

Determination of Diffusion Coefficients of L-(+) - Tartaric Acid in Water and Sodium Hydroxide Solutions at 25°C

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Abstract

Food additives play a vital role in today's bountiful and nutritious food supply and their migration in water is a diffusion process governed by Fickian diffusion laws. The study looked into the rate of diffusion of L-(+) - tartaric acid at 25°C in water and in NaOH solution of different concentrations. L-(+) - tartaric acid and its salts are approved as food additives, with acidulant, antioxidant synergist, buffering and sequestrating functions. The objective of the work was to determine the diffusion coefficient using spectrophotometric method and moving boundary method and to compare the diffusion coefficients obtained with those calculated from limiting ionic conductance at infinite dilution. The absorbance at different height levels (x) were measured at a given time and at specific wavelength. The boundary heights (x) at a time t and concentration were also recorded. The diffusion coefficients were obtained from slopes of the $\ln A$ vs x^2 and x^2 vs t graphs which are governed by the Fick's second law and square-root relationship respectively. The spectrophotometric diffusion coefficient ranged from -2.5445×10^{-05} to -7.96674×10^{-06} cm^2/sec and for moving boundary method were between 1.2974×10^{-04} and 7.4908×10^{-05} cm^2/sec for L-(+) - tartaric acid. Spectrophotometric diffusion coefficients values were in a close agreement with the expected D_o value unlike moving boundary which gives reasonable but rough estimation of diffusion coefficients in the range of $x \times 10^{-05}$ cm^2/sec . Spectrophotometric method is also preferred because of its practicability and applicability under ordinary laboratory conditions which is in agreement with the principles for the establishment of the codex method of analysis.

Key Words: Diffusion, Coefficient, Absorbance, Boundary Height, Food Additive

Introduction

The use of food Additives has become more prominent in recent years due to the increased production of prepared, processed and convenience foods. Additives are used for flavor and appeal, food preparation and processing, freshness and safety. However, consumers and scientists across the globe have raised questions about the necessity and safety of these substances. Food and food additive are assimilated in the body basically through diffusion and this mass transfer in food systems is described by Fick's second law, which in many cases can be analytically solved if experimental data, as well as initial, and boundary conditions are provided, in order to yield an effective mass transfer coefficient (Danae *et al.*, 2011). There are several food additives. In this paper, L (+)-tartaric acid has been examined.

Though the intake of food and food additives is basically a diffusion process, less research has been done to quantify levels and rate of diffusion. The quantitative measurements of the rate at which a diffusion process occurs are usually expressed in terms of diffusion coefficient (D) which is the rate of transfer of diffusing substance across unit area of a section, divided by the space gradient of concentration at the section (Crank, 1975). Various methods of determining D have been reported (Irina, 1985, 1980). The Taylor dispersion method (Cussler, 1997) is the most commonly used method for determining molecular D at infinite dilution due to its versatility and experimental simplicity. An optical technique, the laser-induced grating method, has been used by Butenhoff *et al.* (1996) to determine diffusion of concentrated solutions.

The author used simple-rapid UV/VIS spectrophotometric method and moving indicator method to obtain D data of L (+)-tartaric acid from subsequent graphical plots using boundary heights and absorbance and compare these experimental Ds in relation with those calculated from conductance at infinite dilution (D_0). The obtained Ds formed a set of data base for reference and provided an alternative method for laboratories not equipped with expensive materials in the aim to preserve security of the consumers, especially in the developing countries.

L (+)-Tartaric acid (E334) (Figure 1) is listed in Annex I of European Parliament and Council Directive No 95/2/EC and is a generally permitted additive, allowed at *quantum satis* in all foods except those for which there is a defined list of permitted additives. Of this latter group, it is permitted in cocoa and chocolate products at a

maximum permitted level of 0.5%, and in jams, jellies, marmalades, canned and bottled fruit and vegetables and fresh pasta at *quantum satis*. It may also be present in biscuits and rusks as weaning foods for infants and young children in good health, at a maximum level of 5 g/kg residue.

Tartaric acid occurs naturally in fruits and wine (120-180 mg/100 ml) and L-tartaric acid and its salts are approved as food additives, with acidulant, antioxidant synergist, buffering and sequestrant functions. L-Tartaric acid is soluble in water, methanol, ethanol, propanol, ether and glycerol, and is insoluble in chloroform (The Merck Index, 13th ed).

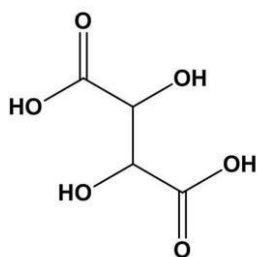


Figure 1. Structure of Tartaric Acid

Materials and Methods

Apparatus

Spectrophotometric measurements were carried out using Spectro-UV11 spectrophotometer and UV-1600 series spectrophotometers with 1cm matching quartz cell. Crystal pellets were made using Infrared crystal pellet maker. The pH values were determined using pH 211- Microprocessor pH meter.

Reagents

L-(+) - Tartaric acid and sodium hydroxide were purchased from Sigma-Aldrich. Phenol red and bromothymol blue were supplied by Kobian Scientific. All the reagents were of analytical grade and were used without further purification. Redistilled and deionized water was used in preparing solutions.

Preparation of standard solutions. A stock solution of 0.1M NaOH was prepared by dissolving 0.4g in 1litre of redistilled water. Concentrations (0.002-0.008M) were made by suitable dilution of the stock. Lower concentrations (0.002-0.010M) were made by

suitable dilution of stock. All solutions were sonicated for ten minutes and all solutions were incubated at 25°C. Fresh solutions were prepared for each set of experiments.

Preliminary sample preparation. Solutions of varied concentration of each of the five food additives (0.002-0.010M) were prepared, and were scanned using UV1600 to obtain their respective maximum absorption wave length. This was aimed at determining if the samples obey Beer Lambert Law. Crystal pellets of L-(+) - Tartaric acid were made by pressing 0.25g of each additive using the infrared crystal. The pH of L-(+) - Tartaric acid at 25° C was 6.21 and phenol red and bromothymol blue were considered appropriate indicators for this study.

Procedure

UV/VIS spectrophotometry. Using a sharp pencil a reference line was drawn on the ground glass side of 1cm quartz sample cell along the edge of the spectrophotometer. Four other horizontal lines were drawn at 2mm intervals parallel to up and below the reference line. The temperature of the sample and the reference compartment were kept constant at $25.0 \pm 0.1^\circ\text{C}$.

Three millilitres of distilled water was pipeted into each of the samples and reference cells and then the contents were allowed to stand for one hour to gain thermal and mechanical equilibrium. The maximum absorption wavelengths (210nm) was set on spectrophotometer and then the crystal agiven mass of L-(+)-Tartaric acid was dropped into the sample cell. At an interval of one hour, the absorbance at 5 different levels were recorded. These readings were obtained by sliding the sample cell up and down and aligning the pencil mark on the ground glass side with the top edge of the partition between the sample and the reference compartments. The absorbance of the sample at infinity were obtained from a homogenous solution of the sample cell.

Moving boundary method. Two set of 0.002M to 0.010M NaOH solutions were prepared and to 3mls of each solution in a calibrated 1cm plastic cuvette, two drops of bromothymol blue indicator were added to one set, mixed and allowed to stand for over night while closed using a fitted stopper at 25°C so as to attain thermal and mechanical equilibrium. Two drops of phenol red indicator was used in the remaining set after

which sample pellet of tartaric acid of a given weight was dropped to each cuvette and time recorded. At different time intervals, boundary heights between basic and acidic solution was recorded. This was also done using phenol red indicator. The entire procedure was repeated using citric acid.

Results

Spectrophometric Method

Records of measured absorbance for each of L (+)-tartaric acid samples are shown in Table 1 and Table 2.

Table 1. Data of Transmittance (T) and Absorbance (A) of L (+)-Tartaric Acid with Respect to Time (min) and Boundary Height x (cm)

Mass of Tartaric acid: 0.0025 g													
Time (min)		20			60			120			170		
x	x^2	T	A	ln A	T	A	ln A	T	A	ln A	T	A	ln A
1.0	1.00	97.5	0.011	-	94.7	0.024	-	75	0.125	-	57.7	0.24	-
				4.5099			3.7297			2.0794			1.4271
0.8	0.64	91.2	0.040	-	91.7	0.037	-	62.1	0.208	-	38.9	0.526	-
				3.2189			3.2968			1.5702			0.6425
0.6	0.36	58.1	0.237	-	88.0	0.055	-	45.7	0.342	-	15.0	0.834	-
				1.4397			2.9004			1.0729			0.1815
0.5	0.25	43.0	0.368	-	76.1	0.119	-	25.4	0.600	-	7.5	1.148	0.1380
				0.9997			2.1286			0.5108			
0.4	0.16	25.2	0.604	-	57.9	0.238	-	11.2	0.966	-	2.6	1.658	0.5056
				0.5042			1.4355			0.0346			
0.3	0.09	4.4	1.401	0.3372	35.2	0.456	-	4.9	1.344	0.2957	1.0	2.189	0.7834
							0.7853						
0.2	0.04	6.0	1.252	0.2247	17.2	0.772	-	1.2	2.086	0.7352	0.8	2.359	0.8582
							0.2588						
∞		7.2	1.164										

Table 2. Data of Transmittance (T) and Absorbance (A) of L (+)-Tartaric Acid with Respect to Time (min) and Boundary Height x (cm)

Mass of Tartaric acid: 0.0039 g										
Time (min)		480			720			800		
x	x^2	T	A	ln A	T	A	ln A	T	A	ln A
0.8	0.64	2.5	1.603	0.4719	1.3	1.884	0.6334	0.1	1.991	0.6886
0.6	0.36	0.4	2.452	0.8969	1.2	2.727	1.0032	0.2	2.679	0.9854
0.4	0.16	0.2	2.812	1.0339	0.1	2.886	1.0599	0.2	2.821	1.0371
0.2	0.04	0.1	2.860	1.0508	0.1	2.903	1.0657	0.1	2.863	1.0519
∞		7.2	1.164							

The center beam for the sample compartment was 1.40 cm from the inside of the bottom of the holder. This gave the maximum value of the monitoring distance x - from the plane where diffusion began - to be equal to 1.40 cm. The displacement of the cell upwards was limited to 1cm because greater displacements showed anomalies of the cells and refracted source beam.

The experimental conditions are for an amount of diffusing substance deposited at the time $t = 0$ in the plane $x = 0$ approximating to those of a reflection boundary.

Hence at

$x = 0, c = c_0$; but for all values of $x > 0, c = 0$. For these conditions, Moore $t = 0,$

(1955) shows that

$$\frac{c}{c_0} = Dt^{1/2} e^{-x^2/4Dt} \quad 1$$

The diffusion coefficient was obtained by plotting, for a given time, graph of $\ln A$ versus x^2 . For dilute solutions, the amount of light absorbed at a specific wavelength is directly proportional to the concentration of the solution. Since absorbance A is directly proportional to concentration, graphs $\ln A$ versus x^2 gives straight line graphs

$$(4Dt)^{-1}$$

whose slope equals . These plots are given in Figure 3 and Figure 4 using the data in Table 1 and Table 2.

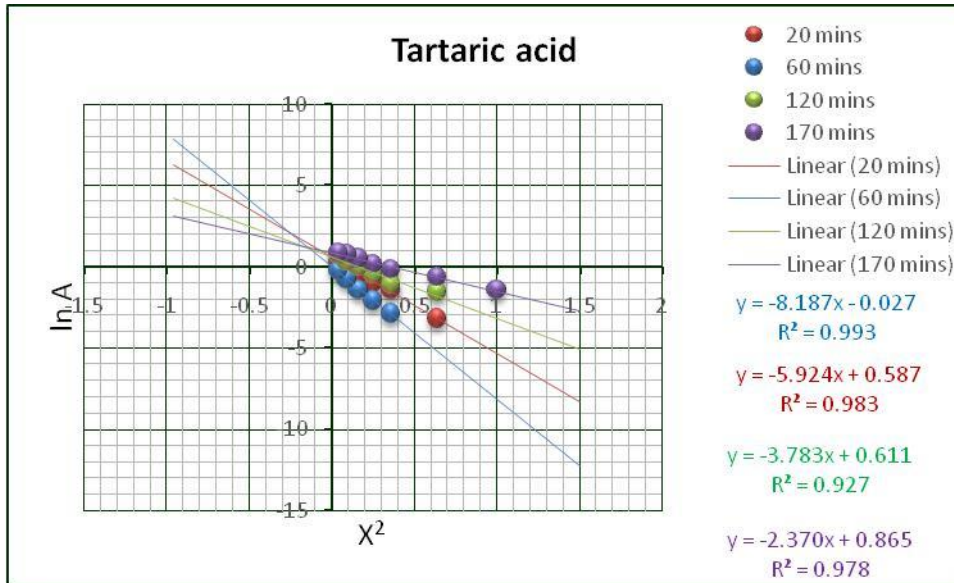


Figure 3. Graph of Natural Log of Absorbance of 0.0025g Tartaric Acid against Square Boundary Height (x^2) at 210nm λ

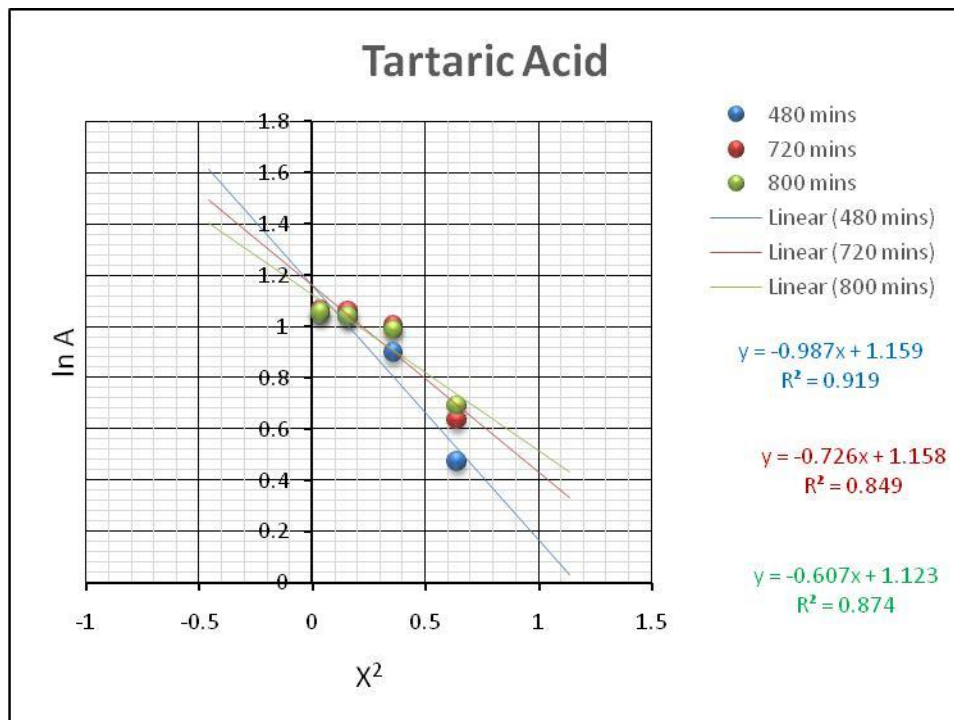


Figure 4. Graph of Natural Log of Absorbance of 0.0039g Tartaric Acid against Square Boundary Height (x^2) at 210nm λ

The calculated D values from spectrophotometry for L (+)-tartaric acid at their respective time and wavelength are given in Table 3 together with their respective correlation value (R^2), standard deviation (SD), variance and relative standard deviation (RSD).

Table 3. D, Correlation Value (R^2), Standard Deviation (SD), Variance and Relative Standard Deviation (RSD) Values of L (+)-Tartaric Acid at 25°C; UV-Vis Spectroscopy

UV-Vis spectroscopy								
Mass	Time	Equation of the line		R^2	D	SD	Variance	RSD
0.0025	20	y = -8.1876x	- 0.0279	0.9937	-2.5445E-05	0.56	0.31	1.006
	60	y = -5.9245x	+ 0.5872	0.9839	-1.17216E-05	0.27	0.07	1.147
	120	y = -3.7836x	+ 0.6115	0.9275	-9.17703E-06	0.71	0.51	0.877
	170	y = -2.3706x	+ 0.8651	0.9788	-1.03391E-05	0.81	0.66	0.638
0.0039	480	y = -0.987x	+ 1.1595	0.9197	-8.79489E-06	0.58	0.33	0.239
	720	y = -0.7264x	+ 1.1585	0.8496	-7.96674E-06	0.48	0.23	0.186
	800	y = -0.6076x	+ 1.1231	0.8741	-8.57198E-06	0.41	0.16	0.156

Moving Boundary Method

Table 4 and Table 5 show typical data of x of L (+)-tartaric acid in 0.002M-0.010M NaOH solutions with time.

Table 4. Boundary Height x (cm) for L (+)-Tartaric Acid in 0.002M to 0.01M NaOH Solutions

Indicator used: Bromotymol blue										
Mass (g)	0.0041		0.0043		0.0047		0.0049		0.0050	
Concentration. (M)	0.002		0.004		0.006		0.008		0.010	
Time (min)	X	x^2	X	x^2	X	x^2	X	x^2	X	x^2
10	0.40	0.1600	0.39	0.1521	0.28	0.0784	0.30	0.0900	0.30	0.0900
30	0.55	0.3025	0.48	0.2304	0.42	0.1764	0.40	0.1600	0.40	0.1600
50	0.60	0.3600	0.62	0.3844	0.60	0.3600	0.55	0.3025	0.51	0.2601
80	0.82	0.6724	0.72	0.5184	0.70	0.4900	0.70	0.4900	0.65	0.4225
110	0.90	0.8100	0.80	0.6400	0.75	0.5625	0.72	0.5184	0.69	0.4761
140			1.00	1.0000	0.90	0.8100	0.84	0.7056	0.76	0.5776
170			1.02	1.0404	0.98	0.9604	0.96	0.9216	0.87	0.7569
210					1.10	1.2100	1.04	1.0816		

250
270

1.15 1.3225 1.08 1.1664
1.15 1.3225

Table 5. Boundary Height x (cm) for L (+)-Tartaric Acid in 0.002M to 0.01M NaOH Solutions

Indicator used: Phenol red										
Mass (g)	0.0040		0.0044		0.0048		0.0048		0.0050	
Concentration (M)	0.002		0.004		0.006		0.008		0.010	
Time (min)	X	X ²	X	X ²	X	X ²	X	X ²	X	X ²
10	0.45	0.2025	0.45	0.2025	0.40	0.1600	0.30	0.0900	0.30	0.0900
30	0.55	0.3025	0.50	0.2500	0.55	0.3025	0.55	0.3025	0.40	0.1600
50	0.75	0.5625	0.65	0.4225	0.63	0.3969	0.60	0.3600	0.56	0.3136
80	0.88	0.7744	0.80	0.6400	0.79	0.6241	0.70	0.4900	0.69	0.4761
110	1.10	1.2100	0.85	0.7225	0.83	0.6889	0.80	0.6400	0.76	0.5776
140			1.02	1.0404	0.92	0.8464	0.84	0.7056	0.87	0.7569
170			1.20	1.4400	1.10	1.2100	1.00	1.0000	0.93	0.8649
210					1.26	1.5876	1.10	1.2100		

It was observed that the rate of rise of L (+) - tartaric acid was proportional to time and was dependent on the concentration of NaOH solution and the mass of the sample (food additive). The plots of the square boundary heights against time gave straight line passing close to the origin (Figure 4 and Figure 5). The rate at which food additives diffused in NaOH decreased with increased concentration of NaOH which agreed with the expectation of diffusion with chemical reaction. The results also agree with the square root relationship for diffusion into a semi-infinite medium involving the dimensionless parameter (Crank, 1975).

$$\frac{x}{2\sqrt{Dt}} \quad \text{Expression (1)}$$

In two aspects:

- The distance obtained by any given concentration (indicated by the sharp blue/colorless boundary in our experiment) was proportional to the square root of the time.
- The time needed for any point to reach a given concentration was proportional to the square of its distance from the surface where diffusion begins.

The experimental conditions are for an amount of diffusing substance deposited at $x = 0$ in the plane approximating to those of a reflection boundary. Bockris and Reedy (1970) have reported that these experimental conditions are expressed as

$$n = \frac{n_{total}}{(\pi Dt)^{1/2}} e^{-x^2/4Dt} \quad (2)$$

or

$$\frac{n}{n_{total}} = \frac{1}{(\pi Dt)^{1/2}} e^{-x^2/4Dt} \quad (3)$$

Where n is the number of ions at a distance x at a time t and n_{total} is the total number of ions placed in the plane $x = 0$ at the time $t = 0$. In the present work, n and n_{total} are constant. Therefore, the plot of $\ln \frac{n}{n_{total}}$ with t gives a straight line whose slope equals $-4D'$, where D' is the D at a given acid concentration. Hence:

$$D = \frac{\text{slope}}{4} \quad (4)$$

The masses of the samples used exceeded molar mass of NaOH solutions which allowed the square root for diffusion into semi-infinite medium involving the dimensionless expression (Expression 1) to be observed.

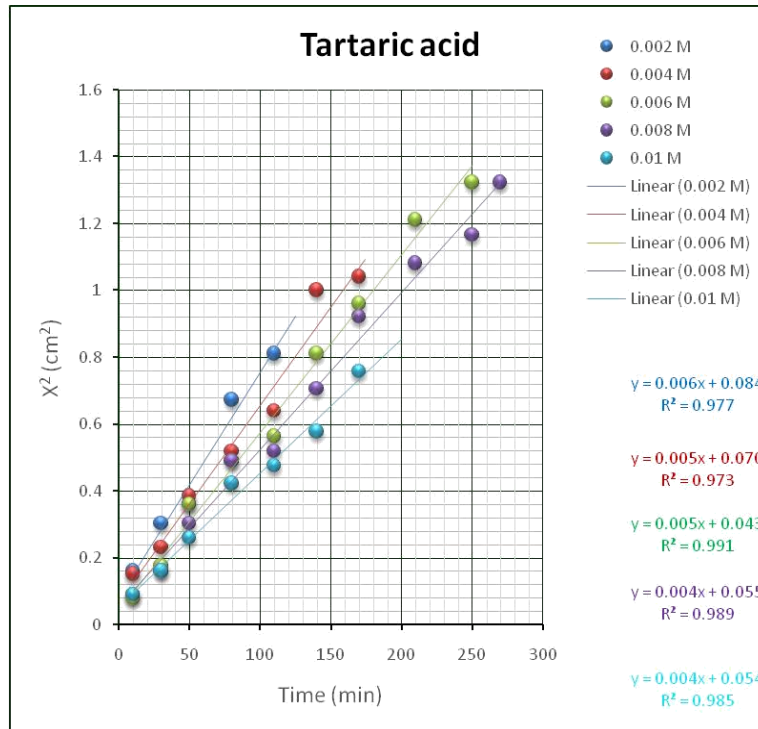


Figure 4. Graph of Square Boundary Height x^2 (cm²) of L (+)-Tartaric Acid against Time (min) NaOH Solution using Bromothymol Indicator

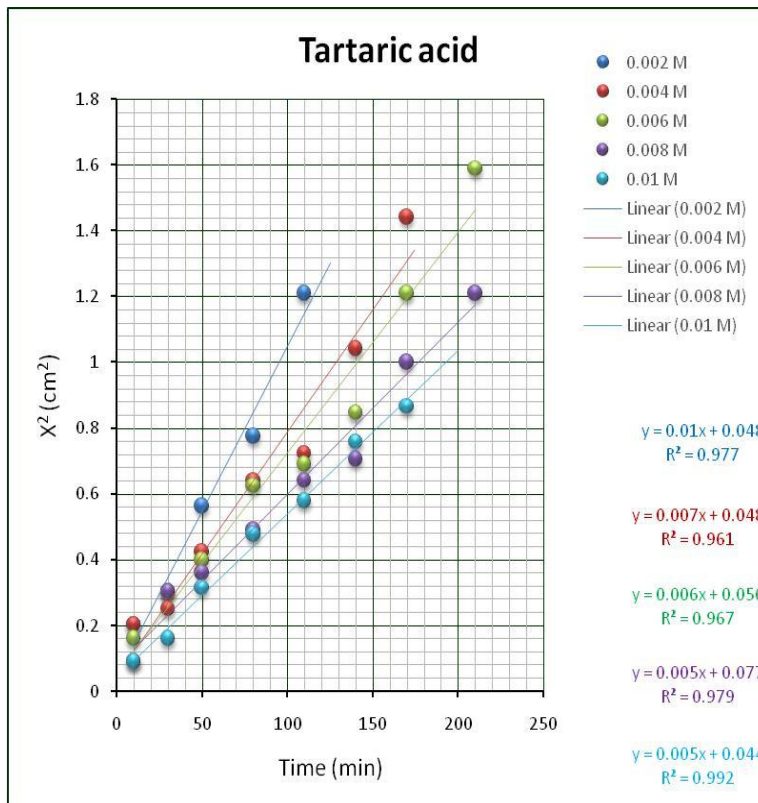


Figure 5. Graph of Square Boundary Height x^2 (cm²) of L (+)-Tartaric Acid against Time (min) in NaOH Solution using Phenol Red Indicator

The calculated D values from the plot of x^2 with time for L (+)-tartaric acid with respective NaOH concentrations are given in Table 6 to Table 7 together with their R^2 , SD, variance and RSD values.

Table 6. D, R^2 , SD, Variance and RSD Values of L (+)-Tartaric Acid at 25°C using Bromothymol Blue Indicator

Bromothymol blue indicator							
Mass	Equation of the line	R^2	Concentration	Calculated D	SD	variance	RSD
0.0041	$y = 0.0067x + 0.0845$	0.9770	0.002	7.4908E-05	0.20	0.04	0.311
0.0043	$y = 0.0059x + 0.0709$	0.9731	0.004	9.3287E-05	0.24	0.05	0.336
0.0047	$y = 0.0053x + 0.0437$	0.9910	0.006	1.0263E-04	0.29	0.08	0.389
0.0047	$y = 0.0047x + 0.0553$	0.9893	0.008	1.0510E-04	0.29	0.08	0.377
0.0049	$y = 0.004x + 0.0543$	0.9859	0.010	1.0000E-04	0.20	0.04	0.339

Table 7. D, R^2 , SD, Variance and RSD Values of L (+)-Tartaric Acid at 25°C using Phenol Red Indicator

Phenol red indicator							
Mass	Equation of the line	R^2	Concentration	Calculated D	SD	variance	RSD
0.0040	$y = 0.01x + 0.0489$	0.9777	0.002	1.1180E-04	0.25	0.06	0.347
0.0044	$y = 0.0074x + 0.0486$	0.9612	0.004	1.1700E-04	0.27	0.07	0.347
0.0048	$y = 0.0067x + 0.0560$	0.9672	0.006	1.2974E-04	0.28	0.08	0.351
0.0048	$y = 0.0052x + 0.0777$	0.9793	0.0013	1.1628E-04	0.25	0.06	0.348
0.005	$y = 0.005x + 0.0448$	0.9926	0.0012	1.2500E-04	0.23	0.05	0.365

Discussion

Comparing the values of obtained (D values from spectrophotometry) with the diffusion coefficients for strong electrolytes at infinite dilutions D_0 , obtained using equivalent cationic and anionic limiting conductance values (Vanyšek Petr, 1993) of L (+)-tartaric acid at infinite dilution. Using equation 5, D_0 of was determined using respective values in Table 8.

$$D_0 = \frac{8.936 \times 10^{-10} T (\gamma_1 + \gamma_2) \lambda_1^{\circ} \lambda_2^{\circ}}{\gamma_1 Z_1 (\lambda_1 + \lambda_2)} \quad (5)$$

Where T is the absolute temperature, γ_1 and γ_2 are the number of the cations and the anions from the dissolution of one molecule of L (+)-tartaric acid, z_1 is the cationic charge and λ_1 and λ_2 is the equivalent cationic and anion limiting conductance.

Table 8. D_0 Values Calculated from Limiting Conductance at 25°C

Food additive	λ_{+}°	λ_{-}°	γ_1	γ_2	Z	calculated D_0 (cm^2/sec)
Tartaric acid	394.65	59.6	2	1	1	2.0683×10^{-05}

The obtained D values were within the range of expected D_0 value. The average of D values at 20 and 60 minutes citric acid and L (+)-tartaric acid was $1.85833 \times 10^{-05} \text{ cm}^2/\text{sec}$ which is in close to the expected $2.0683 \times 10^{-05} \text{ cm}^2/\text{sec}$ (D_0).

The moving boundary method involved the use of dilute solutions which is easier to understand in physical terms; the experimental D were remarkably low and were in the range of 10^{-4} and $10^{-5} \text{ cm}^2/\text{sec}$ (Table 6 and Table 7) which was in agreement with what Cussler (2009) observes, that diffusion coefficients in liquids cannot be reliably estimated and cluster around a value $10^{-5} \text{ cm}^2/\text{sec}$. Most D values (Table 6), at low concentration, fall close to $10^{-5} \text{ cm}^2/\text{sec}$, with exception of Table 7 which could be due to formation of macromolecules between their dissociated ions of L (+)-tartaric acid with phenol red indicator altering D. Different masses of each sample were used and it was observed that moving boundary method is sensitive to weight within the base matrix. The D varied with molecular weight of the diffusing L (+)-tartaric acid. With time, the moving boundary becomes less distinct, fades away and the boundary heights become more of approximate values which in turn affect the certainty of D values.

Linearity was assessed by repeated measurements ($n = 5$) for spectrophotometric method and ($n = 6$) for indicator method. Acceptability of linearity of data was judged by examining the correlation coefficient (R^2) of the graphs. A R^2 values greater than 0.950 was considered sufficient to demonstrate linearity of the method. The calibration curves were all linear except Figure 4 and R^2 values ranged between 0.9275 and 0.9937. The equation of the regression lines are given in each plot graph and in Table 3, Table 6 and Table 7.

The precision was investigated through repeatability and reproducibility. The analysis was repeated six times in the same day. The calculations of the respective standard deviation (SD) and relative standard deviation (% RSD) gave low values (Table 3, Table 6 and Table 7) indicating acceptable level of precision. The % RSD are also less than 2% as required by United States Pharmacopeia and International Conference on Harmonization guideline.

The accuracy was assessed by comparing the values of D obtained from UV-Vis spectrophotometry and moving boundary method with D_0 values and unlike moving boundary method, UV-Vis spectrophotometry D values were in close agreement with D_0 values.

Errors and Assumptions

In the calculation, D has been assumed to be independent of the concentration of diffusing solution since equation 4.1 applies only when D is independent of concentration. There was a marked variation in absorbance at infinity brining variation

x

of C_0 values at $x = 0$, an evidence that D varies with concentration. The calculations also assumed viscous friction in pure water and the interaction forces between water molecules and solvent-ion and ion-ion interactions which could increase or decrease the D values.

The initial disturbance due to the presence due to the dropping of the crystals and the convective effects of the cells was neglected. The sliding of the sample cells up and down caused no significant side disturbance. The used sample pellets/crystals were obtained by mechanically breaking the large crystals and it was difficult to obtain samples of equal masses.

Conclusion

This work describes a simple, rapid and valid spectrophotometric method that gives D values that are close to those calculated from limiting conductance within experimental error. The diffusion process was dependent on time. For moving boundary method, D values varied inversely with concentration of NaOH solution and were sensitive to the molecular weight of the sample used. Though moving boundary method,

D values could not be reliably estimated, they cluster around $10 \times 10^{-5} \text{ cm}^2/\text{s}$, which gives reasonable but rough estimation of expected diffusion coefficients. Spectrophotometric method was preferred because of its practicability and applicability under ordinary laboratory conditions which is in agreement with the principles for the establishment of the codex method of analysis and the method was specific, linear, accurate and stable and could be adopted as an official method for routine analysis of food additives.

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