



RESEARCH ARTICLE



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Study of Solvent and Temperature Effects on Volumetric, Viscometric and Conductometric Studies of Amlodipine Besylate

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Abstract

Volumetric, viscometric and conductometric studies of 0.8×10^{-3} to 8×10^{-3} mol/Kg solutions of amlodipine besylate in distilled water and methanol were done at temperature range of 293.15, 298.15, 303.15, 308.15, 313.15, 318.5 and 323.15 K. from the determined values of density apparent molar volume (V_{ϕ}) and viscosity (η) were evaluated.

Density data was analyzed and discussed using Masson empirical relationship and that of viscosity to Jones-Dole equation to evaluate partial molar volume (V_{ϕ}^0), intrinsic co-efficient SV, viscosity A and B parameters. All parameters were discussed as a function of concentration and temperature. For all set of dilutions conductometric studies in both solvents of study were employed. Obtained data was analyzed to judge the ion-ion and ion-solvent interactions. Structure making and breaking aspects of amlodipine besylate in polar solvents were discussed. These all parameters were studied to disclose the mechanism of action of drug and its transportation across biological membranes.

Keywords: Apparent molar volume; partial molar volume; intrinsic co-efficient; Masson equation; Jones-dole equation; ion-ion interactions; ion-solvent interactions; viscosity A parameter; viscosity B parameter; electrical conductivity

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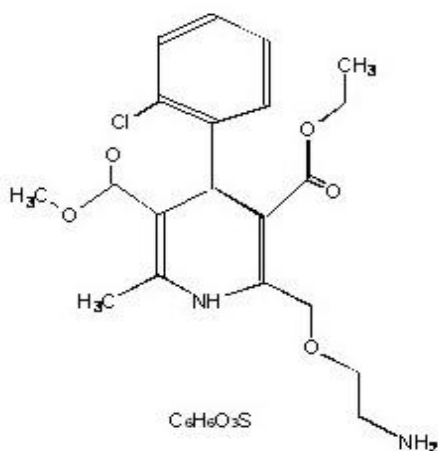
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INTRODUCTION

Volumetric properties play an important role in disclosing the various types of interactions occurring in solution⁸. These are also helpful in elucidating the nature and effect of solute in solvent, intermolecular interactions and penetration of drug across biological membranes. The best way of having insight of volumetric properties is through density determinations with subsequent evaluation of apparent molar volume, partial molar volume and intrinsic co-efficient.

Viscosity is an important physical property of liquids⁴ which measures the viscous flow of drug between adjacent layers of liquids. From pharmaceutical point of view, viscosity is related to the absorption of drug in body leading to its metabolic and physical activities across biological membranes. Conductivity is the material's ability to conduct electricity and is related to the ionic contents of the solution. Conductivity measurements are the most inexpensive and reliable way of measuring ionic contents of the solution. These all parameters are helpful in inferring the structure of electrolyte solution¹².

Amlodipine besylate is generally used and employed as anti-arrhythmic, anti-hypertensive and antiangial agent. Being the calcium channel blocker and having anti-hypertensive potential, it is regarded as an important drug in biology, pharmacy, pharmacology and medical field. Due to its immense importance volumetric, viscometric and conductometric studies as a function of solvent and temperature were employed.



Structure of Amlodipine Besylate

It has been proved by number of workers (1-3), (5-7), (9-1) that volumetric properties play an important role to judge intermolecular interactions occurring in solutions. Our present work deals with the study of different parameters including apparent molar volume, partial molar volume, intrinsic co-efficient, viscosity A and B parameters and conductometric studies and

dependence of these parameters on concentration and temperature were discussed in terms of ion-ion and ion-solvent interactions.

EXPERIMENTAL

MATERIALS AND INSTRUMENTATION:

Chemicals including Amlodipine besylate manufactured by Amsal chemicals India, recrystallized before use, solvents: distilled water and methanol (99% pure) by Merck Germany.

Instruments including Pars Azma oven, Electronic balance, Thermostatic water bath, Ostwald's viscometer, conductivity meter, laboratory based glassware. All other reagents used were of analytical grade.

METHODOLOGY

Volumetric studies

The best way of studying the volumetric properties of solvents, mixed solvents as well as their solutions is through density determination. Density determination of the 0.8×10^{-3} to 8×10^{-3} mol/kg dilute solutions of amlodipine besylate in distilled water and methanol was done with pre-calibrated specific gravity bottles (made up of Pyrex glass) under thermostatic conditions at temperature range of 293.15, 298.15, 303.15, 308.15, 313.15, 318.15 and 323.15 K.

Viscometric Studies

Viscosity is an important physical quantity. Many different apparatus are available for its determination; we have used Ostwald's viscometer in the present study. In present study, evaluation of viscosity was done under thermostatic conditions at temperature range of 293.15, 298.15, 303.15, 308.15, 313.15, 318.15 and 323.15 K.

Conductometric Studies

Conductivity determination was done with the help of pre-calibrated HANNA 8633 conductivity meter under thermostatic conditions at temperature range of 293.15, 298.15, 303.15, 308.15, 313.15, 318.15 and 323.15 K. Calibration of the apparatus was done with 0.1M KCl solution.

RESULTS AND DISCUSSION

Volumetric Studies

Density

Density determinations were done in order to have insight of volumetric properties which in turn are helpful in disclosing the nature of intermolecular interactions. Following Table 1, 2 contains the data regarding the density for all dilutions of amlodipine besylate in distilled water and methanol respectively.

Molality (mol/kg)	Density (d) (g/cm ³)						
	293.15 K	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
0.0008	0.99822	0.99702	0.99561	0.9940	0.99219	0.990189	0.987984
0.0010	0.99823	0.99702	0.99562	0.99401	0.99220	0.990191	0.987985
0.0012	0.99824	0.99703	0.99562	0.99401	0.992203	0.990194	0.987986
0.0016	0.99826	0.99705	0.99564	0.99402	0.99221	0.990195	0.987987
0.0020	0.99828	0.99707	0.99565	0.99403	0.99222	0.99020	0.987992
0.0025	0.99831	0.99709	0.99567	0.99405	0.99223	0.99021	0.988000
0.0040	0.99841	0.99718	0.99575	0.99412	0.99229	0.99026	0.998042
0.0050	0.99848	0.99724	0.99581	0.99418	0.99234	0.99030	0.988077
0.0060	0.99856	0.99731	0.99587	0.99424	0.99240	0.99035	0.988129
0.0080	0.99873	0.99747	0.99603	0.99438	0.99252	0.99046	0.988231

Table 1: Data of density (d) for the aqueous solutions of amlodipine besylate at all working temperatures (K)

Molality (mol/Kg)	Density (d) (g/cm ³)						
	293.15 K	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
0.0008	0.79103	0.78653	0.78202	0.77702	0.77201	0.76751	0.76301
0.0010	0.79104	0.78653	0.78203	0.77702	0.77202	0.76752	0.76301
0.0012	0.79105	0.78654	0.78203	0.77703	0.77202	0.76752	0.76302
0.0016	0.79107	0.78655	0.78205	0.77704	0.77203	0.76753	0.76302
0.0020	0.79109	0.78657	0.78206	0.77706	0.77204	0.76754	0.76303
0.0025	0.79112	0.78659	0.78208	0.77707	0.77206	0.76755	0.76304
0.0040	0.79121	0.78664	0.78215	0.77713	0.77211	0.76760	0.76308
0.0050	0.79127	0.78672	0.78220	0.77717	0.77214	0.76763	0.76311
0.0060	0.79135	0.78678	0.78225	0.77722	0.77218	0.76767	0.76314
0.0080	0.79151	0.78690	0.78227	0.77732	0.77226	0.76775	0.76321

Table 2: Data of density (d) for the solutions of amlodipine besylate in methanol at all working temperatures (K)

From above table an increasing trend of viscosity with concentration has been observed. This is attributed to the decrease in volume due to decrease in mean distance between the solute and solvent molecules leading to enhanced solvation capacity of solute. Furthermore the value of density got decreased with rise in temperature due to the volume expansion caused by decrease in intermolecular forces between amlodipine besylate and solvent molecules. This trend is followed by both solvents of study.

3.1.1 Apparent Molar Volume

From the determined values of density apparent molar volume for the solutions of amlodipine besylate in distilled water as well as in methanol was calculated using following expression:

$$V_{\phi} = \frac{M}{d^0} - \frac{1000}{c} \left[\frac{d-d^0}{d^0} \right] \quad (1)$$

Following Tables 3, 4 contain the apparent molar volume data of all the solutions of amlodipine besylate in both solvents of study at all working temperatures:

Molality (mol/Kg)	Apparent Molar Volume (V_{ϕ}) (Cm ³ .mol ⁻¹)						
	293.15 K	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
0.0008	539.0998	549.4055	558.2841	564.8168	575.9898	586.5332	594.4241
0.0010	534.5006	543.9467	553.3914	563.3094	571.0879	581.4793	588.9769
0.0012	533.0562	541.1384	549.8122	560.1769	568.9874	577.7764	585.1941
0.0016	528.8238	537.8775	546.4567	557.4166	564.4358	575.7832	582.5592
0.0020	526.4126	534.0228	544.1271	554.2507	562.1698	573.3220	578.1178
0.0025	523.3552	531.7059	540.6328	550.0134	558.6988	568.0097	573.7166
0.0040	516.0929	524.4326	531.7183	540.3132	548.6121	557.9765	563.2783
0.0050	512.2457	520.7055	527.4845	534.2422	542.1304	552.0664	558.1960
0.0060	508.2022	516.6024	524.2218	530.6481	538.0912	547.5242	551.9185
0.0080	501.0803	509.6079	515.6797	522.8471	530.3521	539.7642	544.3745

Table 3: Data of apparent molar volume (V_{ϕ}) for the aqueous solution of amlodipine besylate at all working temperatures (K)

Molality (mol/Kg)	Apparent Molar Volume (V_{ϕ}) ($\text{Cm}^3\cdot\text{mol}^{-1}$)						
	293.15 K	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
0.0008	655.1856	671.3896	684.5286	696.6767	708.2568	713.9077	726.1082
0.0010	652.6204	669.2499	678.4109	690.3594	707.4945	711.8880	724.4911
0.0012	651.3198	668.1741	678.6832	690.0082	702.5992	710.2636	721.2856
0.0016	649.2458	666.3407	676.6832	689.0064	700.8108	706.5992	718.4299
0.0020	646.3364	663.2787	673.5167	684.1134	699.0947	704.6836	717.0753
0.0025	643.1706	661.5443	671.5787	681.3151	696.7978	701.8612	715.8461
0.0040	633.5406	654.6207	664.5978	675.5277	689.9597	696.7182	709.5961
0.0050	629.2770	649.4894	660.5449	672.6707	684.9757	693.4112	706.3627
0.0060	624.5675	645.2358	657.0553	669.7616	684.5288	690.6658	703.7641
0.0080	615.1277	639.0853	657.2732	663.4484	679.1359	684.8944	698.6610

Table 4: Data of apparent molar volume (V_{ϕ}) for the solution of amlodipine besylate in methanol at all working temperatures (K)

From the above determined data it is cleared that the value of AMV decreased with rise in concentration and increased with the increase in temperature. The decreased value with concentration is related to the compression in volume due to change in mean distance and solvophobic hydration between amlodipine besylate polar groups and solvent. The increased value of AMV with temperature is attributed to the reduction in electrostriction due to weaker intermolecular interactions. Furthermore gradual high value of AMV in case of methanol as compared to distilled water is related to the methyl group in methanol leading to steric hindrance in the way of electrostriction causing a change in volume.

Partial Molar Volume

In order to have insight of solute-solvent interactions the obtained data was analyzed using Masson equation to evaluate partial molar volume (V_{ϕ}^0) from intercept and intrinsic co-efficient (S_V) from the slope of graphs between apparent molar volume and square root of molar concentration using following relationship:

$$V_{\phi} = V_{\phi}^0 + S_V C^{1/2}$$

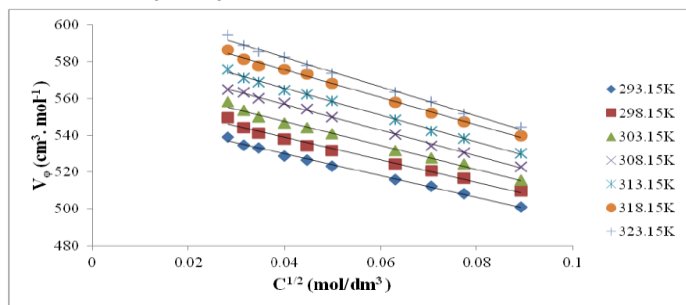


Figure 1: Graph showing the linear trends of apparent molar volume (V_{ϕ}) VS square root of concentration ($C^{1/2}$) for the aqueous solution of amlodipine besylate at all working temperatures

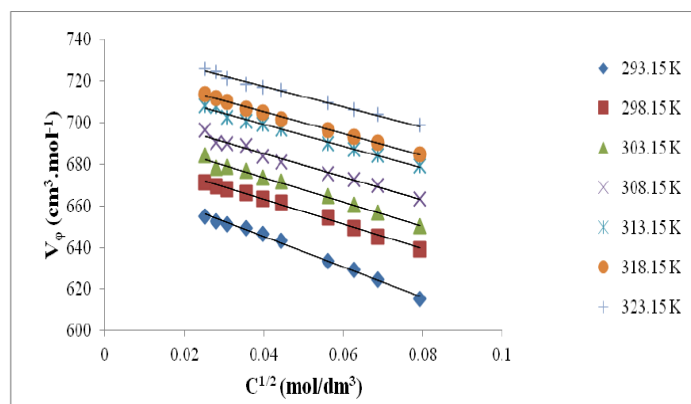


Figure 2: Graph showing the linear trends of the apparent molar volume (V_{ϕ}^0) and square root of molar concentration ($C^{1/2}$) of amlodipine besylate in methanol at all working temperatures

The value of PMV is found to be positive and increased in linear manner with temperature indicating strong solute-solvent interactions. This may be attributed to the solvation and hydration behaviour of drug, reduction in electrostriction and change in volume due to migration of solvent from second layer around amlodipine besylate to the bulk solvent. The value of intrinsic co-efficient was found to be negative indicating weak solute-solute interactions and hydrophobic character of amlodipine besylate molecule leading to insufficient counter ion binding with solute molecules which becomes less appreciable with concentration. The increase or decreased negative value of S_V with temperature is indicative of decreased dissociation pattern of amlodipine besylate molecules in solution.

Temperature (K)	Data of drug in distilled water		Data of drug in methanol	
	V_{ϕ}^0	S_V	V_{ϕ}^0	S_V
293.15	553.5	-591.4	674.2	-726.3
298.15	563.0	-606.8	686.8	-596.3
303.15	573.9	-658.0	697.1	-589.4
308.15	585.1	-707.2	707.8	-569.8
313.15	594.8	-735.0	720.5	-536.1
318.15	605.6	-752.8	726.0	-528.6
323.15	614.3	-801.4	737.2	-498.7

Table 5: Data of partial molar volume (V_{ϕ}^0) and intrinsic coefficient (S_V) for the solution of amlodipine besylate in distilled water and methanol at all working temperatures

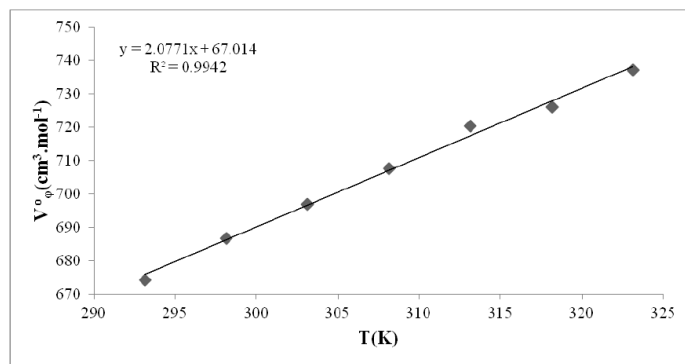


Figure 4: Graph of the partial molar volume (V_{ϕ}^0) for the solution of amlodipine besylate in methanol at all working temperatures

VISCOMETRIC STUDIES

Viscosity is among the earliest studies of solution chemistry and is useful in developing the idea of solution process. From pre-determined density values co-efficient of viscosity was determined using following expression:

$$\eta_1 = \frac{d_1 t_1}{d_2 t_2} \eta_2 \tag{3}$$

η_1 = co-efficient of viscosity of solution

η_2 = co-efficient of viscosity of solvent taken from literature

d_1 = density of the solution, d_2 = density of the solvent

t_1 = time of flow of solution, t_2 = time of flow of solvent

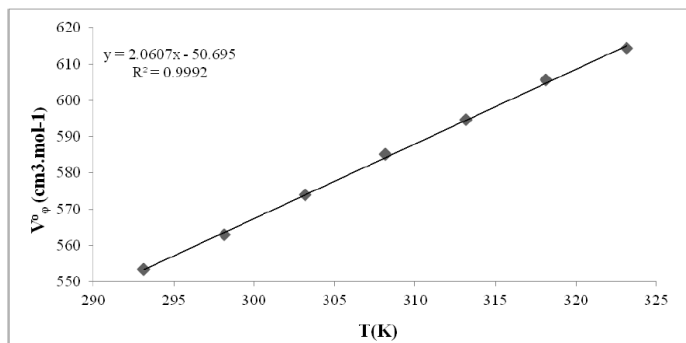


Figure 3: Graph of the partial molar volume (V_{ϕ}^0) for the aqueous solution of amlodipine besylate at all working temperatures

M(mol/kg)	Co-efficient of Viscosity (η) (mPa.s)						
	293.15K	298.15K	303.15K	308.15K	313.15K	318.15K	323.15K
0.0008	1.0214	0.9213	0.8285	0.7522	0.6893	0.6305	0.5857
0.0010	1.0334	0.9278	0.8374	0.7614	0.6997	0.6385	0.5917
0.0012	1.0388	0.9336	0.8438	0.7687	0.7067	0.6477	0.6003
0.0016	1.0495	0.9472	0.8584	0.7828	0.7211	0.6606	0.6124
0.0020	1.0589	0.9589	0.8705	0.7944	0.7379	0.6756	0.6260
0.0025	1.0729	0.9744	0.8890	0.8121	0.7565	0.6907	0.6416
0.0040	1.1118	1.0185	0.9331	0.8635	0.8092	0.7412	0.6899
0.0050	1.1379	1.0470	0.9650	0.9003	0.8434	0.7757	0.7216
0.0060	1.1647	1.0755	0.9969	0.9309	0.8747	0.8101	0.7528
0.0080	1.2144	1.1417	1.0607	0.9934	0.9396	0.8779	0.8157

Table 6: Data of viscosity (η) for the aqueous solution of amlodipine besylate at all working temperatures

M (mol/kg)	Co-efficient of Viscosity (η) (mPa.s)						
	293.15 K	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
0.0008	0.5996	0.5718	0.5286	0.5037	0.4753	0.4498	0.4284
0.0010	0.6093	0.5827	0.5397	0.5173	0.4905	0.4641	0.4380
0.0012	0.6164	0.5889	0.5453	0.5358	0.4969	0.4705	0.4454
0.0016	0.6261	0.6007	0.5564	0.5403	0.5098	0.4841	0.4586
0.0020	0.6366	0.6089	0.5657	0.5525	0.5247	0.4969	0.4729
0.0025	0.6483	0.6225	0.5935	0.5695	0.5477	0.5151	0.4907
0.0040	0.6835	0.6593	0.6221	0.6187	0.5855	0.5660	0.5357
0.0050	0.7108	0.6898	0.6474	0.6501	0.6152	0.5989	0.5663
0.0060	0.7311	0.7183	0.6734	0.6807	0.6457	0.6306	0.6025
0.0080	0.7768	0.7692	0.7251	0.7210	0.7045	0.6752	0.6638

Table 7: Data of viscosity (η) for the solution of amlodipine besylate in methanol at all working temperatures

From the above tabulated data it is evident that value of viscosity got increased with the concentration of solution due to the development of solute-solute and solute-solvent interactions, the resistance in the flow of drug solution develops. The viscosity of the amlodipine besylate solutions in water and methanol decreased due to the breakdown of solvent and drug molecules connected through hydrogen bonds due to the

attainment of excessive kinetic energy leading to decreased strength of intermolecular strength.

Viscosity A and B Parameters

The obtained data of viscosity was analyzed using Jones-Dole parameter:

$$\Psi = \frac{\eta_r - 1}{C^{1/2}} \tag{3}$$

$$\Psi = A + BC^{1/2} \tag{4}$$

M (mol/kg)	Intrinsic viscosity (Ψ) (dm ³ .mol ⁻¹)						
	0.6852	1.2886	1.4428	1.6957	1.9205	2.1208	2.2399
0.0008	0.6852	1.2886	1.4428	1.6957	1.9205	2.1208	2.2399
0.0010	0.9928	1.3833	1.6449	1.9208	2.2219	2.3252	2.3458
0.0012	1.0597	1.4495	1.7399	2.0481	2.3346	2.5661	2.5922
0.0016	1.1849	1.6428	1.9672	2.2630	2.5747	2.7724	2.8031
0.0020	1.2722	1.7624	2.0994	2.3855	2.8843	3.0452	3.0585
0.0025	1.4185	1.9258	2.3423	2.6330	3.1461	3.2356	3.3083
0.0040	1.7346	2.3089	2.7326	3.2170	3.7706	3.9107	4.0106
0.0050	1.9223	2.5212	3.0138	3.6043	4.1157	4.3203	4.4048
0.0060	2.1021	2.7171	3.2713	3.8436	4.3819	4.6989	4.7558
0.0080	2.3774	3.1899	3.7347	4.3066	4.9138	5.3537	5.4013

Table 8: Data of viscosity (η) for the aqueous solution of amlodipine besylate at all working temperatures

In order to have insight of solute-solute and solute-solvent interactions A and B parameters were evaluated from the intercept and slope of the graphs between Ψ and $C^{1/2}$. The value of A parameter was small and positive but gradually got increased with rise in temperature. This is attributed to the weaker ion-ion interactions at initial but got strength at higher

temperature due to thermal movement of molecules leading to enhanced solvation capacity and solute-solute interactions. Viscosity B parameter was found to be large and positive indicating strong ion-solvent interactions. Furthermore the B values gradually increased with rise in concentration and temperature indicating the structure making capacity of amlodipine besylate in both solvents of study.

M (mol/kg)	Intrinsic viscosity (Ψ) (dm ³ .mol ⁻¹)						
	293.15 K	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
0.0008	1.0640	1.3512	1.4498	1.6576	1.8694	2.7979	2.8739
0.0010	1.5436	1.9098	2.0847	2.4904	2.8838	3.2595	3.4441
0.0012	1.7974	2.1194	2.2550	2.8571	3.0794	3.4791	3.7382
0.0016	2.0310	2.4275	2.5733	3.3208	3.4928	3.9193	4.1965
0.0020	2.2673	2.5494	2.7642	3.6112	3.9540	4.2851	4.6723
0.0025	2.4755	2.8404	3.7083	4.0264	4.6846	4.7969	5.1893
0.0040	3.0297	3.4299	3.9363	5.0120	5.2102	5.9648	6.1439
0.0050	3.4540	3.9507	4.3141	5.5309	5.7138	6.5791	6.7462
0.0060	3.6625	4.3564	4.6838	5.9734	6.2020	7.1102	7.4971
0.0080	4.1600	4.9375	5.3435	6.2358	7.0284	7.5034	8.4571

Table 9: Data of viscosity (η) for the solution of amlodipine besylate in methanol at all working temperatures

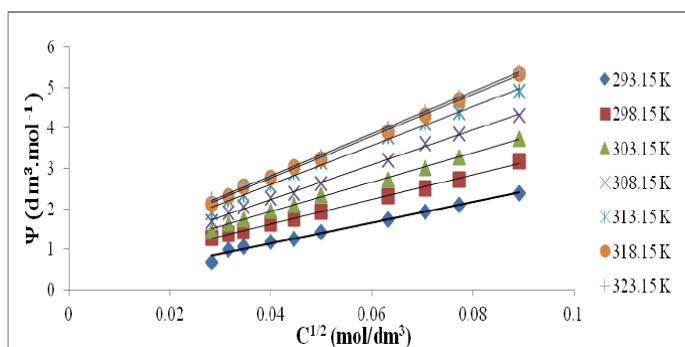


Figure 5: Graph showing the linear trends between the intrinsic viscosity (Ψ) and square root of molar concentration ($C^{1/2}$) for the aqueous solution of amlodipine besylate at all working temperatures (K)

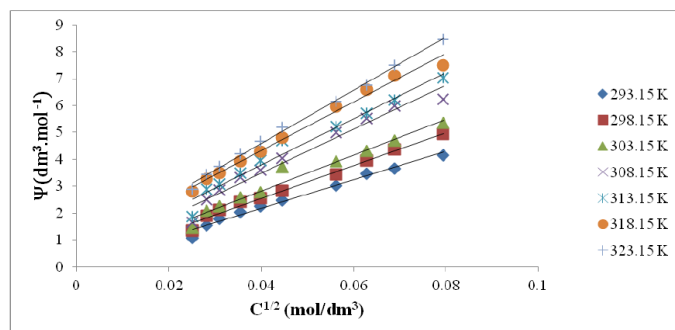


Figure 6: Graph showing the linear trends between the intrinsic viscosity (Ψ) and square root of molar concentration ($C^{1/2}$) for the solution of amlodipine besylate in methanol at all working temperatures (K)

Temperature (K)	Data of Drug in Distilled Water		Data of Drug in Methanol	
	A	B	A	B
293.15	0.120	25.59	0.049	53.06
298.15	0.411	30.40	0.104	61.36
303.15	0.477	36.35	0.169	67.08
308.15	0.540	42.64	0.214	82.64
313.15	0.665	48.55	0.354	87.19
318.15	0.698	51.93	0.490	95.47
323.15	0.744	52.12	0.627	100.9

Table 10: Data of A and B parameters of Jones-Dole equation for the solutions of amlodipine besylate in distilled water and methanol at all working temperatures

CONDUCTOMETRIC STUDIES

Electrical conductivity plays an important role in elucidating the nature of solution and is in good agreement to judge the intermolecular interactions. The value of electrical conductivity in present study was found to be increased with concentration as well as temperature as indicated by following table and graphical representations. This is attributed to the fact that no. of charge carriers get enhanced with

concentration and the mobility of the carriers get enhanced with temperature. Both of these factors are related to increased value of electrical conductivity. Furthermore the increased values of conductivity are not so prominent with the rise in temperature and concentration probably due to intermolecular interactions that offer steric hindrance to the mobility of charge carriers. This trend was followed by both solvents under study.

m (mol/kg)	Specific Conductance (κ) ($\mu\text{S. cm}^{-1}$)						
	293.15 K	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
0.0008	113	145	170	237	315	356	397
0.0010	122	160	185	255	335	381	425
0.0012	131	171	197	271	358	405	462
0.0016	149	196	223	308	399	462	507
0.0020	165	217	248	335	433	511	563
0.0025	186	245	284	375	483	569	628
0.0040	255	319	377	485	615	739	829
0.0050	297	372	436	557	712	851	958
0.0060	340	425	510	637	801	969	1081
0.0080	419	534	625	779	985	1195	1345

Table 11: Data of specific conductance (κ) for the aqueous solutions of amlodipine besylate at all working temperatures (K)

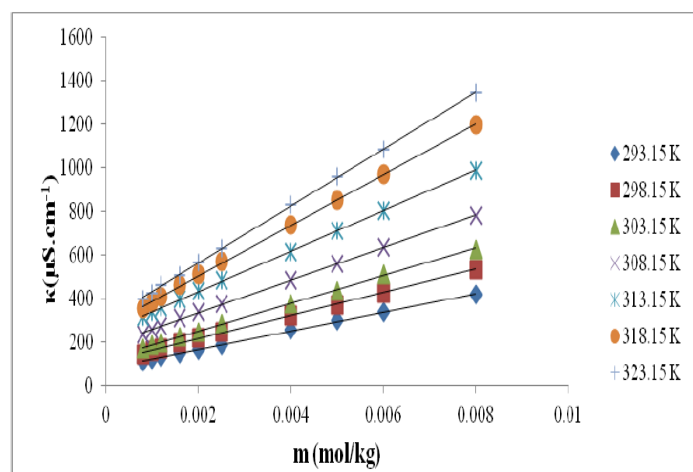


Figure 7: Graph between molality (m) and specific conductance (κ) for the solutions of amlodipine besylate in methanol at all working temperatures (K)

m (mol/kg)	Specific Conductance (κ) ($\mu\text{S.cm}^{-1}$)						
	293.15 K	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
0.0008	119	149	189	229	259	305	351
0.0010	149	186	223	260	297	355	395
0.0012	165	217	254	301	339	397	445
0.0016	195	259	303	372	405	480	532
0.0020	239	318	369	435	479	569	625
0.0025	277	386	425	507	553	675	731
0.0040	425	568	675	769	829	948	1057
0.0050	536	680	804	928	1012	1156	1285
0.0060	624	785	936	1072	1198	1329	1477
0.0080	809	1018	1205	1375	1497	1697	1897

Table 12: Data of specific conductance (κ) for the solutions of amlodipine besylate in methanol at all working temperatures (K)

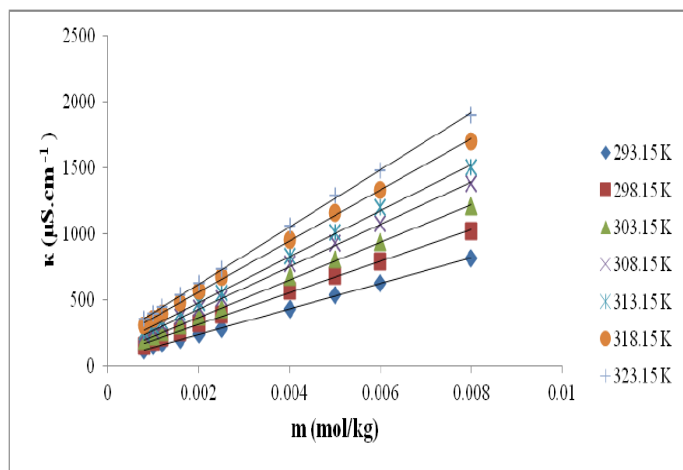


Figure 8: Graph between molality (m) and specific conductance (κ) for the solutions of amlodipine besylate in methanol at all working temperatures (K)

CONCLUSION

Amlodipine besylate is regarded as an important antihypertensive agent. Due to its immense importance there is a need to have insight of its mechanism of action across biological membranes. Volumetric, viscometric and conductometric studies of it are of significant importance in evaluating the solute-solvent interactions and transport of drug across body membranes. Additionally variation of discussed parameters with temperature shows importance regarding the storage conditions of drug. Usefulness and benefits of the study can be further enhanced by studying the described parameters in solution having pH in close collaboration of human body.

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