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Development of an Equation for the Determination of Water Content in Alcohol Employing Certain Physical Properties

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Abstract

Food drinks, drugs and fuels are normally produced in standard ratios of the components making up the final products that give their best performances. Once the standard mixing ratio of the components of any standard liquid mixture is altered, the liquid product gives less performance and in some cases the liquid product causes accidents. Most of the machines used in the analyses of the components of liquids employ chemical analyses/properties of the liquids, which is always cumbersome and time consuming. Simple, quick and reliable techniques which use physical analyses/properties of the liquid can also be employed for the determination of solute content in a solution. A viscometer was designed and constructed and used to determine the time of flow, reduced time ratios, viscosity coefficients, reduced viscosity ratios of distilled water, alcohols, and their mixtures. Simple techniques were then developed that can determine the type and percentage volume concentration of water in alcohols at 308 K. A general equation was developed for the percentage water concentration in alcohol with the temperature fluctuations slightly above or below the calibration temperature, Tcal = 308 K as , where   is the reduced time ratio or reduced viscosity ratio, T is the Kelvin temperature at which   is determined, and D and S are values from the calibrated curve of that liquid mixture. For reduced time ratio, D = 0.821, S = 0.00142 for methanol (CH3OH) and D = 1.418, S = 0.00398 for ethanol (C2H5OH). For reduced viscosity ratio, D = 0.681, S = 0.00245 for methanol (CH3OH) and D = 1.261, S = 0.00475 for ethanol (C2H5OH). This equation was tested on ethanol collected from different sources and found to be much workable.

Introduction

Ethanol is largely used in Brazil as an automotive bio-fuel since the 1970s, (Lucas et al, 2008) and as a gasoline additive in an attempt at reducing petrol imports. In Brazil, it is obtained basically from the fermentation and subsequent distillation of sugar cane. Its production is regulated by a governmental agency, Brazilian National Agency of Petrol, Natural Gas and Bio-fuels. Hydrated ethanol (approximately 7% water volume) is obtained from distillation process and can be used directly on engines specially adapted for

ethanol or in dual-fuel (flex-fuel) engines, which can use both ethanol and gasoline. On the other hand, anhydrous ethanol, obtained from further dehydration of distilled ethanol, is blended (roughly 20% in volume) to all gasoline fuel sold in Brazil. Unfortunately, several dishonest dealers sell fake mixtures in disagreement with the regulations, which can cause serious damages to engines. The most common adulteration is to add extra water to hydrated ethanol; or add extra anhydrous ethanol to the gasoline. In case of gasoline, other organic solvents have also been used for adulteration; (Santos, 2003) some of them produce carcinogenic vapors. Car manufacturers also suffer from this problem since they provide a minimum one-year warranty.

In order to determine the concentration of water in an alcohol, we can rely on either chemical or physical properties. However, techniques using chemical analysis are time consuming and expensive. There is an increasing interest in proper estimation of impurities present in Active pharmaceutical ingredients (APIs). There are various methods of detecting and characterizing both components and concentrations of the impurities such as (Sanjay et al, 2007), Thin Layer Chromatography, (TCL), High Pressure Liquid Chromatography, (HPLC),

combination of HPLC and TL, (HPTLC), Atomic absorption spectroscopy, (AAS), Mass Spectroscopy, (MS), Nuclear Magnetic Resonance, (NMR), Fourier Transform Ion Cyclotron Mass Spectroscopy, ((FTICR-MS), Gas chromatography, (GC), Tandem Mass spectrometry, (TMS) among other techniques. Sanjay et al, (2007) have stated guidelines and designated approaches for isolation and identification of process-related impurities and degradation of pharmaceutical products, using MS, NMR, HPLC, Fourier, FTICR-MS and TMS, etc. Most of these techniques have capabilities of detecting impurities in ppm (parts per million) and some (e.g., GC) in ppb (parts per billion) ranges and thus are highly accurate and suitable for impurity estimations in pharmaceuticals where in majority cases detection of impurity below 0.05% is not necessary according to Internal Conference on Harmonization, (ICH) of technical requirements for registration of pharmaceuticals for human consumption. This translates to 1 mg a day in drugs consumed by average patient.

The above techniques are capable of estimating impurity concentrations both in solid and solutions. Most of these techniques require highly sophisticated equipment, experienced technologist and obviously

constant supply of electric power. Application of such techniques in African countries is still at a premature stage, especially because of cost-prohibitive and lack of trained man-power.

Physical properties of substances are perhaps the most important variables that enter into the design of different types of equipment. Thus, physical analysis can be conveniently employed to carry out the studies. The physical properties to be employed should be such that they are sensitive to the type of the liquid and the concentration of the solute or impurities in the liquid. Preliminary investigations reveal that combination of density and viscosity coefficients can be employed for this study. In many solvent liquids viscosity coefficient is sensitive to nature and concentration of impurities. Density can be rightly measured without much difficulty. Thus, the combination of density and viscosity can be uniquely employed to determine the nature of the liquid; the type of impurity in a known liquid solvent and the concentration of the impurity in the liquid. The apparatus required for the measurement of viscosity coefficient can be very economically constructed and operated.

The main focus in this study will be its application to determine the concentration of water in a known alcohol and how this is affected by change in temperature. Hence, techniques can be developed and readily be applied to distinguish between pure and contaminated alcohol. The viscosity method of estimating concentrations of known components as presented in this work has the capability of estimating concentration of components easily within 0.05 % with a larger tube (QP) in Fig.1 and a better optical time counting device (capable of measuring time accurately up to 100 ms), it should find application to estimation of water levels (around 0.1%) and concentrations of Immunization Virus, (IV) solutions. The present viscosity method requires very simple but reliable equipment that can be constructed and operated upon at the local level --

Methodology

In practice a liquid of known viscosity and density is used to calibrate the Ostwald viscometer. The viscosity of water is normally the reference and will compare the influence of mixing on viscosity. The viscosity of water at 25°C is 0.8904 centipoises (cP). The resistance offered by liquid to the shearing stress is called viscosity. In laminar flow, a molecule to go ahead must squeeze past pushing aside the neighboring molecules. To achieve this, the molecule flowing ahead must acquire a definite amount of energy which may be called activation energy, E_a. Then,

with Boltzmann factor, the number of molecules having such energy will be proportional to exp(-Ea/kBT), (Shiekh and De, 2002). The rate of flow and hence fluidity would depend on this number, and the viscosity is given by

$$\mu = B \exp\left(E_a / k_B T\right) \qquad (1)$$

where B is a constant for a given liquid, k_B is the Boltzmann constant; T is the absolute temperature and E_a the activation energy for viscous flow. This is the energy barrier that must be surmounted in order for a molecule to squeeze by its neighbors. A plot of lnμ vs. 1/T should be linear and with a slope equal to E_a/k_B, and intercept equals lnB. The activation energy of the original pure liquid should change when a solute molecule is added to it since the addition may change the bond strength of the given liquid or the solvent molecule as the solute molecule tends to occupy the neighboring positions. The change obviously will depend on the type and the concentration of the solute molecule. This forms the scientific basis of developing the present new technique of analyzing the extent of impurities in a given liquid.

Design of the Temperature Control Viscometer

The temperature control viscometer, which was designed and costructed to incorporate temperature control facility, is shown in Picture (1), (Dikko, 2013). QP is a vertical uniform cylindrical plastic tube of diameter 2.5 cm and length 6.0 cm. At the two ends are attached two narrow glass tubes of internal diameter 7 mm). There are horizontal fixed marks A and B on these tubes. At the bottom end of the lower tube was attached a uniform capillary tube of length, (LD) 30.8 cm, and the internal radius, r of the very uniform laser drilled capillary brass tube, is 0.6 mm, whereas the outer radius is 1.0 mm. While AQPB is held vertical, the capillary tube was enclosed inside and at the bottom of the cubical container, EFGHIJKM, filled with water, and fixed as horizontally as possible at right angle with the narrow glass tube, PBL.

Level-indicators were used to ensure constant vertical alignment of the apparatus during different runs of experiments. The apparatus also incorporates the temperature controller, T (PHYWE product) with a heating coil, C and a stirrer, S, and required temperatures were set using the variable switch, V on the temperature controller. The whole portion XAQPBLD was filled with the liquid solution under investigation, and the time t taken by the liquid to descend from A to B was recorded by a sensitive stop watch. If the experiment was to be carried at temperatures

above room temperature, the AC cord was fixed to electric power outlet and the required temperature set and left for 10 minutes before running the experiment.

 A 100°C calibrated thermometer was also inserted into the water to ensure accurate readings of the set temperature. As the upper tube was held vertically fixed, the solution descends and flows through the capillary tube and drops in a beaker. The time, required by the liquid to descend from mark A to B was

noted with the help of a stopwatch with accuracy of 0.01 second. The capillary tube chosen must be in good condition to avoid turbulence flow. The constructed temperature control viscometer is shown in Picture (2). The components used in the construction of the temperature controller viscometer shown in Plate I were: glass tubes, plastic tubes, temperature bath, capillary tubes, thermometer, lagging material, Level screws and level indicators.

Picture 1: Design of Temperature Control Viscosity
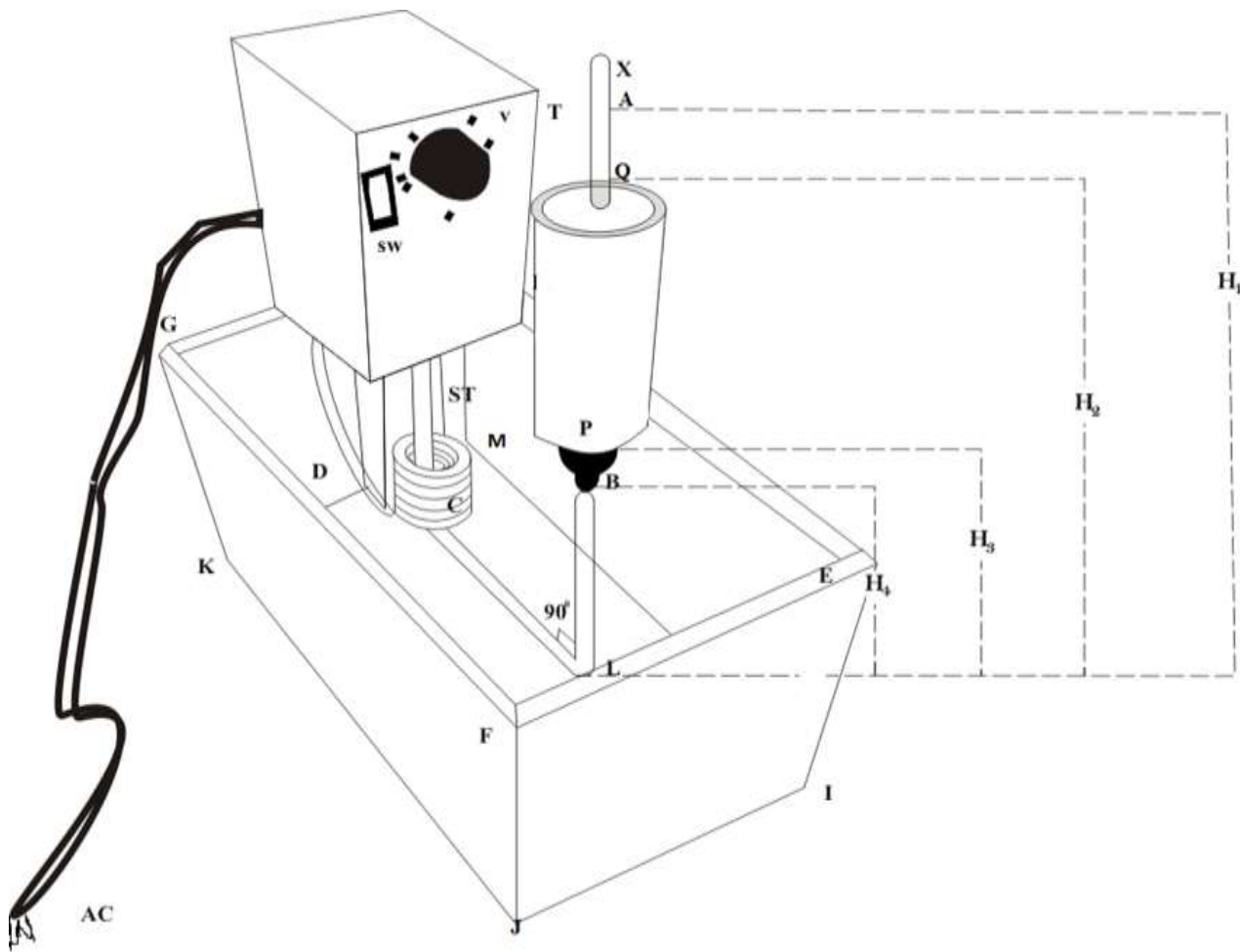
Picture 2: Plate I   Photograph of the Constructed Temperature Control Viscometer

The time taken by the liquid level to flow from mark A to B in Fig (1) is given by;

$$t = \frac{Q\mu}{\rho} , \tag{2}$$

Where,

$$Q = \left(a\ln\frac{H_1}{H_2} + b\ln\frac{H_2}{H_3} + c\ln\frac{H_3}{H_4} \right) \frac{8L}{\pi g r^4} \tag{3}$$

μ is the viscosity coefficient, ρ is the density, g is the acceleration due to gravity, H1, H2, H3 and H4 are the heights of the liquid levels as indicated in Fig (1), a, b, and c are cross sections of tubes AQ, QP and PB, (Shiekh, 2002). For inclination of the upper tube axis at an angle θ with the vertical, g in above equation should be replaced by gcosθ (assuming that the capillary still remains horizontal). Q remains constant, as long as the vertical inclination of the apparatus in Fig (1) is held constant for different sets of experiments conducted at a given place. With the same vertical inclination of the apparatus and at the same place (i.e., constant g), let t2 and t1 are the respective times for the solution under investigation and pure water (at a given temperature, T) to descend from mark A to mark B (Fig 1). Then from equation (2) we get

$$\mu_2 = \frac{\mu_1 \rho_2 t_2}{\rho_1 t_1} \qquad (4)$$

An equivalent measurement is carried on a liquid of known viscosity. The unknown viscosity is then calculated using equation (4). Similarly, the above theory is applicable to Fig. (1). As long as the vertical inclination of the apparatus Plate (1) is held constant for different sets of experiments conducted at a given place, (i.e., constant g), and t2 and t1 be the respective times for the solution under

investigation and pure water (at a given temperature, T) to descend from mark A to mark B (Fig 1), then equation (4) holds, (Sinha *et al,* (1969), Smith, (1972), and Shiekh, (2002)). The density ρ_2 can be easily determined using the simple relation of density equals mass/volume. ρ_1 at the given temperature can be easily known from a handbook of physical constant. For most purposes ρ_1 of distilled water can be taken as 1 kg/liter. The accuracy of determining μ_2 from equation (4) then depends on the accuracy of noting the time t_1 and t_2 and the accuracy of knowing μ_1. For manual observation, the inaccuracy in noting time t_1 and t_2 can be easily kept within ± 0.01 sec. From equation (4) we see that

$$\frac{t_2}{t_1} = \frac{\mu_2 \rho_1}{\mu_1 \rho_2} \qquad (5)$$

Note that the quantities on the right side equation (5) depend only on the physical properties of the solution and that of pure water under investigation, whereas both t_2 and t_1 depend also on the apparatus. Thus, the ratio t2/t1 (when precise vertical inclination is maintained throughout the experiments at constant temperature T) is a unique signature of the nature and concentration of the solute in the particular solvent and independent of the physical dimensions of the apparatus used and

the place where the experiment is carried out.

The change in activation energy, ΔE_a arising from different concentrations of the impurity (solute) in the mixture (solution) at constant temperature can be calculated. From equation (2) we can get the change in activation energy as,

$$\Delta E_a = K_B T \ln\left(\frac{\mu_2}{\mu_1}\right) \qquad (6)$$

For the effect of temperature on the determination of unknown concentration of impurity in a solution or mixture using a calibrated curve at a particular temperature, T_{cal}, we may use equation (6) to obtain μ at any other temperature, T. Using this equation, it can easily be shown that, for small variation of T around the calibration temperature T_{cal}, which may not affect activation energy, E_a of the liquids and that of pure water appreciably,

$$\left(\frac{\mu_2}{\mu_1}\right)_{T_{cal}} = l\left(\frac{\mu_2}{\mu_1}\right)_{T} \exp\left(T / T_{cal}\right) \qquad (7)$$

Substituting the value of (μ_2/μ_1) in equation (4) in equation (7), we get

$$\left(\frac{\rho_2 t_2}{\rho_1 t_1}\right)_{T_{cal}} = \left(\frac{\rho_2 t_2}{\rho_1 t_1}\right)_{T} \exp\left(T / T_{cal}\right) \qquad (8)$$

Where not too high accuracy is required and T differs from T_{cal} by only a few degrees, (De

and Dikko, 2012), $(\rho_2/\rho_1)_T$ may be reasonably assumed to be equal to $(\rho_2/\rho_1)_{cal}$ since the density of a liquid does not change significantly with small change of temperature. Then from equation (8) we get

$$\left(\frac{t_2}{t_1}\right)_{T_{cal}} = \left(\frac{t_2}{t_1}\right)_{T} \exp\left(T / T_{cal}\right) \qquad (9)$$

Or

$$\left(\frac{t_2}{t_1}\right)_{T} = \left(\frac{t_2}{t_1}\right)_{T_{cal}} \exp\left(T_{cal} / T\right) \qquad (10)$$

Thus, from equation (9), one can easily determine $(t_2/t_1)_{T_{cal}}$ (that will correspond to the calibration curve by finding out $(t_2/t_1)_T$ experimentally and thus the unknown water concentration, (C_w).

Table 1: Time of Flow, Reduced Time Ratio, Viscosity Coefficient and Reduced Viscosity of Ethanol-water Mixture at 308 K

C_w $(v_w/v_a)100\%$	t_2 (sec) ± 0.01	t_2/t_1 ± 0.0001	μ_{ex} (10^{-4}Pa.sec) ± 0.01	μ_2/μ_1 ± 0.0001
0	2189.65	1.418	9.11	1.261
5	2205.62	1.428	9.26	1.283
10	2246.11	1.454	9.43	1.306
15	2276.35	1.474	9.60	1.330
20	2293.32	1.485	9.78	1.355
25	2322.88	1.504	9.95	1.378
30	2354.77	1.525	10.12	1.402
35	2381.52	1.542	10.28	1.423
40	2412.98	1.562	10.45	1.447
45	2444.23	1.583	10.62	1.471
50	2482.55	1.607	10.81	1.497
Water only	1544.03	1.000	7.22	

$C_w = V_w/V_a(\%)$ = water concentration. t_2 = Average of three readings of time of flow; t_2/t_1 = reduced time ratio; μ_{ex} = experimental viscosity co-efficient. The time taken by distilled water level to descend from mark A to B (Fig 1) is t_1 = 1544.03 and μ_1 = 7.22 10^{-4}Pa.sec is the viscosity coefficient of distilled water.

Table 2: Time of Flow, Reduced Time Ratio, Viscosity Coefficient and Reduced Viscosity of Methanol-water Mixture at 308 K

$C_{w} = (v_{w}/v_{a})100\%$	t_2 (sec) ±0.01	t_2/t_1 ±0.0001	μ_{ex} (10^4Pa.sec) ±0.01	μ_2/μ_1 ±0.0001
0	1268.04	0.821	4.92	0.681
5	1281.12	0.830	5.03	0.697
10	1293.11	0.837	5.12	0.709
15	1304.13	0.844	5.20	0.720
20	1315.01	0.851	5.29	0.732
25	1325.01	0.858	5.37	0.744
30	1336.20	0.865	5.46	0.756
35	1346.10	0.872	5.55	0.767
40	1357.12	0.879	5.64	0.781
45	1369.02	0.887	5.74	0.795
50	1380.00	0.894	5.83	0.807
Water only	1544.03	1.000	7.22	

$C_w=V_w/V_a(\%)$ = water concentration. t_2 = Average of three readings of time of flow; t_2/t_1 = reduced time ratio; μ_{ex} = experimental viscosity co-efficient. The time taken by distilled water level to descend from mark A to B (Fig 1) is t_1 = 1544.03 and μ_1 = 7.22 10^{-4}Pa.sec is the viscosity coefficient of distilled water.

From Tables (1 and 2), it was observed that, the change in time of flow with water concentration has been found to be quite appreciable for both ethanol and methanol. This observation justifies the assumptions made in the theory section that the viscosity coefficient (μ) of a liquid should sensitively depend on the type and concentration of the solute. It was found that for the methanol, the flow time increases from 1268.04 sec. to 1380.00 sec. as C_w increases from 0 to 50%. For ethanol, the corresponding increase in time of flow is from 2189.65 sec to 2482.55 sec.

Fig 1: Variation of reduced time ratio with water concentration in alcohols (Tables 1 and 2)
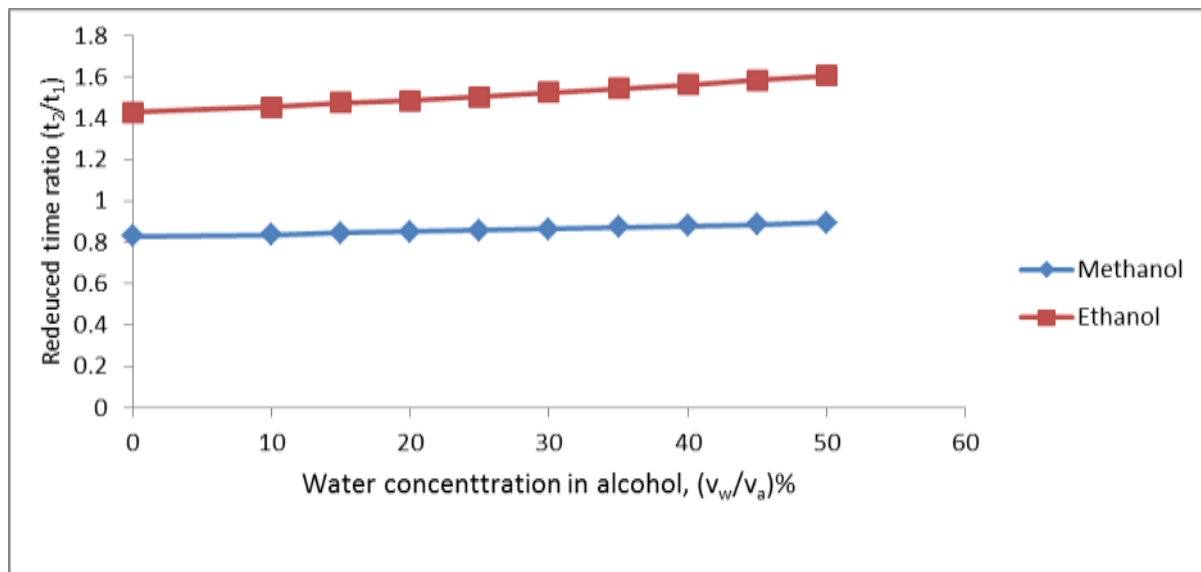
The total uncertainty in C_w thus determined above consists of the following parts: (i) due to uncertainty in t_2/t_1 ; (ii) due to uncertainty in the C_w used in the calibration curve (Fig 1); (iii) due to variation in temperature from that of the calibration curve. Let us address these three sources of errors. As mentioned above, for manual observation, the uncertainty in (t_2/t_1) is around 2×10^{-5}.

Fig (1) shows that a change of C_w from 0% to 50 % causes a change of t_2/t_1 from 0.821 to 0.894 in methanol while t_2/t_1 changes from 1.418 to 1.605 in ethanol. Therefore, one can easily see that the average uncertainly in C_w due to factor (i) is 0.053% in ethanol and 0.14% in methanol; the uncertainty in the measured volume is $\pm$ 0.5 cc. This leads to

uncertainty in C_w ~ 0.16% Thus, the total uncertainty in the unknown water concentration, C_w that can result by using Tables (1 and 2) or Fig (1) is roughly around 0.30% for methanol and 0.21% for ethanol respectively. On the other hand if one employs the variation of density, from Tables. (1 and 2) one can easily see the minimum errors in determination of C_w are 5.6% for methanol-water mixture and 2.3% for ethanol-water mixture. Thus the measurement of time of flow through a capillary tube will yield much higher accuracy. The major source of error in the present technique are the errors in the measurement of volumes of the components during calibration and noting the time for the liquid to descend through the marks A and B (Fig 1). The viscosities of both methanol and ethanol mixtures show an

increasing linear trend with increase in water concentration in alcohol as can be seen from Fig (2). It is also noticed from Fig (2) that the viscosity of the mixture of ethanol and water is higher than the viscosities of methanol and water mixtures. For water-alcohol mixtures, the hydrogen bonding between water and alcohol molecules is much more predominant than for water-water, and alcohol-alcohol molecule pairs, (Nagasawa et al, 2003 and Atamas et al, 2009). Various molecules may mix and dissolve in each other if they have approximately the same type of polarity. In the case of water and ethanol, this is the situation.

Fig 2: Variation of viscosity coefficient with water concentration in alcohol (Tables 1 and 2)
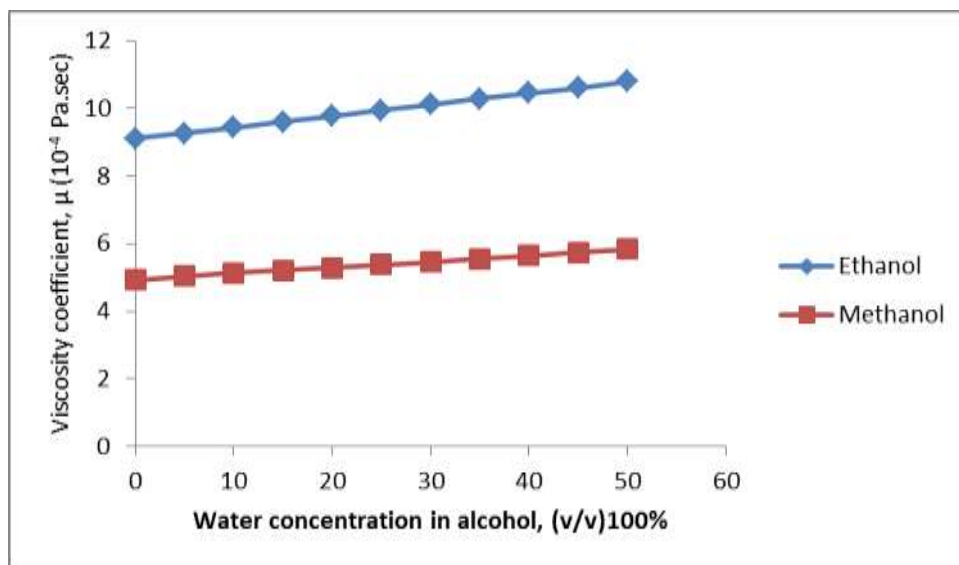
The hydrogen of the -OH group on alcohol is polar as it is in the water molecule. Also, in solvents such as alcohol, which can take part in hydrogen bond formation, the self-association of alcohols may be increased in favor of hydrogen - bonded forms between solute and solvent.

The reduced viscosity ratio, Fig (3), shows a linear relationship with water concentration in alcohol. Ethanol-water mixture has higher reduced viscosity ratio than that of methanol-water mixture. This feature can be utilized to differentiate between ethanol-water mixture and methanol-water mixture.

Fig 3: Variation of reduced viscosity ratio with water conc. in alcohol (Tables 1 and 2)
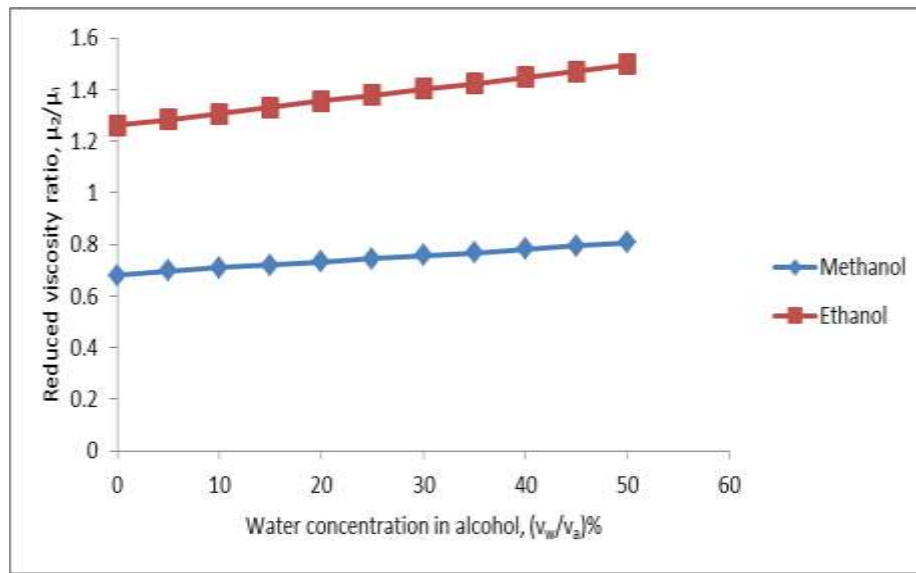
Percentage Water Concentration in Alcohol

The following techniques can be adopted to get the extent of water concentration in methanol or ethanol.

(i) The relationship between reduced time ratio, (t_2/t_1) and percentage water concentration, C_w, from Fig (1) is a linear one at 308K, i.e, $(t_2/t_1)_{T_{cal}} = SC_w + D$. Hence, C_w in alcohol can be determined by the equation,

$$C_w = \frac{(t_2/t_1)_{Tcal} - D}{S} \qquad (11)$$

where S is the slope and D is the intercept on the (t_2/t_1) axis.

Hence, from the plot of (t_2/t_1) versus C_w, Fig. (1), the slope S value is determined to be

$S_m = 0.00142$ and $D_m = 0.821$ for methanol and $S_e = 0.00398$ and $D_e = 1.418$ for ethanol after noting t_1 and t_2 experimentally.

For small temperature variation around the calibration temperature, T_{cal}, 308 ± 2 K, the value of $(t_2/t_1)_{T_{cal}}$ in equation (11) is replaced using equation (9). Thus, the percentage water concentration is given by,

$$C_W = \frac{(t_2/t_1)_T \exp(T/T_{cal}) - D}{S} \qquad (12)$$

Equation (12) has been tested on ethanol collected from five different places and presented in Table (3) below. The results for the percentage water concentration, C_w, calculated show good workability of the equation.

Table 3: Calculated Percentage Water Concentrations, C_w in Ethanol (C_2H_5OH) Collected from Different Sources Using the Developed Equation (12)

Source	Temperature, T (K)	Reduced time ratio, $(t_2/t_1)_T$	C_w $(v_w/v_a)\%$
Dept of Microbiology, MAUTECH Yola	309	0.52326	2.36
Dept of Chemistry, University of Jos	310	0.52248	2.89
Federal Medical center, Yola	309	0.52111	0.78
Namdi medical laboratories, Abuja	309	0.52197	1.31
Adamu Bala Clinic, Jalingo	310	0.52285	3.14

(ii) Similarly, the following equation can be used to get the amount of water in the alcohol, as the relationship between water concentration, C_w and reduced viscosity (μ_2/μ_1) in Fig (3) is linear. Thus,

$$C_w = \frac{(\mu_2/\mu_1)_{Tcal} - D}{S} \quad (13)$$

where S is the slope and D is the intercept on the (μ_2/μ_1) axis, and $S_m = 0.00245$ and $D_m = 0.681$ for methanol and $S_e = 0.00475$ and $D_e = 1.261$ for ethanol, (from Fig (4)).

For small temperature variation around the calibration temperature, $T_{cal} = 308 \pm 2$ K, the value of $(\mu_2/\mu_1)T_{cal}$ in equation (13) is replaced using equation (7). Thus, the percentage water concentration is given as,

$$C_W = \frac{(\mu_2/\mu_1)_T \exp(T/T_{cal}) - D}{S} \quad (14)$$

(iii) Generally, for a one solute or impurity component in a solution or mixture, the percentage volume concentration is given by

$$C_W = \frac{(X_2/X_1)_T \exp(T/T_{cal}) - D}{S} \quad (15)$$

where (X_2/X_1) is the reduced time ratio or reduced viscosity ratio, T_{cal} is the calibration temperature, T is the absolute temperature at which the experiment is conducted, and D and S are values of the intercept and slope respectively from the calibration graph of that liquid mixture.

CONCLUSION

Determining the time of flow of a solution and that of pure water through a sensitive viscometer, in particular, the techniques from this research have provided us with a method of determining the concentration of water in methanol and ethanol. In general, one can determine the concentration of the impurity or contaminant in the mixture or the solution using the techniques developed in this study. The effect of temperature variation on such determination could be taken care of by the new common strategy formulated for easy application of this technique for determination of impurity concentration in a solution, irrespective of the ambient temperature. In general, the technique can be employed to analyze any simple liquids or liquid-mixtures, using some of their physical properties. There is a possibility that with further research the technique can also be extended to determination of concentrations of multi-component solute impurities in a known solvent. The techniques can then find many laboratory and industrial applications and can be adapted to the easy analysis of the components in both liquid and powder milk.

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