

## Analysis of Diffusion Coefficient using Reversed-Flow Gas Chromatography-A Review

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**Abstract: Problem statement:** Since the earliest publication on the technique, Reversed-Flow Gas Chromatography (RF-GC) has been used to determine physicochemical properties by measuring the value of one in the presence of another. The method is precise, accurate and simple compared to other conventional techniques. **Approach:** The experimental setup consists of a small modification of a commercial gas chromatograph, so that it includes a four- or six-port gas sampling valve and a simple cell placed inside the chromatographic oven. **Results:** This cell suppresses the effects of the carrier gas flow on the physicochemical occurrence taking place in the stationary phase. These phenomena pertain to rate coefficients and diffusion coefficients. **Conclusion:** The RF-GC methodologies can be considered as a wise instrumental approach to study the physicochemical phenomenon of liquid pollutants. It can be used to determine the rate coefficients and diffusion coefficients values of samples at various temperatures for the studies of environmental science and physical chemistry research areas. However, the methodologies are not restricted to the current fields of research. It is also relevant in the area of food chemistry, geochemistry, material science, nanotechnology, biological science and chemical technology.

**Key words:** Reversed-Flow Gas Chromatography (RF-GC), diffusion coefficients, rate coefficients, air-liquid, Flame Ionization Detection (FID), Fuller-Shettlar-Giddings (FSG), gas chromatographic

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### INTRODUCTION

The air-liquid interface is a region of intense gradients that impose interesting constraints on the lives and productivity of microorganisms (Ahmed *et al.*, 2010). Research in this area requires working at scales far smaller than those normally associated with the bulk processes on either side of the interface and requires new experimental and theoretical approaches (Colakoglu, 2009). Kouchakzadeh (2011) has reported various methods for estimating volatilization or evaporation rates of organic chemicals from liquid bodies. In later years, Pekar (2002) reported that gas chromatography was used to obtain insight of intermolecular interaction capabilities of liquid polybutadienes, which underlie their phase behavior. Mahdi *et al.* (2010) also reported the importance by applying the Gas Chromatography (GC) technique to study the metabolic fingerprinting of Malaysian plant.

Reversed flow gas chromatography is first proposed for kinetic studies in heterogenous catalysis (Karaiskakis *et al.*, 1982a; Karaiskakis *et al.*, 1982b) applied to the dehydration of alcohols and the

deamination of primary amines (Kotinoupulus *et al.*, 1982). The first idea on this differential aspect was that of Phillips *et al.* (Katsanos, 2002) who stopped the flow of the carrier gas for a short time period repeatedly, producing each time extra narrow peaks. By this method, he measured the differential rate (Zahim *et al.*, 2009) of a catalytic reaction taking place on the stationary phase in a gas chromatographic column. The method permitted a direct determination of reaction rates, not only for small conversions to products, or for reaction times around zero, but in the whole range of conversions covering an extended period of time (Katsanos and Karaiskakis, 1982). The method has been extended to the determination of diffusion coefficients (Li *et al.*, 2010) in binary (Karaiskakis *et al.*, 1983a) and in multicomponent (Katsanos and Karaiskakis, 1983) gas mixtures, the variation of these coefficients with the temperature (Katsanos, 1982), as well as to other measurements like those of adsorption equilibrium constant (Karaiskakis *et al.*, 1982a) and of rate constants for removal of solvents from impregnated porous solids (Katsanos and Georgiadou, 1980). Finally, the method was used to study the heterogenous

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kinetics of a complicated reaction with two gaseous reactants, namely the oxidation of carbon monoxide with oxygen over  $\text{CO}_3\text{O}_4$ -containing catalysts. (Karaiskakis *et al.*, 1983b).

In the recent decades, the application of the technique has captured significance attention from the researcher. The technique of Reversed-Flow Gas Chromatography (RF-GC) has been used for various physicochemical measurements, such as of rate coefficients and diffusion coefficients for the evaporation of pollutant liquids under the influence of surfactants, (Rashid *et al.*, 2004) mass transfer coefficients of gases into the liquids, (Rashid *et al.*, 2001) activity coefficients and mass transfer coefficients in multicomponent gaseous mixtures (Gavril *et al.*, 2004) as well as for the estimation of the solubility and interaction parameters in binary liquid mixtures (Gavril *et al.*, 2006).

The application of the method implies continuously switches the system under study from a flow dynamic one to a static system and vice versa, by repeatedly closing and opening the carrier-gas flows. Diffusion and other related phenomena, which are usually negligible during the gas flow, may become important when the flow is stopped. As mentioned above, the technique of RF-GC is applicable and relevant for diverse research areas such as environment, pharmaceutical, medicine, food, physical and biological sciences.

## MATERIALS AND METHODS

The experimental arrangement consists of a small modification of a commercial gas chromatograph, so that it includes a six-port gas-sampling valve and a simple cell placed inside the chromatographic oven. This cell suppresses the effects of the carrier gas flow on the physicochemical phenomena-taking place in the stationary phase.

The apparatus used is a conventional Gas Chromatograph (Shimadzu GC-14B) with a flame ionization detector contained in its oven (Fig. 1) with two columns of lengths  $l'$  and  $l$  of a stainless-steel chromatographic column containing no chromatographic material. The empty stainless steel is  $\frac{1}{4}$  inch chromatographic tubes with 4mm internal diameter and length,  $L = 28.5$  cm and  $l = l' = 57$ cm. they are connected at the junction  $x = l'$  by a  $\frac{1}{4}$  inch Swagelok tee union. Another sample  $\frac{1}{4}$  inch union is used to connect a short tube (2cm) containing 4ml of liquid at the end of diffusion column  $L$ .

A stainless-steel diffusion column, consisted of the section  $z$ , was connected perpendicularly at its upper end to the middle of the column  $l'+l$  (57cm+57 cm).

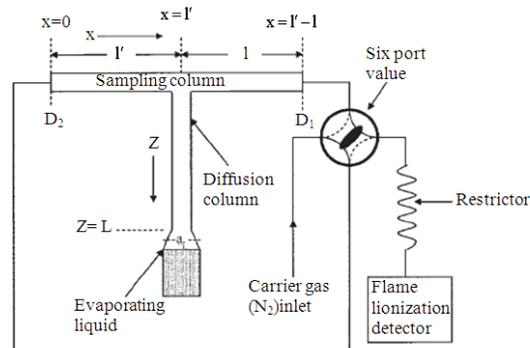


Fig. 1: Instrumentation of the reversed flow gas chromatography technique for the simultaneous measurement of the diffusion coefficients and rate transfer coefficients of the evaporating liquids

The reactant (AR Grade) was injected at a middle point of the column as the stationary phase and the direction of carrier gas flow was reversed from time to time instead of stopping it. This also created extra chromatographic peaks 'sited' on the continuous signal. At the end of column  $z$  is located a container, in which the liquid was contained. The end  $D_1$  of the sampling column  $l'+l$  was connected, via a four-port valve, to the carrier gas (nitrogen) supply, while the other end  $D_2$  was connected to the Flame Ionization Detection (FID) system. After waiting for the monotonously rising concentration-time curve to appear in the detector signal, we started the chromatographic sampling procedure by reversing the direction of the carrier gas flow for 6 s, which is a shorter time period than the gas hold-up time in both column  $l$  and  $l'$ . When the gas flow was restored to its original direction, sample peaks were recorded. The pressure drop along  $l+l'$  was negligible and the pressure inside the whole cell was 1 atm. The carrier gas flow-rate was kept constant ( $1.0 \text{ cm}^3 \text{ sec}^{-1}$ ).

## RESULTS

The following derivation was made by referenced to Fig. 2, which depicts only the sampling column of the cell. The assumptions under which the sampling equation was derived were the adsorption isotherms in the sampling columns were linear and axial diffusion of the gases along coordinated  $x$  or  $x'$  and other phenomena leading to nonideality (such as mass-transfer resistance in the stationary phase) were negligible and the rate process which was taking place in sufficiently short column of the total column length. (Nicholas, 1988) Thus, the common chromatographic sampling equation, describing the elution curves which follow the carrier gas flow reversals was:

$$\begin{aligned}
 c &= c_1(l', t_0 + t' + \tau)u(\tau) + c_2(l', t_0 + t' - \tau) \\
 &[1 - u(\tau - t')]x[u(\tau) - u(\tau - tM')] \\
 &+ c_3(l', t_0 - t' + \tau)u(\tau_0 + \tau - t')\{u(t - t') \\
 &[1 - u(\tau - tM')] - u(\tau - t')[u(\tau - tM')]\}
 \end{aligned}
 \tag{1}$$

where,  $c$  was the concentration of vapor at the detector,  $c_1(l', \dots)$ ,  $c_2(l', \dots)$  and  $c_3(l', \dots)$  were concentrations at the point  $x=l'$  (Fig. 2) for the times shown ( $t_0$ =total time from placing the liquid in column  $L$  to the last backward reversal of gas flow,  $t'$ = time interval of backward flow,  $\tau = t-t_M$ ,  $t$  being the time from the last restoration of the carrier gas flow and  $t_M$  the gas hold-up time of column column  $l$ ); finally the various  $u$ 's were the unit step functions for the arguments shown in parentheses and  $t_M'$  is the gas hold-up time in the column  $l'$ .

For  $t'$  smaller than both  $t_M$  and  $t_M'$ , each sample peak produced by two successive reversals was symmetrical and its maximum height  $h$  from the ending baseline was given by:

$$h \cong 2c(l', t_0) \tag{2}$$

where,  $c(l', t_0)$  was the vapor concentration at  $x=l'$  and time  $t_0$ . The concentration of the liquid can be found from the diffusion equation in the column  $L$  (Fig. 2):

$$\frac{\partial c_z}{\partial t_0} = \frac{D \partial^2 c}{\partial z^2} \tag{3}$$

where,  $D$  was the diffusion coefficient of the vapor into the carrier gas. The solution of Eq. 3 was sought under the initial condition:

$$c_z(z, 0) = 0 \tag{4}$$

the boundary conditions at  $z = L$ :

$$c_z(L, t_0) = c(l, t_0) \tag{5}$$

$$-D(\partial c_z / \partial z)_{z=L} = vc(l', t_0) \tag{6}$$

where,  $v$  was the linear velocity of carrier gas and the boundary condition at  $z = 0$ :

$$-D(\partial c_z / \partial z)_{z=0} = kc(c_0 - c_z(0)) \tag{7}$$

where,  $c_z(0)$  was the actual concentration at the liquid interface at time  $t_0$ ,  $c_0$  the concentration of the vapor which would be in equilibrium with the bulk liquid phase and  $k_c$  was a rate coefficient for the evaporation process. Equation 7 expresses the equality of the

diffusion flux for removal of vapors from the liquid surface and the evaporation flux due to departure of  $c_z$  at the surface from the equilibrium value  $c_0$ .

When the Laplace transform of Eq. 3 is taken with respect to  $t_0$ , a linear second-order differential equation results. It can be solved by using  $z$  Laplace transformation yielding:

$$C_z = C_z(0) \cosh qz + C_z'(0) / q \sinh qh \tag{8}$$

Where:

$$Q = (p_0 / DO)^{1/2} \tag{9}$$

and  $C_z(0)$  and  $C_z'(0)$  were the  $t_0$  Laplace transform of  $c_z(0)$  and  $(\partial c_z / \partial z)_{z=0}$  respectively. If one combines eq 8 with the  $t_0$  transforms of the boundary conditions (5), (6) and (7), the Laplace transform of  $c(l', t_0)$ , denoted as  $C(l', p_0)$ , is found:

$$\begin{aligned}
 C(l', p_0) &= (k_c c_0 / p_0) (1 / (Dq + vkc / Dq) \\
 &\sinh qL + (v + kc) \cosh qL)
 \end{aligned}
 \tag{10}$$

Inverse Laplace transformation of this equation to find  $c(l', t_0)$  was difficult. It can be achieved by using certain approximations which are different for small or for long times. In the first case  $qL$  was large, allowing both  $\sinh qL$  and  $\cosh qL$  to be approximated by  $\exp(qL)/2$ . Then Eq. 10 became:

$$\begin{aligned}
 C(l', p_0) &= (k_c c_0 / p_0 Dq) (2 \exp \\
 &(-qL) / (1 + kc / Dq) (1 + v / Dq))
 \end{aligned}
 \tag{11}$$

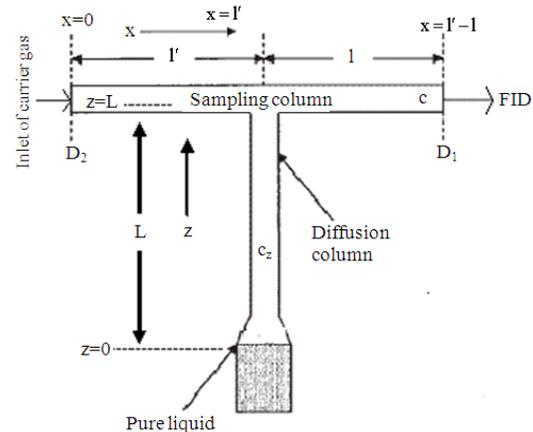


Fig. 2: Schematic arrangement showing the diffusion column  $L$  connected to the chromatographic column  $l'+l$  through which carrier gas flows from  $D_2$  to  $D_1$  or vice versa

which, for high enough flow rates, further reduced to:

$$C(l', p_0) = (2k_c c_0 / vD)(\exp(-qL) / q(q + k_c / D)) \quad (12)$$

Taking now the inverse Laplace transform of this equation, one found:

$$C(l', t_0) = \frac{2k_c c_0}{v} \exp[(k_c L / D) + (k_c 2t_0 / D)] \operatorname{erfc} \left[ \left( \frac{L}{2(Dt_0)^{1/2}} \right) + (t_0)^{1/2} \right] \quad (13)$$

Finally, if one uses the relation  $\operatorname{erfc} x \cong \exp(-x^2) / (\pi^{1/2})$ , which was a good approximation for large values of  $x$ , Eq. 13 became:

$$C(l', t_0) = \frac{2k_c c_0}{v} (D / \pi)^{1/2} \exp [(-L^2 / 4Dt_0)((L / 2t_0)^{1/2} + K_c t_0^{1/2})^{-1}] \quad (14)$$

Coming now to the other extreme, i.e., long time approximations,  $qL$  was small and the functions  $\sinh qL$  and  $\cosh qL$  of Eq. 10 can be expanded in Mc Laurin series (Ford, 1960), retaining only the first three terms in each of them. Then, from Eq. 10 one obtained:

$$C(l', p_0) = (k_c c_0 / p_0) (1 / ((Dq + vk_c / Dq) qL + ((v + k_c)(1 + q^2 L^2 / 2))) \quad (15)$$

and by using Eq. 9 and rearranging this became:

$$C(l', p_0) = \frac{k_c c_0}{L p_0} \frac{1}{p_0 [1 + (v + k_c)L / 2D] + vk_c / D + (v + k_c) / L} \quad (16)$$

For high enough flow rates  $k_c$  can be neglected compared to  $v$  and 1 can be neglected in comparison with  $vL/2D$ . For instance, in a usual experimental situation it was calculated that  $vL/2D = 420$ . Adopting these approximations, Eq. 16 reduces, after some rearrangement, to:

$$C(l', p_0) = \frac{2k_c D c_0}{vL^2 p_0} \frac{1}{p_0 + 2(k_c L + D) / L^2} \quad (17)$$

Finally, inverse Laplace transformation of this relation yielded:

$$C(l', t_0) = \left( \frac{k_c D c_0}{v(k_c L + D)} \right) \{1 - \exp[2(k_c L + D)t_0 / L^2]\} \quad (18)$$

by considering maximum height  $h$  of the sample peaks in Eq. 2 and substituting in it the right hand side of Eq. 18 for  $c(l', t_0)$ , one obtains  $h$  as an explicit function of time  $t_0$ . In order to linearize the resulting relation, an infinity value  $h_\infty$  for the peak height was required:

$$h_\infty = 2k_c D c_0 / [v(k_c L + D)] \quad (19)$$

Using this expression, we obtained:

$$\ln(h_\infty - h) = \ln h_\infty - [2(k_c L + D) / L^2] t_0 \quad (20)$$

Thus, the long enough times, for which Eq. 18 was derived, a plot of  $\ln(h_\infty - h)$  vs.  $t_0$  was expected to be linear and from the slope  $-2(k_c L + D) / L^2$  a first approximate value of  $k_c$  can be calculated from the known value of  $L$  and a literature or theoretically calculated value of  $D$ . This value of  $k_c$  can now be used to plot small time data according to Eq. 14 which was substituted now for  $c(l', t_0)$  in Eq. 2. After rearrangement logarithms were taken and there resulted:

$$\ln \left[ h(L / 2t_0^{1/2} + k_c t_0^{1/2}) \right] = \ln \left[ 4k_c c_0 / v(D / \pi)^{1/2} \right] - (L^2 / 4D)(1 / t_0) \quad (21)$$

Now a plot of the left hand side of this relation vs  $1/t_0$  will yield a first approximation of experimental value for  $D$  from the slope  $-L^2/4D$  of this new linear plot. This  $D$  value can be reused in the slope found from the plot of Eq 20 to calculate a more accurate value for  $k_c$ . In turn, the latter is utilized to report Eq 21, so that a more accurate value for  $D$  was found. These iterations can be continued until no significant changes in the  $k_c$  and  $D$  values results.

The experimental rate coefficients and diffusion coefficient values were obtained by performing a laboratory research scale work. The equation of sampling columns as describe in Eq. 1 was implied to determine the experimental coefficient values where the Eq. 20 and 21. Estimated diffusion coefficient values from the equations are compared with the diffusion coefficient values obtained from Fuller-Shettlar-Giddings (FSG) equation (Fuller *et al.*, 1966).

Example of sample peaks obtained experimentally with gas diffusion rate as a rate process taking place within the sampling cell is shown in Fig. 3.

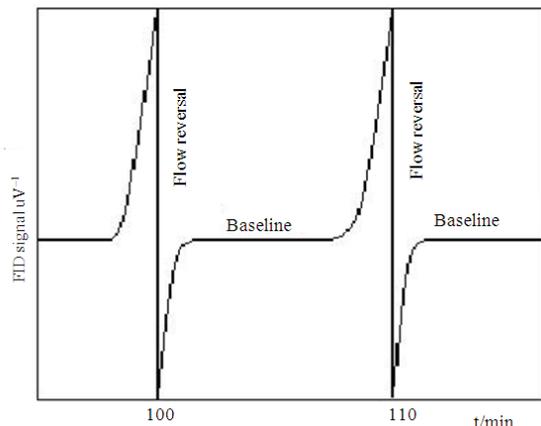


Fig. 3: Reversed flow gas chromatogram showing two sample peaks for the adsorption of liquid pollutant at 312.15K and 1atm ( $v = 1.0\text{cm}^3\text{sec}^{-1}$ )

The result was a sharp symmetrical and narrow peak, its width at half height being exactly equal to the duration of the flow reversal. The example in Fig. 3 showed the peaks were corresponding to various times from the beginning. The pressure drop along  $l+l'$  was negligible and the pressure inside the whole cell was 1 atm. The carrier gas flow-rate was kept constant ( $1.0\text{cm}^3\text{SEC}^{-1}$ ).

### DISCUSSION

Figure 4 shows the rise of the sample peak height with time for the diffusion of liquid vapor. In the analysis, the height,  $h$  of the sample peaks was used as a function of time  $t_0$ , when the flow reversal was made, which is plotted on the semilogarithmic scale. It showed the steep rise and then leveling off with time when peaks height ( $h$ ) were plotted against time ( $t_0$ ).

The rise of the peaks has stop at respective time and become stagnant for the whole period of experiment. The phenomenon happened because the diffusion column was fully diffused by evaporated molecules. Each of the experiment was performed for more than 300 minutes. From the plot of Fig. 4, the highest peak value was taken for plotting  $\ln(h_\infty-h)$  Vs  $t_0$  (Fig. 5). The height deviation which was taken as  $\ln(h_\infty-h)$  of the sample peaks was used as a function of time  $t_0$ ,

The result from Fig. 5 was iterated, leaving 3-4 points, which corresponded to small times and the rest of the experimental points were plotted according to Eq. 20, as shown in Fig. 6. The iteration process was performed to reduce the variance gap between the points. From the plot, a linear graph with negative slope will be obtained. The process was performed to all liquid pollutants samples and experimental temperatures.

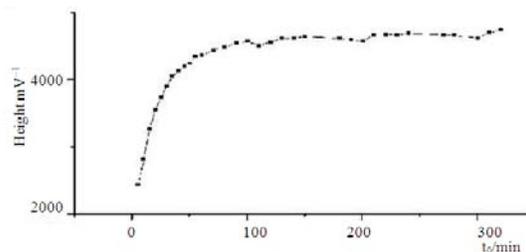


Fig. 4: The rise of the sample peak height with time for the diffusion of liquid vapor into nitrogen ( $v = \text{cm}^3\text{sec}^{-1}$ ) at 1 atm

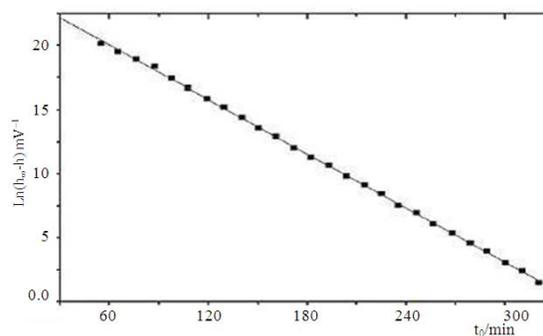


Fig. 5: Example of plotting Eq. 20 for the diffusion of liquid vapor (ethanol) into carrier gas at 313.15K and 1atm ( $v = 1.0\text{cm}^3\text{sec}^{-1}$ )

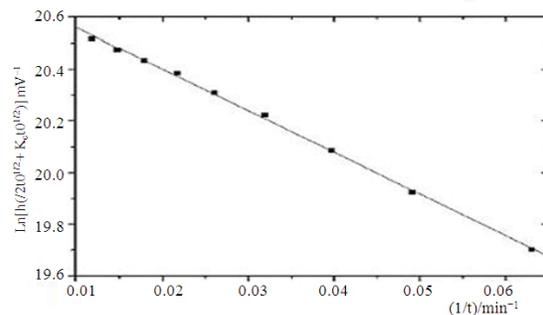


Fig. 6: Data from evaporation of liquid vapor (ethanol) into carrier gas at 313.15K and 1atm ( $v = 1.0\text{cm}^3\text{sec}^{-1}$ ), plotted according to Eq 21

The slope also showed the preliminary indicator for the correctness of the research, based on the plotted graphs. For example, negative slope was considered the analysis is in the rights track in comparison to the positive slope. From the slope of this plot (Fig. 5), which is equal to  $-2(k_cL+D)/L^2$ , according to Eq. 20, using the theoretically calculated value for diffusion coefficient,  $D^{\text{theory}}$  from FSG equation and the actual

value of  $L$ , the theoretical value of rate coefficient,  $k_c$  for the evaporation process was calculated. This approximate value was now used to be plotted according to Eq. 21 as shown in Fig. 6.

From the slope of this plot (Fig. 6), which equals to  $-L^2/4D$ , as shown previously, a value for experimental diffusion coefficient,  $D^{\text{exp}}$  of vapors into carrier gas,  $N_2$  was found. The value was combined with the slope of the previous plot (Fig. 5) and the experimental value of  $k_c$  was calculated. The values were further used to replot the data according to Eq. 21. When the value for experimental diffusion coefficient,  $D^{\text{exp}}$  found coincides with theoretical diffusion coefficient,  $D^{\text{theory}}$ , the iteration procedure stopped.

By using this chromatographic sampling equation, one can simply determine the experimental diffusion coefficients and rate coefficients of the interest analytes based on the perturbation of reversed-flow gas chromatographic methodologies. The application of the sampling equation also contributes to analytical approach for determining physicochemical properties of the sample (Karaiskakis and Gavril, 2004). The precision of the method, defined as the relative standard deviation (%), can be judged from the data of the theoretical values. The experimental values of diffusion coefficient compared with those calculated theoretically by the Equation of FSG. The precision was a measure of the deviation of the values found by the RF-GC method from the calculated ones, defined as:

$$\text{Precision(\%)} = \frac{|D_{AB}^{\text{exp}} - D_{AB}^{\text{theory}}|}{D_{AB}^{\text{exp}}} \times 100$$

The precision values between the experimental and theoretical values were less than 3% (Karaiskakis *et al.*, 1986). This verifies the low deviation values between theoretical and experimental diffusion coefficients which obtained from RF-GC methodologies for analyzing interest analytes. In comparison of literature and experimental values of diffusion coefficients to find the accuracies of the data, the assessment are difficult because limited literatures were reported for the temperatures ranged from 40-100°C (Fuller *et al.*, 1966; Karaiskakis and Gavril, 2004; Karaiskakis *et al.*, 1986; Hoffman, 1932). In addition, correlative studies with previous literature are impossible for the reason that earlier papers have used different temperatures, liquid pollutants samples and carrier gas flow rate. However, the precision of the technique is undeniably amazing (Karaiskakis *et al.*, 1982b; Fuller *et al.*, 1966; Karaiskakis and Gavril, 2004; Karaiskakis *et al.*, 1986; Hoffman, 1932).

## CONCLUSION

The presented methodology of reversed-flow gas chromatography can be used to concurrently determine accurate absolute evaporation rates and vapors diffusivities of pure liquids. The use of RF-GC can be clearly extended in the topics that are related to processes of significant environmental interest, such as: (i) Studies from dilute aqueous solutions of organic and inorganic pollutant compounds; (ii) Investigation of water evaporation (sea, lake, river water bodies); (iii) Effects of ionic and zwitterionic surfactants; (iv) Experiments with a longer duration (more than 6 hours) in order to examine the change of the evaporation rate in a broader time scale (determination of evaporation rates at a time resolved procedure) and moreover, the life span of the studied liquid pollutants; and (v) Experiments with surfactants under steering conditions in order to investigate their durability. The application of RF-GC methodologies contributed to a long list of significant environmental interest (as mentioned above) which is not exhaustive.

More work is well under way concerning the application of the RF-GC version for the future research in this field. From the findings, one observed a free surface influenced of surfactant where the molecules of the analytes evaporated in absence of layer thickness. However, the idea of the influence of surfactant on the surface of liquid body has the potential to contribute to environmental pollution controls. The multilayer thickness of surfactant has the capabilities of reducing the evaporation rate of liquid pollutants resulting in low concentration of evaporated pollutants in atmosphere which has not been reported yet or cited in any research papers. The method can also be used to study the evaporation rate of liquid pollutants under the influenced of other liquids. The rate of evaporation of liquid molecules can be compared to the studies on the competency of diffusion coefficients and rate coefficients of the liquid pollutants presence in a liquid body. The findings may reveal the evaporation rate of liquid pollutants and the effects of activation energy of respective liquid pollutants in the presence of other liquids. As much as the ideas are concerned, the current findings will contribute to the references for later studies.

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