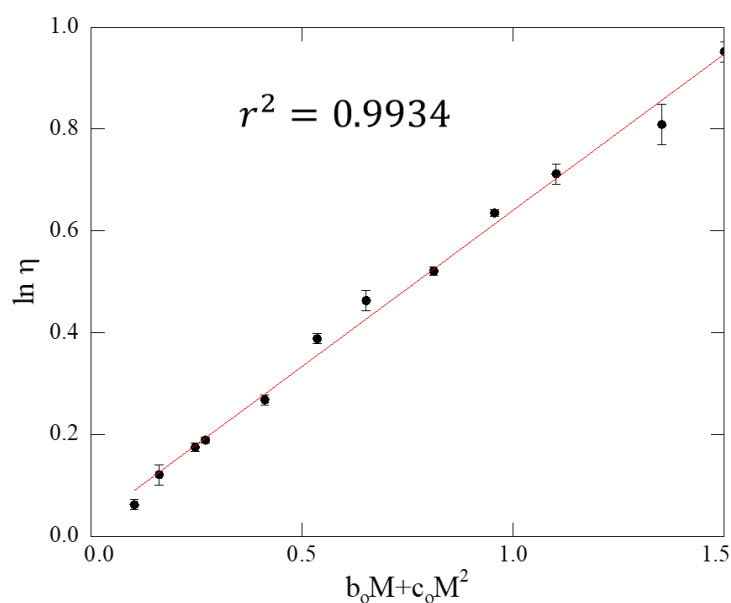
A plot of $\ln \eta$ against M, gives a parabola and non-linear least squares fit enables the determination of the constants and are in table 5.

The three constants decrease with the rise in temperature and the values for the chromium salt are generally higher than those for the iron salt. The plots of $\ln \eta$ against $(b_o M + c_o M^2)$ for the iron and chromium salts are shown in Figures 1 and 2 respectively. The degree of linearity ($R^2 = 0.993$ to 1.0) shows the extent of the applicability of the Mahuiddin equation at high solution concentrations.

Table 5: Values of a_o , b_o and c_o for the Solutions of Chromium and Iron Sulphates at Different Temperatures.

Temperature(K)	$\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$		
	a_o	b_o	c_o
298	1.081	2.160	0.663
303	1.017	1.967	0.578
308	0.896	1.802	0.513
313	0.891	1.556	0.323
318	0.816	1.394	0.284
323	0.677	1.250	0.157

Temperature(K)	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$		
	a_o	b_o	c_o
298	0.868	1.017	0.642
303	0.842	0.941	0.562
308	0.798	0.923	0.508
313	0.749	0.877	0.221
318	0.722	0.814	0.114
323	0.686	0.732	0.052

**Figure1:** Plot of $\ln \eta$ against $(b_o M + c_o M^2)$ for Iron (II) Sulphate Pentahydrate

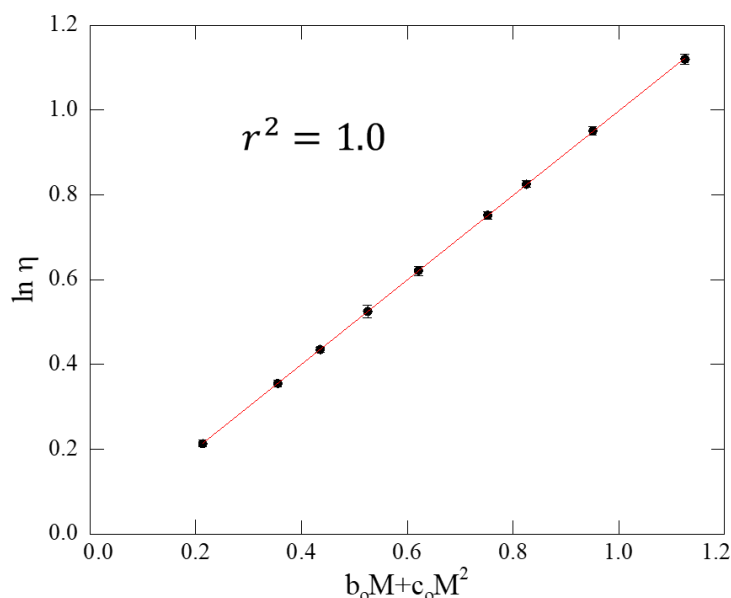


Figure 2: Plot of $\ln \eta$ Against $(b_0M + c_0M^2)$ for Chromium (III) Sulphate Pentadecahydrate

The product of a_0 and b_0 had been equated by Mahuiddin and co-worker to the B coefficient obtained from the Jones-Dole equation. Table 6 shows the values of the B coefficients from the two routes.

Table 6: Values of B Coefficients from the Jones-Dole and Mahuiddin Equations for Iron (II) and Chromium (III) Sulphates at Different Temperatures.

Temperature(K)	FeSO ₄ .7H ₂ O	
	Jones-Dole B	B = ($a_0 \times b_0$)
298	2.215	0.883
303	2.107	0.891
308	1.721	0.736
313	1.400	0.657
318	1.347	0.588
323	1.327	0.502
Temperature(K)	Cr ₂ (SO ₄) ₃ .15H ₂ O	
	Jones-Dole B	B = ($a_0 \times b_0$)
298	5.912	2.335
303	5.773	2.000
308	5.561	1.615
313	3.366	1.386
318	3.348	1.138
323	3.311	0.846

The B coefficients from the Jones-Dole plots for both salts at the temperature coverage of this study are generally higher than those from the Mahuiddin plots. Both methods gave the same correlation of B with the temperature. It is tempting to speculate from the data in table 6 that the extent of the solute-solvent interaction is more pronounced than predicted by the Mahuiddin data. This view may be lacking in substance because as earlier noted, the Jones-Dole equation gives a low accuracy beyond 0.30 molar concentration. However, similar to the Jones-Dole B-coefficient, the Mahuiddin B-coefficient for the chromium salt at each temperature in the 298 - 323 K range is higher than the value for the iron salt.

It has been observed [26, 27] that the temperature derivative of the B-coefficient is a better decisive parameter for determining the structure making or breaking of any solute rather than the B coefficient. The value of $\frac{dB}{dT}$ was calculated from the slope of the plot of the B-coefficient against the temperature. The values are presented in table 7.

Table 7: Values of dB/dT for Iron (II) and Chromium (III) Sulphates from the Jones-Dole and Mahiuddin B-Data.

Method	Temperature derivative of B (dB/dT)	
	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	$\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$
Jones-Dole	- 0.04	- 0.14
Mahiuddin	- 0.02	- 0.06

The sign of $\frac{dB}{dT}$ gives information about the structure making-breaking tendencies and in general, negative $\frac{dB}{dT}$ value has been attributed to a solute that functions as a structure maker of a solvent while positive $\frac{dB}{dT}$ value classifies a solute into the structure breaker of a solvent. It has been reported [20] that ions in solution can be classified into four groups based on the values of the B-coefficient and its first derivative with respect to temperature. The groups are,

- Ions that are structure makers require that $B > 0$ and $\frac{dB}{dT} < 0$.
- Ions that are anomalous structure makers require that $B > 0$ and $\frac{dB}{dT} > 0$.
- Ions that are structure breakers require that $B < 0$ and $\frac{dB}{dT} > 0$.
- Ions that are large structure makers with a peripheral solvation require $B > 0$ and $\frac{dB}{dT} < 0$.

Peripheral solvation is a process in which an ion is unhydrated with the energy change for the movement of solvent molecules away from the ion much less than the energy of their moving away from another solvent molecule. Both the Jones-Dole and Mahiuddin methods gave $B > 0$ and $\frac{dB}{dT} < 0$. Thus, the Nightingale classification makes it unambiguous that both iron (II) and chromium (III) sulphates are structure makers. It is tempting to suggest that the higher B and the more negative $\frac{dB}{dT}$ values for the chromium salt may make it a more powerful structure maker than the iron (II) salt. This assertion could have easily been substantiated if the B coefficient could be partitioned into the B^+ and B^- coefficients for the cations and anions respectively such that the contribution to the structure of the solvent making by the Fe^{2+} and Cr^{3+} ions can be quantitatively assessed. Unfortunately, at the moment, there is no clear general method of how such partition can be carried out. The strong perception, however, is that the higher charge of +3 carried by the chromium ion as against +2 for the iron ion makes the ionic radius, $r_{\text{Cr}^{3+}} < r_{\text{Fe}^{2+}}$. As noted earlier, contribution to the magnitude of B comes from the size of ions, the solvation and the hydrodynamic effects. These factors should be more dominant in the solution of $\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$ and make the salt a better structure maker than the iron salt. In fact, we speculate that the iron salt is a structure maker to a low extent while the chromium salt veer into group four of Nightingale classification, a large structure maker.

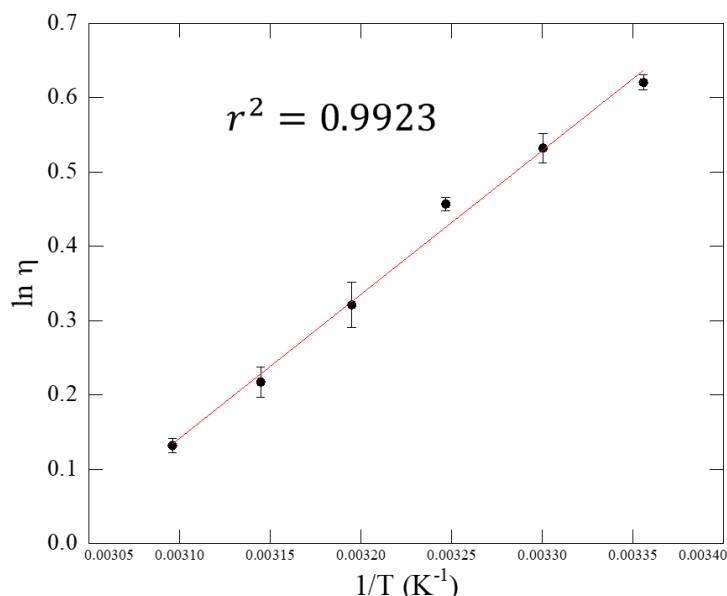


Figure 3: Plot of $\log \eta$ against Inverse of Absolute Temperature for 0.25 mol dm^{-3} Chromium (III) Sulphate

The activation energy for the viscous flow was obtained from the Arrhenius-type equation:

$$\ln(\eta_{rel}) = \ln A + \frac{E_{\eta_{rel}}}{RT} \quad (5)$$

A is the pre-exponential factor and $E_{\eta_{rel}}$ is the energy required to transfer one molecule of the solute from one equilibrium position to another. It is envisaged that each molecule in a liquid occupies roughly an equilibrium position and before it can pass through another such position in the direction of flow, it must acquire a definite energy of activation. This energy is associated with the interionic forces in solution and generally, the higher this energy, the greater is the viscosity of liquids, although some other factors may also be at play. The activation energy for the viscous flow of 0.15 mol dm^{-3} chromium (III) sulphate, as an example, is shown in Figure 3. The activation energy obtained from the slopes of such plots for the viscous flows of different concentrations of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$ in the temperature range of 298 - 323 K are shown in table 8.

Table 8: Values of the Activation Energy (kJ mol^{-1}) for Different Concentrations of Aqueous Solutions of Iron (II) and Chromium (III) Sulphate at 303 – 323 K.

Concentration (M)	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	$\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$
0.10	13.8	15.8
0.15	14.4	16.1
0.20	15.9	17.5
0.25	16.8	18.5
0.30	18.3	20.8
0.40	19.8	21.9
0.50	21.1	23.4

The table shows that the activation energy for the viscous flow increases with increase in concentration. The values for the chromium salt solutions are slightly higher than those for the iron salt solutions of equal concentration. The mechanism of viscous flow implicated the interwoven hydrogen bonds in water (solvent matrix) to create vacant holes or sites to which ions of the solute will “occupy and vacate” as flow progresses. At higher concentrations, ions will cause obstruction to the flow due to ion association that can temporarily block holes. This presents a net hindrance to the flow and hence a higher energy of activation will be required to set the units of flow on a sustained streamline motion and because there are more ions causing such obstruction in the solution of the chromium salt than in that of the iron salt, the former solution will be more viscous with higher activation energies for all the solutions of corresponding strength.

IV. CONCLUSION

Density and viscosity data of Iron (II) and Chromium (III) sulphate in aqueous solutions have been measured as a function of temperature. The specific volume, Jones-Dole B-coefficients and the temperature derivative of the B-coefficients were determined using the experimental data and used to classify the effect of the solutes on the structure of the solvent (water) based on several theoretical classifications. Results show that both the B-coefficient and the $\frac{dB}{dT}$ obtained from two separate classifications suggest that both solutes are structure makers for water as a solvent. The activation energy of viscous flow, calculated from the Arrhenius-type equation shows higher values for the Chromium sulphate than those of the Iron sulphate suggesting greater interionic forces for the former, and thus its higher viscosity.

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