

World Journal of Engineering and Physical Sciences Vol. 3 (4), pp.051-057, December 2015 Available online at http://wsrjournals.org/journal/wjeps ISSN 2331-1878 ©2015 World Science Research Journals

Full Length Research

Analysis of solvent effects on the reactivity of carboxylic acids with diazodiphenylmethane using Catalan's and Krygowski-Fawcett's approaches

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Accepted June 20, 2015

Solvent effects on the reactivity of eight carboxylic acids with diazodiphenylmethane in some pure organic solvents were examined in the light of Catalan's three-parameter and Krygowski-Fawcett's twoparameter equations using multiple linear regression analysis. Findings reveal that the results from the two different approaches are in good agreement in explaining the specific solute-solvent interaction. It was also shown that the nonspecific dipolarity/polarizability term in the Catalan equation contributes small but significant impact on the observed solvent effects. The fitness of models based on Krygowski-Fawcett's equation is adjudged to be relatively poor due to the neglect of nonspecific solute-solvent interaction in the models. Better insight into the mechanism of how solvents affect the reactivity of carboxylic acids was provided by Catalan's approach.

Keywords: Carboxylic acids, Diazodiphenylmethane, Multiple Regression, Reactivity, Solvent Effects.

INTRODUCTION

The observation that solvents have influence on chemical reactivity was first noticed over a century ago by Berthelot, Pean de Saint-Gilles, Menshutkin, Claisen, Knorr, and Wilslicenus (Reichardt, 1994). Nowadays, it is generally known to all chemists that the rates and equilibrium positions of chemical reactions, as well as the position and intensity of absorption bands in ultraviolet/visible, infrared, nuclear magnetic resonance, and electron spin resonance spectroscopy, are solventdependent (Whetsel and Kagarise, 1962; Amis and Hinton, 1973; Huisgen, 1980; Reichardt, 1982; Diakovitch et al, 1984; Blandamer and Burgess, 1990; Burgess, 1991; Engberts et al, 1998; Reichardt, 2003; Homocianu et al, 2011). As pointed out by Abraham et al (1988), there are two main reasons for studying solvent effects on reaction rates and other processes. The first objective is the prediction of rate constants in other solvents while the second objective is to reach some understanding of

the various influences that might affect reaction rate.

In order to take two or more aspects of solvation into account, several multiparameter equations have been proposed by different scholars (Taft et al, 1985; Katrizky et al, 2003). According to Reichardt (2003), these equations generally take the form of equation 1(a) and are based on the assumption that solvent-dependent processes can be represented as a linear combination of two or more independent but complementary solvent empirical parameters or/and bulk solvent physical properties. In equation 1(a), A is the value of a solventdependent physicochemical property (log K, log k, hv, etc.) in a given solvent and A_0 is the statistical quantity corresponding to the value of this solvent-dependent property in the gas phase or in an inert solvent; B, C and D represent independent but complementary solvent parameters, which account for the different solute-solvent interaction mechanisms; b, c and d are the regression

coefficients describing the sensitivity of the solventdependent properties.

Catalan's three-parameter approach and Krygowski-Fawcett's two-parameter approach are among the numerous multiparameter approaches that have so far been proposed. In the Catalan's equation shown in equation 1(b), k and k_0 represent the reaction rate constants in a given solvent and in a gas phase (or inert solvent) respectively; SPP corresponds to the solvent dipolarity/polarizability; SA corresponds to the solvent hydrogen bond donor (HBD) acidity; SB corresponds to the solvent hydrogen bond acceptor (HBA) basicity; a, b and c are the regression coefficients describing the sensitivity of log k to SPP, SA and SB respectively (Reichardt, 2003). Similarly, in the Krygowski-Fawcett's equation shown in equation 1(c), k and k_0 represent the reaction rate constants in a given solvent and gas phase (or inert solvent) respectively; $E_T(30)$ and DN are the Dimroth-Reichardt solvent polarity parameter and the Gutmann's donor number, representing a measure of Lewis acidity of the solvent and a measure solvent basicity respectively; α and β are the regression coefficients describing the sensitivity of $\log k$ to electrophilic and nucleophilic solvent properties respectively (Reichardt, 2003).

Reactions with of carboxylic acids diazodiphenylmethane have been studied extensively by many investigators and the mechanisms of these reactions are fairly understood (Chapman et al, 1968; Jovanovic et al. 2000: Nikolic et al. 2000: Uscumlic et al. 2005; Nikolic and Uscumlic, 2007; Drmanic et al, 2009). It is for this reason that chemists generally choose the reaction, as well as other model reactions, for testing the validity of newly proposed multiparameter equations. The main thrust of the current paper therefore is to carry out multiple regression analysis on the second-order rate constants for the reactions of eight carboxylic acids with diazodiphenylmethane in some pure organic solvents, in the light of Catalan's three-parameter and Krygowski-Fawcett's two-parameter equations, with a view to unravel the influence of solvents on the reactivity of these reactions.

| $A = A_0 + b.B + c.C + d.D +$ | 1(a) |
|---|------|
| $\log k = \log k_0 + a.SPP + b.SA + c.SB$ | 1(b) |

| $\log k = \log k_0 + \alpha E_T(30) + \beta DN$ | 1(c) |
|---|------|

METHODOLOGY

Second-order rate constants for the reactions of benzoic acid, cyclopent-1-ene-carboxylic acid, cyclohex-1-enecarboxylic acid, cyclohept-1-ene-carboxylic acid, phenylacetic acid, cyclopent-1-ene-acetic acid, cyclohex-1-ene-acetic acid and cyclohept-1-ene-acetic acid with

diazodiphenylmethane in fifteen organic solvents were obtained from literature (Uscumlic and Nicolic, 2009). Catalan's solvatochromic parameters (SPP, SA and SB) were taken from the compilation of Catalan (2001) while Dimroth-Reichardt solvent polarity parameter $(E_T(30))$ and Gutmann donor number (DN) were taken from the compilation of Marcus (1998). Multiple linear regression analyses, in line with Catalan's three-parameter approach and Krygowski-Fawcett's two-parameter approach, were carried out with these data using Microsoft Excel. The goodness of fit of the models derived from these analyses was evaluated using coefficient of determination, R^2 (the closer to 1 the better), standard error of the estimate, s (the smaller the better) and Fisher index of reliability, F (the larger the better). Shown in Tables 1 and 2 are the Catalan's parameters and Krygowski-Fawcett's parameters for some selected organic solvents used in this investigation. The secondorder rate constants for the reactions under study were presented in Tables 3 and 4.

RESULTS AND DISCUSSION

The results of multiple regression analyses of log k_2 for the reactions of benzoic acid, cyclopent-1-ene-carboxylic acid, cyclohex-1-ene-carboxylic acid, cyclohept-1-enecarboxylic acid, phenylacetic acid, cyclopent-1-ene-acetic acid, cyclohex-1-ene-acetic acid and cyclohept-1-eneacetic acid with diazodiphenylmethane in fifteen organic solvents, by means of Catalan's three-parameter equation, are presented in equations 2(a) - 2(h). As shown in the equations, the rates of reaction between the eight carboxylic acids and diazodiphenylmethane are satisfactorily described by solvent dipolarity/polarizability, solvent HBD acidity, and solvent HBA basicity as indicated by the values of coefficients of determination $(R^2 \ge 0.909)$. On the basis of the standard errors of the regression coefficients, the sensitivity of the rates of these reactions is adequately precise to both solvent HBD acidity and solvent HBA basicity but somewhat imprecise to the solvent dipolarity/polarizability. Although the fitness of the correlations obtained in the present study is adjudged to be fairly satisfactory on the basis of the multiple correlation coefficients presented in equations 2(a) - 2(h), more excellent correlations with R^2 values as high as 0.992 have been reported in the literature using Kamlet-Taft three-parameter approach (Uscumlic and Nicolic, 2009).

Judging by the signs of the coefficients of the last three terms in equations 2(a) - 2(h), it can be inferred that the solvents influence the rates of reactions between carboxylic acids and diazodiphenylmethane by two opposing effects. The positive signs of the coefficients of the terms SPP and SA indicate that the rates of these reactions increase with increasing solvent dipolarity/polarizability and solvent HBD acidity. Hence,

| Solvent | SPP | SA | SB |
|----------------------|-------|-------|-------|
| Methyl acetate | 0.785 | 0.000 | 0.527 |
| Cyclohexanone | 0.874 | 0.000 | 0.482 |
| Diethyl ketone | 0.883 | 0.000 | 0.557 |
| Carbon tetrachloride | 0.632 | 0.000 | 0.044 |
| Chloroform | 0.786 | 0.047 | 0.071 |
| Ethyl acetate | 0.795 | 0.000 | 0.542 |
| Cyclopentanone | 0.908 | 0.000 | 0.465 |
| Dioxane | 0.701 | 0.000 | 0.444 |
| Acetonitrile | 0.895 | 0.044 | 0.286 |
| Acetone | 0.881 | 0.000 | 0.475 |
| Methanol | 0.857 | 0.605 | 0.545 |
| Ethanol | 0.853 | 0.400 | 0.658 |
| Ethylene glycol | 0.932 | 0.565 | 0.534 |
| Dimethyl sulfoxide | 1.000 | 0.072 | 0.647 |
| Tetrahydrofuran | 0.838 | 0.000 | 0.591 |

Table 1. Catalan's parameters for some selected solvents

Table 2. Krygowski-Fawcett's parameters for some selected solvents

| Solvent | <i>E_T</i> (30) | DN |
|----------------------|---------------------------|------|
| Methyl acetate | 38.9 | 16.3 |
| Diethyl ketone | 39.3 | 15.0 |
| Carbon tetrachloride | 32.4 | 0.0 |
| Chloroform | 39.1 | 4.0 |
| Ethyl acetate | 38.1 | 17.1 |
| Dioxane | 36.0 | 14.8 |
| Acetonitrile | 45.6 | 14.1 |
| Acetone | 42.2 | 17.0 |
| Methanol | 55.4 | 30.0 |
| Ethanol | 51.9 | 32.0 |
| Ethylene glycol | 56.3 | 20.0 |
| Dimethyl sulfoxide | 45.1 | 29.8 |
| Tetrahydrofuran | 37.4 | 20.0 |

solvation of the transition states is dominated by the dipolarity/polarizability and HBD acidity of the solvents. The negative sign of the coefficient of the term SB however, indicate that the rates of these reactions decrease with increasing HBA basicity of the solvents. This implies that the solvent HBA basicity stabilizes the initial states before the commencement of the reactions and is therefore responsible for the decrease in the reaction rates. From the magnitudes of the regression coefficients, the percentage contributions of Catalan's parameters (SPP, SA and SB) were calculated and the results are given in Table 5. Except for the reaction of benzoic acid, substantial contributions of solvent effects to the reactivity of the carboxylic acids were due mainly to

the specific solute-solvent interactions, with the solvent HBA basicity playing the greatest role (more than 50% in all but one case). Table 5 also show that with the exception of the reaction of benzoic acid, only small proportions of the observed solvent effects can be ascribed to the nonspecific solute-solvent interaction as less than 15% of the observed solvent effects are due to the solvent dipolarity/polarizability term in the equations. The results just presented agree with the findings reported by Uscumlic and Nicolic (2009) for the analysis of solvent effects on the reactivity of carboxylic acids with diazodiphenylmethane using Kamlet-Taft approach.

The results of multiple regression analyses of log k_2 for the reactions of benzoic acid, cyclopent-1-ene-carboxylic

| Table 3. Second-order rate constants for the reaction of benzoic acid and cycloalkene-carboxylic acids with diazodiphenylmethane in various |
|---|
| solvents (dm ³ mol ⁻¹ min ⁻¹) |

| Solvent | Benzoic acid | Cyclopent-1-ene- carboxylic acid | Cyclohex-1-ene- carboxylic acid | Cyclohept-1-ene- carboxylic acid |
|----------------------|--------------|-------------------------------------|------------------------------------|-------------------------------------|
| Methyl acetate | 0.260 | 0.044 | 0.032 | 0.031 |
| Cyclohexanone | 0.220 | 0.028 | 0.020 | 0.019 |
| Diethyl ketone | 0.265 | 0.073 | 0.053 | 0.051 |
| Carbon tetrachloride | 0.638 | 0.399 | 0.329 | 0.286 |
| Chloroform | 12.30 | 5.373 | 4.335 | 3.378 |
| Ethyl acetate | 0.180 | 0.038 | 0.025 | 0.016 |
| Cyclopentanone | 0.293 | 0.036 | 0.025 | 0.025 |
| Dioxane | 0.058 | 0.088 | 0.065 | 0.062 |
| Acetonitrile | 3.730 | 0.430 | 0.318 | 0.199 |
| Acetone | 0.350 | 0.059 | 0.048 | 0.039 |
| Methanol | 2.470 | 1.106 | 0.818 | 0.654 |
| Ethanol | 0.995 | 0.534 | 0.417 | 0.332 |
| Ethylene glycol | 4.020 | 2.452 | 1.962 | 1.570 |
| Dimethyl sulfoxide | 0.141 | 0.012 | 0.008 | 0.007 |
| Tetrahydrofuran | 0.105 | 0.027 | 0.019 | 0.016 |

Table 4. Second-order rate constants for the reaction of phenylacetic acid and cycloalkene-acetic acids with azodiphenylmethane in various solvents (dm³ mol⁻¹ min⁻¹)

| Solvent | Phenylacetic acid | Cyclopent-1-ene- acetic acid | Cyclohex-1-ene-acetic acid | Cyclohept-1-ene acetic acid |
|----------------------|-------------------|---------------------------------|-------------------------------|--------------------------------|
| Methyl acetate | 0.132 | 0.181 | 0.144 | 0.098 |
| Cyclohexanone | 0.153 | 0.187 | 0.149 | 0.102 |
| Diethyl ketone | 0.279 | 0.268 | 0.214 | 0.148 |
| Carbon tetrachloride | 6.628 | 2.161 | 1.759 | 1.299 |
| Chloroform | 613.0 | 46.06 | 37.84 | 29.02 |
| Ethyl acetate | 0.210 | 0.036 | 0.028 | 0.017 |
| Cyclopentanone | 0.117 | 0.139 | 0.110 | 0.074 |
| Dioxane | 0.169 | 0.319 | 0.255 | 0.177 |
| Acetonitrile | 8.919 | 1.535 | 1.294 | 0.972 |
| Acetone | 0.233 | 0.246 | 0.194 | 0.146 |
| Methanol | 2.539 | 2.237 | 1.652 | 1.299 |
| Ethanol | 1.139 | 0.828 | 0.659 | 0.614 |
| Ethylene glycol | 5.049 | 4.080 | 3.020 | 2.237 |
| Dimethyl sulfoxide | 0.014 | 0.031 | 0.024 | 0.016 |
| Tetrahydrofuran | 0.057 | 0.071 | 0.056 | 0.039 |

acid, cyclohex-1-ene-carboxylic acid, cyclohept-1-enecarboxylic acid, phenylacetic acid, cyclopent-1-ene-acetic acid, cyclohex-1-ene-acetic acid and cyclohept-1-eneacetic acid with diazodiphenylmethane in thirteen organic solvents, by means of Krygowski-Fawcett's twoparameter equation, are presented in equations 3(a) - 3(h). As shown in the equations, the rates of reaction between the eight carboxylic acids and diazodiphenylmethane poorly correlated with Dimroth-Reichardt solvent polarity parameter ($E_T(30)$) and Gutmann donor number (DN) as indicated by the values of the coefficients of determination ($R^2 \leq 0.896$). Comparing the standard errors of the estimate and the Fisher indices of reliability in equations 3(a) - 3(h) with

| Solvent | %SSP | %SA | %SB |
|---------------------------------|------|-----|-----|
| Benzoic acid | 38 | 24 | 39 |
| Cyclopent-1-ene-carboxylic acid | 3 | 47 | 50 |
| Cyclohex-1-ene-carboxylic acid | 2 | 47 | 51 |
| Cyclohept-1-ene-carboxylic acid | 0 | 48 | 51 |
| Phenylacetic acid | 14 | 28 | 58 |
| Cyclopent-1-ene-acetic acid | 11 | 34 | 55 |
| Cyclohex-1-ene-acetic acid | 11 | 34 | 55 |
| Cyclohept-1-ene-acetic acid | 11 | 34 | 55 |

Table 5. Percentage contributions of Catalan's parameters to the reactivity of the studied reactions

their corresponding values in equations 2(a) - 2(h) also revealed that the quality of the linear equations obtained by means of Catalan's approach is better than the quality of the linear equations obtained by means of Krygowski-Fawcett's approach. The poor correlation observed in the models derived from Krygowski-Fawcett's approach can be ascribed to the supposition that nonspecific solutesolvent interactions are negligible (Reichardt, 2003).

However, in spite of this drawback, some qualitative information about the reactivity of the reactions under investigation can be deduced from the models derived from Krygowski-Fawcett's approach. The positive sign on the coefficients of $E_T(30)$ term in equations 3(a) - 3(h) indicated that the rates of reactions between carboxylic

acids and diazodiphenylmethane increase with increasing electrophilic solvent property. Conversely, the negative sign on the coefficients of DN term in equations 3(a) – 3(h) indicated that the rates of reactions between carboxylic acids and diazodiphenylmethane decrease with increasing nucleophilic solvent property. It can be interpreted from these findings that while solvation of the transition state is dominated by the Lewis acidity of the solvents, the Lewis basicity of the solvents is responsible for the solvation of the initial state. The result just presented for Krygowski-Fawcett's approach is in good agreement with the result presented in the preceding paragraph for Catalan's approach in explaining the specific solute-solvent interaction.

Benzoic acid

| log $k_2 = -1.79 + (3.36 \pm 1.08)$.SPP + (2.10 ± 0.39).SA - (3.40 ± 0.53).SB $R^2 = 0.920$, s = 0.30, n = 15, F = 20.25 | 2(a) |
|---|------|
| Cyclopent-1-ene-carboxylic acid log $k_2 = 0.14 + (0.18 \pm 1.22)$.SPP + (3.03 ± 0.44).SA - (3.22 ± 0.60).SB $R^2 = 0.929$, s = 0.33, n = 15, F = 22.98 | 2(b) |
| Cyclohex-1-ene-carboxylic acid log $k_2 = 0.08 + (0.14 \pm 1.25)$.SPP + (3.10 ± 0.45).SA – (3.33 ± 0.62).SB $R^2 = 0.929$, s = 0.34, n = 15, F = 23.09 | 2(c) |
| Cyclohept-1-ene-carboxylic acid log $k_2 = 0.07 + (0.00 \pm 1.21)$.SPP + (3.03 ± 0.44).SA - (3.21 ± 0.61).SB $R^2 = 0.929$, s = 0.33, n = 15, F = 23.11 | 2(d) |
| Phenylacetic acid log $k_2 = 1.01 + (1.45 \pm 1.97)$.SPP + (2.86 ± 0.71).SA – (5.97 ± 0.98).SB $R^2 = 0.909$, s = 0.54, n = 15, F = 17.45 | 2(e) |
| Cyclopent-1-ene-acetic acid log $k_2 = 0.54 + (0.84 \pm 1.38)$.SPP + (2.59 ± 0.49).SA – (4.15 ± 0.68).SB $R^2 = 0.919$, s = 0.38, n = 15, F = 19.96 | 2(f) |

| Cyclohex-1-ene-acetic acid log $k_2 = 0.45 + (0.87 \pm 1.39)$.SPP + (2.55 ± 0.50).SA – (4.18 ± 0.69).SB $R^2 = 0.917$, s = 0.38, n = 15, F = 19.51 | 2(g) |
|---|------|
| Cyclohept-1-ene-carboxylic acid log $k_2 = 0.34 + (0.85 \pm 1.46)$.SPP + (2.68 ± 0.52).SA – (4.24 ± 0.73).SB $R^2 = 0.914$, s = 0.40, n = 15, F = 18.59 | 2(h) |
| Enzoic acid log $k_2 = -3.82 + (0.12 \pm 0.02)$. $E_T(30) - (0.08 \pm 0.02)$.DN $R^2 = 0.896$, s = 0.34, n = 13, F = 20.33 | 3(a) |
| Cyclopent-1-ene-carboxylic acid log $k_2 = -4.61 + (0.13 \pm 0.02)$. $E_T(30) - (0.09 \pm 0.02)$.DN $R^2 = 0.882$, s = 0.42, n = 13, F = 17.65 | 3(b) |
| Cyclohex-1-ene-carboxylic acid log $k_2 = -4.83 + (0.13 \pm 0.02)$. $E_T(30) - (0.10 \pm 0.02)$.DN $R^2 = 0.884$, s = 0.43, n = 13, F = 17.99 | 3(c) |
| Cyclohept-1-ene-carboxylic acid log $k_2 = -4.78 + (0.13 \pm 0.02)$. $E_T(30) - (0.09 \pm 0.02)$.DN $R^2 = 0.875$, s = 0.44, n = 13, F = 16.40 | 3(d) |
| Phenylacetic acid log $k_2 = -4.07 + (0.16 \pm 0.04)$. $E_T(30) - (0.15 \pm 0.03)$.DN $R^2 = 0.862$, s = 0.67, n = 13, F = 14.57 | 3(e) |
| Cyclopent-1-ene-acetic acid log $k_2 = -3.78 + (0.13 \pm 0.03)$. $E_T(30) - (0.11 \pm 0.02)$.DN $R^2 = 0.864$, s = 0.49, n = 13, F = 14.77 | 3(f) |
| Cyclohex-1-ene-acetic acid log $k_2 = -3.84 + (0.13 \pm 0.03)$. $E_T(30) - (0.11 \pm 0.02)$.DN $R^2 = 0.862$, s = 0.50, n = 13, F = 14.55 | 3(g) |
| Cyclohept-1-ene-acetic acid log $k_2 = -4.13 + (0.13 \pm 0.03)$. $E_T(30) - (0.11 \pm 0.02)$.DN $R^2 = 0.854$, s = 0.53, n = 13, F = 13.45 | 3(h) |

CONCLUSION

Multiple linear regression analyses were carried out on the second-order rate constants for the reactions of eight carboxylic acids with diazodiphenylmethane using Catalan's three-parameter and Krygowski-Fawcett's twoparameter equations. The results from the two different approaches are in good agreement in explaining the specific solute-solvent interaction on the reactivity of these carboxylic acids. The nonspecific dipolarity/ polarizability term in the Catalan equation was found to play a small but significant role in the observed solvent effects. The relatively poor fitness of Krygowski-Fawcett's models was attributed to the neglect of nonspecific solute-solvent interaction in the models. Catalan's threeparameter approach, like Kamlet-Taft's three-parameter approach reported in the literature, is therefore capable of providing insight into the mechanism by which solvents affect the reactivity of carboxylic acids with diazodiphenylmethane.

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