EFFECT OF ALCOHOL COSOLVENTS ON THE AQUEOUS SOLUBILITY OF TRICHLOROETHYLENE

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ABSTRACT

Trichloroethylene (TCE) is a halogenated organic compound, which due to its unique properties and solvent effects, has been widely used as an ingredient in industrial cleaning solutions and as a universal degreasing agent. Due to its wide use, TCE is prevalent in contaminated soil and Superfund sites. This has generated a high degree of interest in efficient and cost-effective technologies that can be used to remediate soil contaminated with TCE. *In situ* and pump-and-treat chemical and biological remediation techniques work more efficiently in aqueous phase systems. Therefore, extraction of TCE from soil is a widespread area of interest.

Three alcohols (methanol, ethanol, and isopropyl alcohol) were used as cosolvents with water in bench-scale, thoroughly mixed batch systems, and TCE solubility was determined as a function of cosolvent concentration with an input TCE concentration of 10,000 ppm. These cosolvents were selected based on their low toxicity, high solubility, low cost, and other favorable factors. Preliminary results show at low cosolvent additions (<5%), there was no difference between various alcohols and there was no significant TCE solubility enhancement over TCE solubility in water (~1000 ppm). At 25% alcohol addition, TCE solubility was enhanced to the same extent (~3000 ppm) by both ethanol and isopropyl alcohol, which was about 1.5 times the amount of TCE dissolved by methanol. At 50% alcohol addition, isopropyl alcohol performed the best (TCE solubility = 10,000 ppm; maximum TCE available), followed by ethanol (TCE solubility = 8,500 ppm) and methanol (TCE solubility = 6,100 ppm). In pure cosolvents (100% cosolvent, 0% water), all three alcohols dissolved all the available TCE of 10,000 ppm. Three models (power law, quadratic, and exponential) were tested to fit the experimental data. The power law model was the least successful, while both quadratic and exponential models performed very well with R^2 values greater than 0.995 for almost all cosolvents.

Key words: TCE, trichloroethylene, cosolvent, solubility enhancement, extraction

INTRODUCTION

Extent of TCE problem in the environment

Chlorinated organic compounds, such as trichloroethylene (TCE), are the most prevalent contaminants found in soil and groundwater and pose serious health risks even at trace concentrations. TCE is a halogenated organic compound, which due to its unique properties and solvent effects has been widely used as an ingredient in industrial cleaning solutions and as a universal degreasing agent. It is used as an extraction solvent for greases, oils, fats, waxes, and tars, and as a refrigerant.

TCE has not been known to cause cancer but can become a health hazard after being in the soil for a certain amount of time. After TCE stays in the soil, it is chemically converted to vinyl chloride by reductive dehalogenation. Vinyl chloride, as compared to TCE, is a potential carcinogen. EPA drinking water standards proclaim that the maximum contaminant level (MCL) for TCE is 0.005 mg/L, and the maximum contaminant level goal (MCLG) is zero.

Major environmental releases of TCE are due to air emissions from metal degreasing plants. Wastewater from metal finishing, paint and ink production, electrical components, and rubber-processing industries also may contain TCE. These are emissions to the air unless there is a spill. TCE is present in dense nonaqueous-

phase liquids (DNAPLs), especially in the form of pools in the subsurface. From 1987 to 1993, according to the Toxics Release Inventory, TCE releases to water totaled over 100,000 lbs. Releases to land totaled over 191,000 lbs. These releases were primarily from steel pipe and tube manufacturing industries.

Since TCE has been widely used, and is very abundant in the soil much research focus has been devoted to remediating TCE-contaminated soil. Many remedial technologies of groundwater and soil contaminated by DNAPLs, such as TCE, have been developed (Russel et al., 1992). Treatment alternatives include bioremediation, thermal treatment, dehalogenation, chemical oxidation, and electrokinetic remediation. Efforts are being made to develop and demonstrate *in situ* treatment methods that are rapid and extensive, as well as less sensitive to contaminant character and concentration. Advanced oxidation processes (AOPs) have been suggested as possible alternatives. AOPs are based on the production of reactive hydroxyl radicals (OH) with much higher oxidizing power than that of the parent

compound. Chemical oxidation processes involving fluid and gaseous oxidants, such as hydrogen peroxide or potassium permanganate, can be coupled with delivery technologies such as soil fracturing or mixing for the treatment of contaminated soils and sediments. Recently, there has been interest in *in situ* chemical oxidation using potassium permanganate as an oxidant because of its high aqueous solubility, its long half-life in the subsurface, its property of injectability, and its cost-effectiveness. To determine the behavior, transport, and fate of the contaminants in the environment, and to design remediation systems, one must take into consideration the physical and chemical properties of the contaminant. Relevant properties of TCE are presented in Table 1.

TCE solubility

In general, DNAPLs have a low aqueous solubility. A major barrier to the successful implementation of soil treatment technologies is the hydrophobic nature of hazardous compounds, which limits the concentration level in the aqueous phase. Low aqueous-phase contaminant concentration levels lead to low conversion rates, resulting in longer treatment times and therefore, higher cost. In the case of TCE, aqueous solubility of 1000 mg/L and log octanol-water partition coefficient of 2.42 are not as hydrophobic as corresponding values for PAHs and PCBs, but can still be rate controlling in soil treatment systems. Typically, large K_{OW} values are an indication that the substance has a lower affinity for the aqueous phase and not due to a higher solubility in the organic phase. Most organic substances behave ideally in octanol;

Table 1. Physical/Chemical Properties of Trichloroethylene.

Property	Value
Density	1.46 g/ml
Water Solubility	1000 mg/L @ 25 °C
Henry's Law Constant	0.00892 (atm-m³/mol @ 20 °C)
Molecular Weight	131.4 g/gmol
Boiling Point	86.7 °C
Log Octanol-Water Partition Coefficient, K_{OW}	2.42

thus their activity coefficients vary between 1 to 10 in octanol, while the corresponding value in water varies from 0.1 to 10^7, leading to a wide range (several orders of magnitude) of K_{OW} values (Valsaraj, 1995). Hence the K_{OW} value is a strong measure of hydrophobicity of the substance. Ways to increase the aqueous solubility of contaminants would provide improved remediation strategies.

Traditional technologies such as pump-and-treat have been shown to be ineffective in the remediation of DNAPL-contaminated soils, primarily due to their low solubility in water. Surfactants and cosolvents increase the solubility of organic compounds and decrease the interfacial tension, which allows for more effective *in situ* or pump-and-treat technologies. To improve the soil treatment performance, new additives and technologies are being developed to increase contaminant mobilization by increasing the aqueous solubility of the contaminant (Imhoff et al.*, 1995). Cosolvents, in particular alcohols, have been used to increase the solubility and decrease interfacial tension of the contaminants in the soil. Falta (1998) has reviewed and compiled a list of 19 recent experiments performed by researchers to test the effectiveness of tert-butyl alcohol, isopropyl alcohol, and methanol on TCE. These studies showed a very high (>80%) dissolution of TCE when these alcohols were added to the TCE-water systems. Gu et al. (1997) have shown that surfactants and cosolvents can be used to enhance the removal of TCE and polychlorinated biphenyls (PCBs) from contaminated soils. They evaluated the use of zero-valent iron and palledized iron fillings (Fe-Pd) to dechlorinate TCE and a PCB congener (2,3,2', 5-tetrachlorobiphenyl) in a dihexyl sulfosuccinate surfactant solution. Results from their batch experiments indicated that TCE can be rapidly degraded by palledized iron fillings with a half-life of about 27.4 min, suggesting that Fe-Pd fillings in the surfactant solutions may have a potential for *ex situ* treatment of TCE-contaminated soils.

The objective of this study is to determine the effect of selected cosolvents' addition on the solubility of TCE in water. In addition to the capability to enhance solubility, criteria considered for the selection of cosolvents were that the cosolvent: (i) must be non-toxic, (ii) should not form toxic byproducts, (iii) should be biodegradable, and (iv) must be relatively inexpensive. Results from this study will be applied to TCE-contaminated site remediation by combining chemical oxidation or bioremediation with cosolvent-assisted TCE extraction.

Based on a literature review of the use of alcohols as cosolvents, methanol, ethanol, and isopropyl alcohol were selected as cosolvents for TCE. The relevant physical/chemical properties of these alcohols are listed in Table 2 (MSDS Source: www.hazard.com/msds2).

All the alcohols considered have similar characteristics, which leads one to believe that they will give similar results. The only major difference is the vapor pressure and boiling point of methanol, which means that it will go into the vapor phase before the other alcohols. This may pose a problem in using techniques such as thermal enhancement, as the alcohol

may vaporize before fully solubilizing TCE. Methanol is a little cheaper but also slightly toxic and may offset its price advantage if the toxicity issue has to be addressed.

MATERIALS AND METHODS

TCE stock solution

Three stock solutions of TCE in hexane were prepared to use for further dilutions during experimentation. First, a 10,000-ppm stock solution was prepared by taking 10 ml of TCE and adding to it 990 ml of hexane. Next, a 1000-ppm stock solution was prepared by adding 10 ml of 10,000-ppm stock solution to 90 ml of hexane. Finally, a 100-ppm stock solution was prepared by adding 10 ml of 1000-ppm stock solution to 90 ml hexane. Pure TCE was used for higher concentrations when needed.

TCE analysis

TCE analysis was accomplished using a Hewlett-Packard Gas Chromatograph Model No.5890 Series II (GC), equipped with a packed column. Operating conditions for the GC are listed in Table 3.

Solubility experiments

A schematic diagram of the experimental system is shown in Figure 1. Equal volumes of solvent solutions containing cosolvent and water were prepared in dark vials. Percentages of cosolvent in cosolvent-water solution were varied (5%, 25%, and 50%), with a total solution amount of 10 ml. The total volume of each solution was 10.1 ml, when 0.1 ml of TCE was added to each solution after mixing the required amounts of alcohol and water. The vials were connected with mini-nert valves to prevent any TCE from escaping the system via volatilization. After all the solutions were made, the vials were placed on a rotary shaker and allowed to mix for six hours. Samples of 1 μL from the vial contents were then withdrawn and analyzed for TCE using the GC via a flame

Table 2. Cosolvent Properties.

| Property	Methanol	Ethanol	Isopropyl Alcohol
Boiling Point (ºC)	65	79	80
Vapor Pressure @ 20 ºC (mm Hg)	127	40	33
Melting Point (ºC)	-98	N/A	-86
Specific Gravity @ 20 ºC	0.791	0.79	0.786
Solubility in Water	100%	100%	100%
Carcinogenicity	No	No	No
Biodegradability in Soil	Readily	Readily	Moderately
Biodegradability in Water	Readily	Readily	Moderately
Half Life in Soil (days)	1-10	N/A	1-10
Environmental Toxicity	Slightly Toxic	Not Toxic	Not Toxic
LD_{50} (mg/kg)	5628	7060	5045
Bioaccumulation	No	No	No
Price ($/200L)	672.50	917.80	868.15
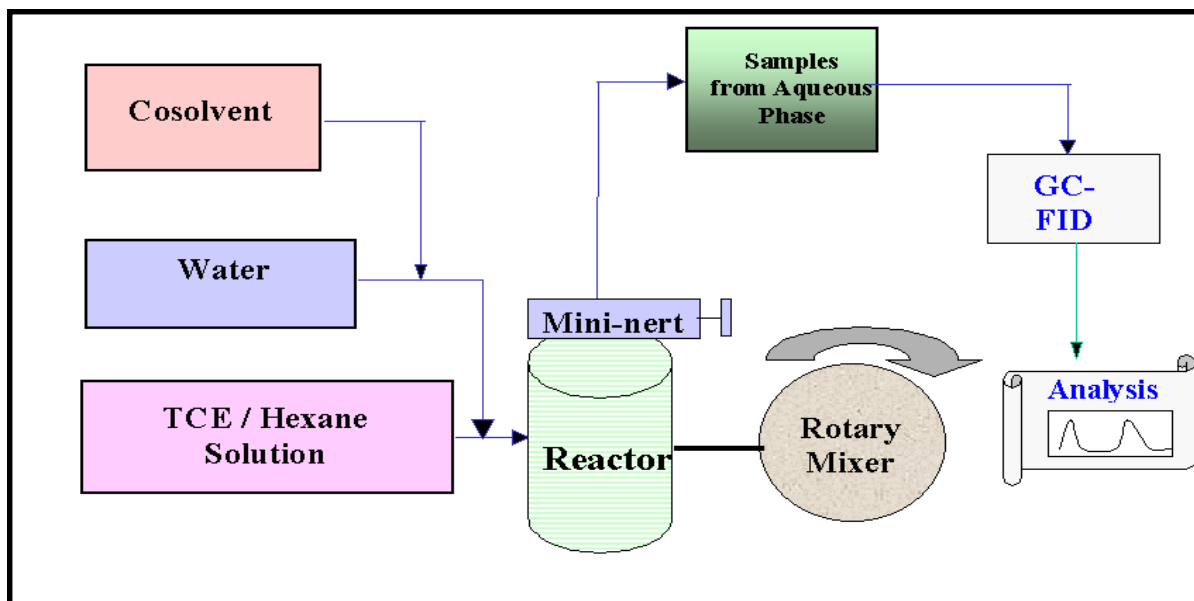
Figure 1. Schematic Flow Diagram for Batch Solubility Experiments.

ionization detector at the conditions reported in Table 3.

RESULTS AND DISCUSSION

Batch solubility studies were performed, analyzing three alcohols as cosolvents in water to increase the solubility of TCE. Mixtures of cosolvent-TCE-water system were analyzed using a gas chromatograph (GC). Figures 2-7 show TCE chromatograms for various conditions and systems. In all cases, 10 ml of cosolvent (methanol, ethanol, isopropyl alcohol) and water solutions were mixed with 0.1 ml of TCE, yielding a TCE concentration of 9990

Table 3. GC/FID Operating Conditions.

Parameter	Value
Injection Temperature	70 °C
Detector Temperature	200 °C
Oven Equilibrium Time	1 min
Oven Max	300 °C
Initial Temperature	50 °C
Rate	10 °C/min
Final Temperature	180 °C

ppm (v/v). The chromatograms are plots of time (seconds) on the x-axis and peak height (counts) on the y-axis. The area under the TCE peak on the chromatograms represents the corresponding TCE concentration.

The chromatograms for TCE in various TCE-methanol-water systems for 5%, 25%, and 50% methanol are shown in Figure 2. TCE concentration is proportional to the area under the corresponding peak and is determined from the calibration curves developed from the known concentrations of TCE in hexane. The 5% methanol peak shown in Figure 2 corresponds to a mixture of 5% methanol, 95% water on a TCE-free basis, and 0.1 ml TCE. This corresponds to a solubility of 11% of the original TCE in the mixture. The second higher peak corresponds to a mixture of 25% methanol, 75% water, and 0.1 ml TCE. As expected, more TCE was dissolved in the mixture of 25% methanol than 5% methanol, corresponding to 19% of the original TCE dissolved at the
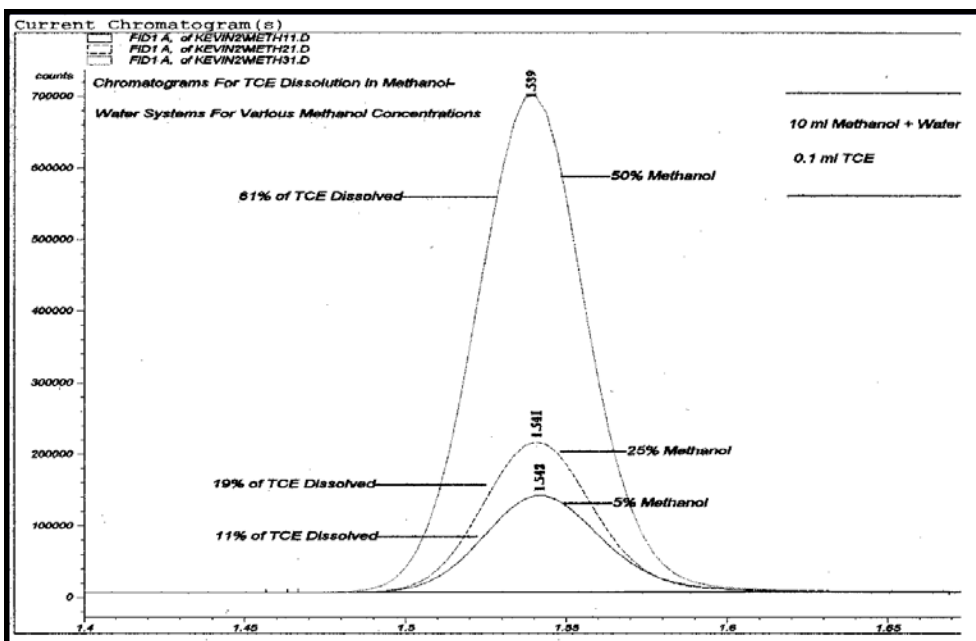
Figure 2. TCE Chromatograms for Methanol-TCE-Water System.

retention time of 1.540 seconds. Finally the cosolvent mixture containing 50% methanol and 50% water dissolved 61% of the original TCE. This increase is shown as the largest peak in the chromatogram in Figure 2.

Similarly mixtures of ethanol-TCE-water systems were analyzed using a GC, and the chromatograms are shown in Figure 3. Again three peaks are shown corresponding to in-creasing percentage of ethanol and resulting increased percentage of dissolved TCE. The 5% ethanol peak was found to dissolve 10% of the TCE. The 25% ethanol peak corresponds to 29% of original TCE dissolved in the solvent mixture. The largest peak on the chromatogram corresponds to a 50% solution of ethanol and was calculated to solubilize 85% of the original TCE.

Finally, solutions of isopropyl alcohol-TCE-water were studied at the same 5%, 25%, and 50% alcohol concentrations. The smallest peak in Figure 4 corresponds to 5% isopropyl alcohol, which dissolves 14% of the TCE. The

next peak for 25% isopropyl alcohol mixture was found to dissolve 31% of the TCE. Finally, the largest peak on the chromatogram refers to a 50% solution of isopropyl alcohol, 50% water, and 0.1 ml TCE. This peak, with a height count greater than 1.4×10^6 units, was calculated to dissolve 100% of the original TCE.

In Figures 5-7, the data have been pre-sented in a format that compares the %TCE dissolved at constant cosolvent fractions of 5%, 25%, and 50%. This format clearly shows the most effective alcohol at each concentration. Figure 4 shows a chromatogram of methanol, ethanol, and isopropyl alcohol, all at a concen-tration of 5%. From Figure 5, it can be seen that methanol and ethanol give about the same result, dissolving 10% and 11% of the TCE. Isopropyl alcohol dissolves 14% but is only slightly better than the other two alcohols at such a low concentration.
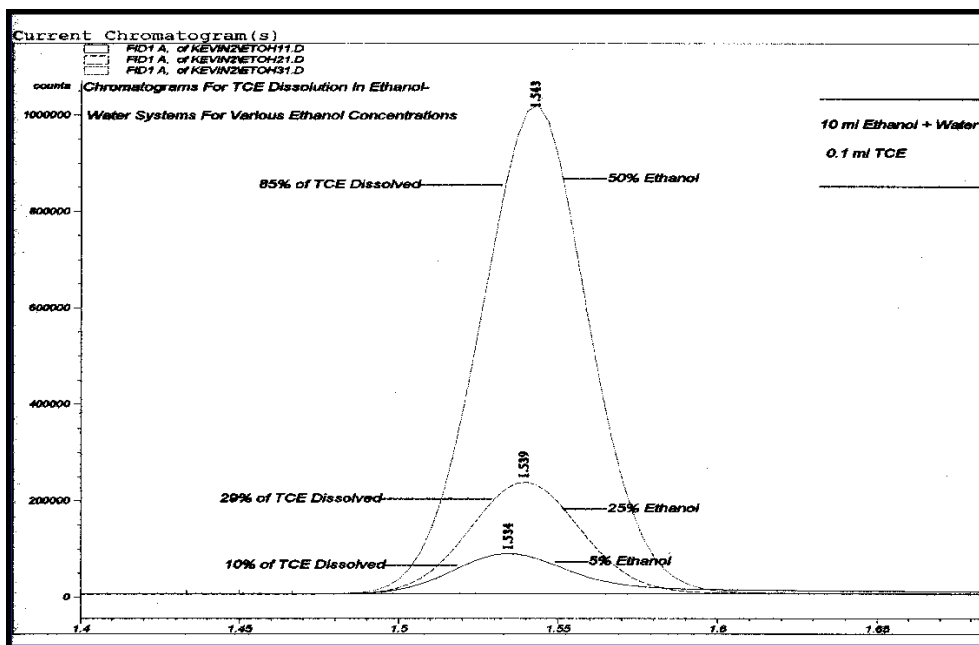
Figure 3. TCE Chromatograms for Ethanol-TCE-Water System.

In Figure 6, the three alcohols are again compared in a solvent mixture consisting of 25% alcohol and 75% water. In this experiment, methanol dissolved the least of the three alcohols with only 19% dissolved TCE. Ethanol and isopropyl alcohol performed about equally well, but about 50% better than methanol dissolving 29% and 31% of the original TCE in the mixture, respectively. Finally in Figure 7, methanol, ethanol, and isopropyl alcohol were analyzed in a solvent mixture consisting of 50% alcohol and 50% water. Results of the experiments show that methanol dissolved only 61% of the TCE, while ethanol outperformed methanol by dissolving 85% of the TCE. The largest peak again corresponds to the 50% isopropyl alcohol mixture, which performed the best of all three alcohols by dissolving all of the original TCE.

MATHEMATICAL MODELS
Power model

Three mathematical models were considered to represent these data. First, a power model representing a logarithmic relationship between cosolvent percentage in the solution on a solute-free basis, x, and TCE solubility, y, was explored. The corresponding mathematical representation of this model is shown in Equations (1) and (2).

$$y = p\,(x^n) \qquad\qquad (1)$$

or $\qquad \log y = \log p + n \log x \qquad (2)$

where p is the power model constant, which depends on the nature of the cosolvent and solute. A plot of log x vs. log y is shown in Figure 8, with percentage of cosolvent on the x-axis and the amount of TCE dissolved on the y-axis. This model was found to be unsuccessful as the data did not fall on a straight line. Table 4 gives a comparison of the power law constants
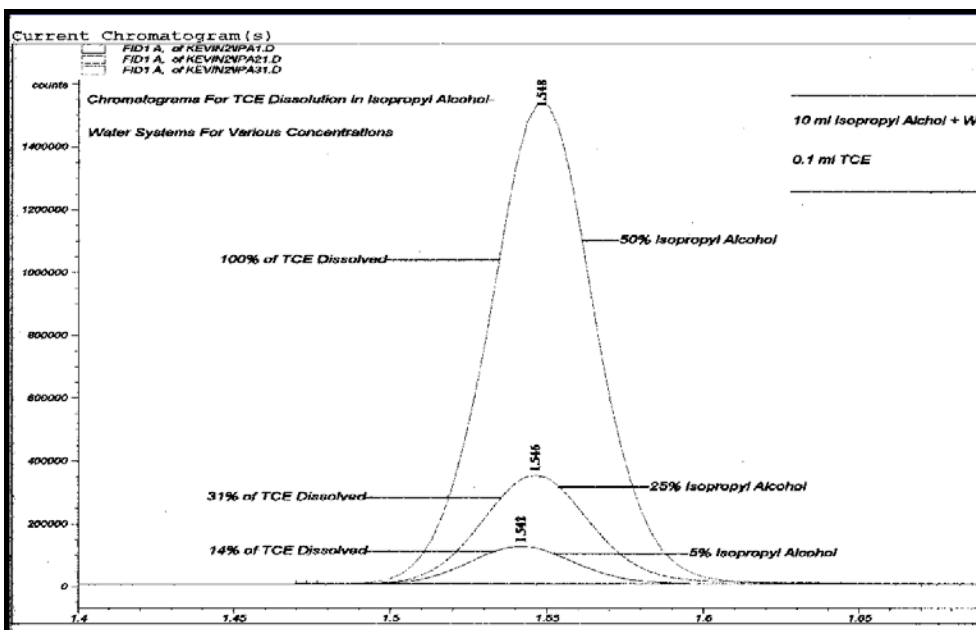
Figure 4. TCE Chromatograms for Isopropyl Alcohol-TCE-Water System.

and R^2 values for each cosolvent based on the power model.

Polynomial model

The second model considered to represent the data was a polynomial relationship. More specifically, quadratic representation of the form presented in Equation (3) was used.

$$\% \text{ TCE dissolved} = a\,x^2 + b\,x + c \qquad (3)$$

where x = percent cosolvent in the cosolvent-water mixture, and a, b, and c are constants.

Figure 9 is a representation of the experimental data for methanol with the percentage of methanol shown on the x-axis and the percentage of TCE dissolved on the y-axis. The equation of the line is given as a quadratic, and

Table 4. Power Model Constant and the R^2 Values for Alcohol Cosolvents.

Cosolvent	p	n	R²
Methanol	345.32	0.6607	0.824
Ethanol	302.44	0.7964	0.9161
Isopropyl Alcohol	202.82	0.967	0.9174

the R^2 value of 0.987 tells us that the function fits the data very well.

Similarly, a graph for ethanol can be found in Figure 10 showing the relative increase of dissolved TCE with respect to increased ethanol concentration. The curve is again quadratic with a higher first-term coefficient than that for methanol. The results display this by having a higher percentage of dissolved TCE for ethanol than methanol at given concentrations. Again, the equation can be seen from the graph, and the degree of accuracy of the function to the data, R^2, is excellent at 0.9987.

Finally in Figure 11, the plot for isopropyl alcohol is shown; this time the coefficient in the leading term was calculated to be higher than the other two alcohols. This, once again, shows the greatest amount of dissolved TCE. The R^2 value from the quadratic representation fit well with a value of 0.9997, making the quadratic equation, as previously for methanol and ethanol, an excellent fit.
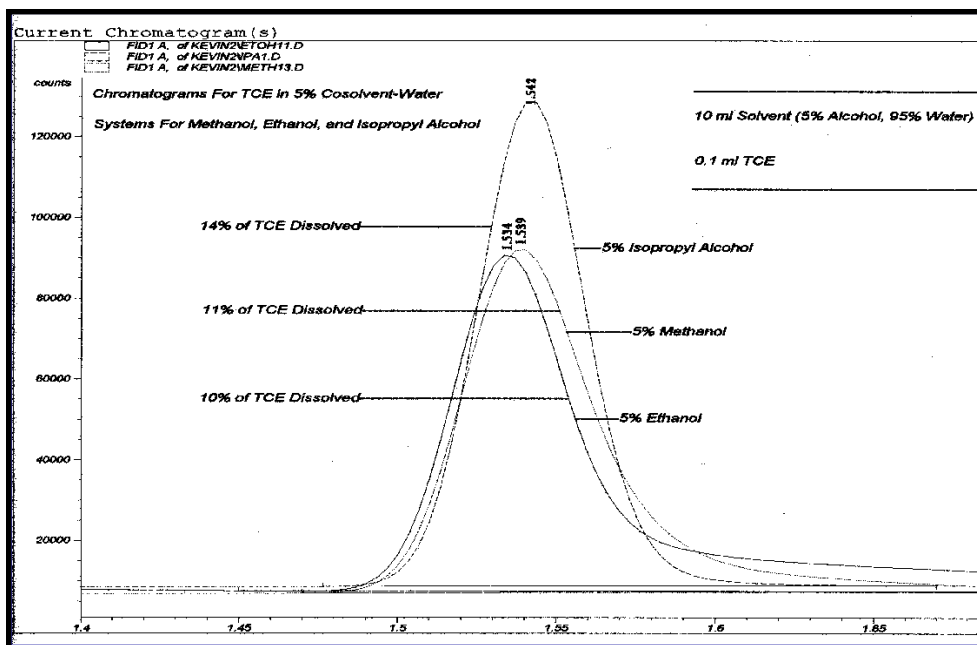
Figure 5. TCE Chromatogram for 5% Cosolvent-TCE-Water System.

The equations for the cosolvent represented by the quadratic equation can be found in Table 5. The R^2 values in the last column are much higher for the polynomial (quadratic) model (> 0.9987) than they are for the power model. This shows, mathematically, that the power model is a poor representation of the experimental data, while the polynomial model represented by the quadratic equation is a much better representation of the data.

In Figure 12, all three cosolvents are shown, giving a direct comparison of the alcohols. It can clearly be seen in Figure 12 that at 5% cosolvent all three alcohols dissolve about the same amount of TCE. At 25%

Table 5. Polynomial Model Constants and the R^2 Values for Alcohol Cosolvents.

Cosolvent	a	b	c	R^2
Methanol	0.0258	-0.2931	10.947	0.9987
Ethanol	0.0307	-0.0421	10.663	0.9997
Isopropyl Alcohol	0.0518	-0.4732	10.842	0.9998

cosolvent, isopropyl alcohol and ethanol dissolve about the same amount of TCE, while methanol dissolves much less. At 50% cosolvent percentage, isopropyl alcohol clearly gives the best results solubilizing all the TCE, followed by ethanol, with methanol solubilizing the least of the three alcohols.

Exponential model

Valsaraj (1995) discusses the situation of a leaking underground storage tank that releases gasoline, which comes in contact with the groundwater. Octane enhancers in the unleaded gasoline, which are completely soluble in water, act as cosolvents and increase the solubility of organics in water. The effect of cosolvent addition is to increase the net organic character of the resulting solvent (water + cosolvent), which decreases the hydrophobic interactions between the solute and water. Mackay (1982) presented a simple approach to quantify the solubility enhancement when cosolvents are
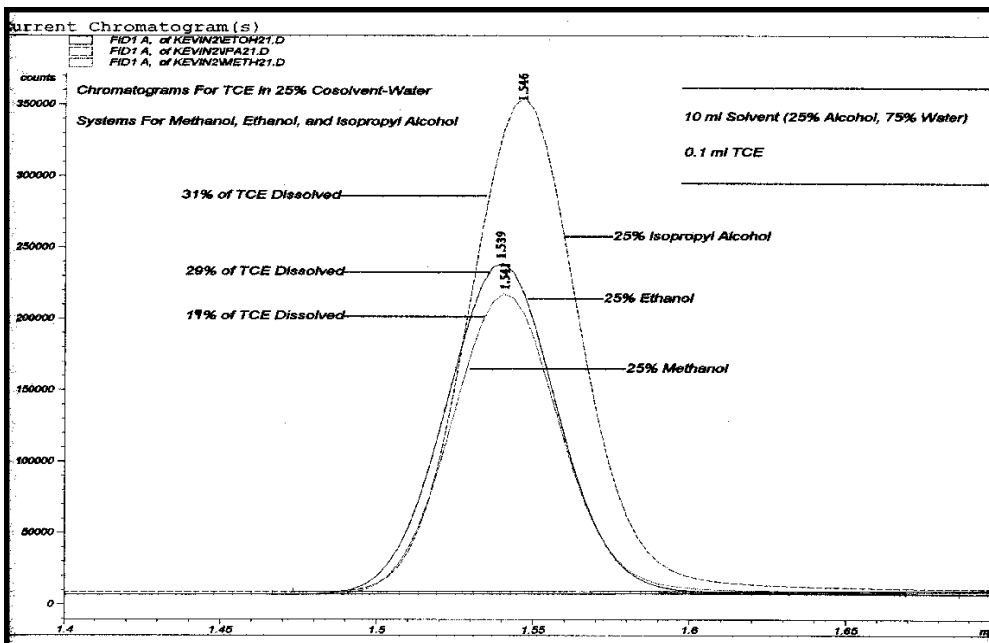
Figure 6. TCE Chromatograms for 25% Cosolvent-TCE-Water System.

added. Using a *two-suffix Margules Equation* involving activity coefficients of solute i in a ternary mixture consisting of water, w, cosolvent, c, and solute, i, the solubility enhancement function is calculated as shown in Equation (4) below:

$$\ln \gamma_{wi} = x_w^2 A_{i/w} + x_c^2 A_{i/c} +$$
$$x_w x_c (A_{i/w} + A_{i/c} - A_{w/c}) \qquad (4)$$

where γ_{wi} is the activity coefficient of solute i in water; x_i, x_w, and x_c are the mole fractions of solute, water, and cosolvent, respectively; and $A_{i/w}$, $A_{i/c}$, and $A_{w/c}$ are solute-water, solute-cosolvent, and water-cosolvent interactions, respectively. For very small solute concentrations (dilute solutions) $x_w + x_c = 1$, and for low cosolvent concentrations, Equation (4) simplifies to:

$$[(x_{wi})/ x_{wi}^0)] = \exp [A_{iwc} x_c] \qquad (5)$$

or

$$\ln [(x_{wi})/ x_{wi}^0)] = A_{iwc} x_c \qquad (6)$$

where x_{wi} is the solubility of solute i in the water-cosolvent mixture; x_{wi}^0 is the solubility of solute i in pure water; and A_{iwc} (= $A_{i/w}$ - $A_{i/c}$ + $A_{w/c}$) is a constant due to relative interactions between the solute, cosolvent, and water. Thus solubility enhancement would vary exponentially with the cosolvent mole fraction (or percent in the solvent), under certain conditions. Experimental data from our study were plotted according to Equation (6), and are shown in Figure 13. It is evident that the exponential model fits the data very well and since it is based on theoretical considerations, this model can be used for relative performance comparisons of various cosolvents. The value of the interaction constant, Aiwc, indicates the relative performance of the cosolvent and is shown in Table 6, along with the R^2 values of the fit according to Equation (3). Results indicate the relative strength of isopropanol to be 41% ($A_{iwc, isopropanol}$/$A_{iwc, methanol}$) and ethanol to be 25% ($A_{iwc, ethanol}$/$A_{iwc, methanol}$) over methanol.
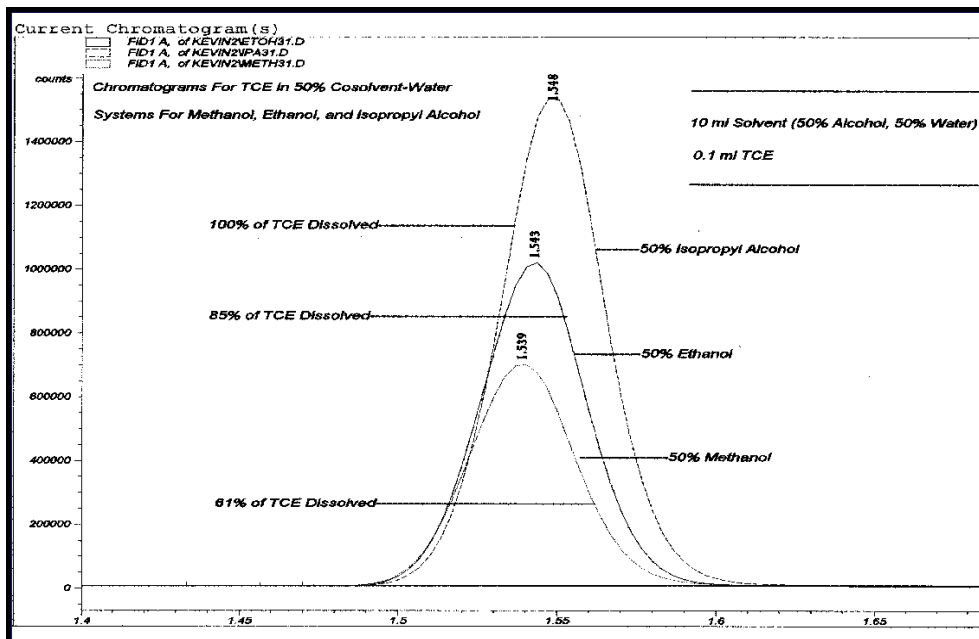
Figure 7. TCE Chromatograms for 50% Cosolvent-TCE-Water System.

Our results corroborate results from earlier studies of cosolvent-assisted solubility enhancement. Nyssen et al. (1987) used methanol, ethanol, and isopropanol to enhance the solubility of p-dichlorobenzene. Munz and Roberts (1986) used methanol and isopropanol for chloroform, and Valsaraj et al. (1991) used methanol, ethanol, and isopropanol to increase the solubility of pentachlorophenol. In all cases, isopropanol performed better than ethanol, which in turn performed better than methanol. Thus their results are qualitatively similar to the ones obtained in this study for TCE solubility enhancement.

Table 6. Exponential Model Constants and the R^2 Values for Alcohol Cosolvents.

Cosolvent	A_{iwc}	R^2
Methanol	0.0341	0.9659
Ethanol	0.0426	0.9993
Isopropyl Alcohol	0.0482	0.9950

Experiments in our laboratory are continuing at lower cosolvent concentrations and soil systems to develop quantitative models and remediation strategies for hazardous waste contaminated-sites and groundwater.

CONCLUSIONS

Results from this study showed that nontoxic cosolvents can be used to enhance the solubility of TCE in aqueous phase, removing a major barrier to *in situ* chemical or biological degradation. It was found that of the three alcohols (cosolvents) tested, 50% isopropyl alcohol solution performed the best, solubilizing of all available TCE (9900 ppm), followed by ethanol and methanol. Results of the experiment also show that solubility enhancement is best fit by a quadratic function of cosolvent concentration.

ACKNOWLEDGEMENTS

Amount Dissolved (mg/L)

100000

10000

Isopropyl Alcohol

$y = 202.82x^{0.967}$
$R^2 = 0.9174$

Ethanol

$y = 302.44x^{0.7964}$
$R^2 = 0.9161$

Methanol

$y = 345.32x^{0.6607}$
$R^2 = 0.824$

1000

1   10   100

Percentage of Cosolvent in Cosolvent-Water Mixture
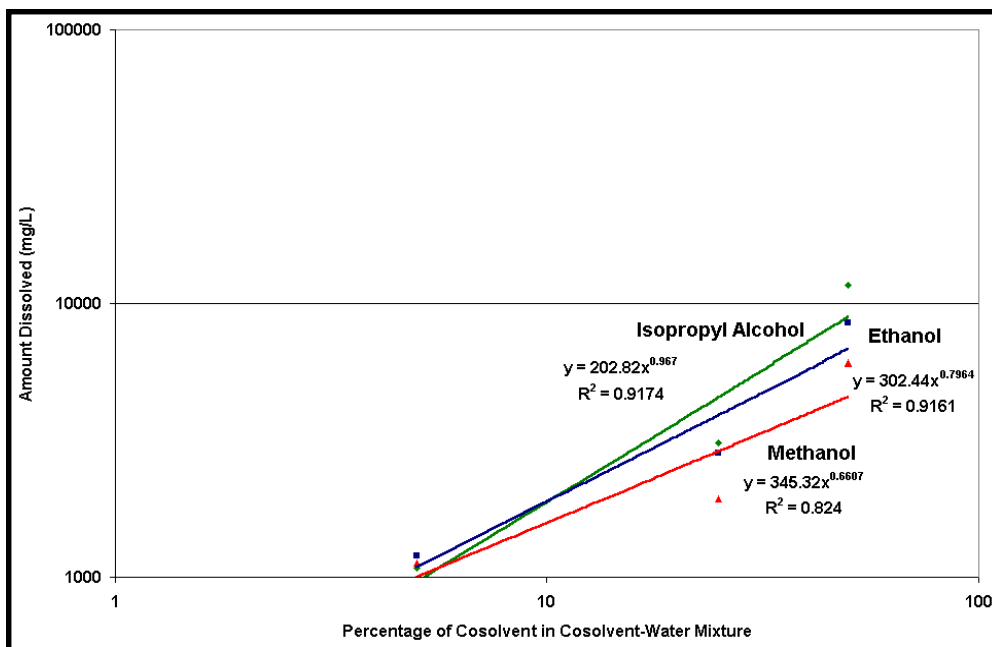
Figure 8. Log-Log Plot of Cosolvent Concentration Vs. TCE Solubility.

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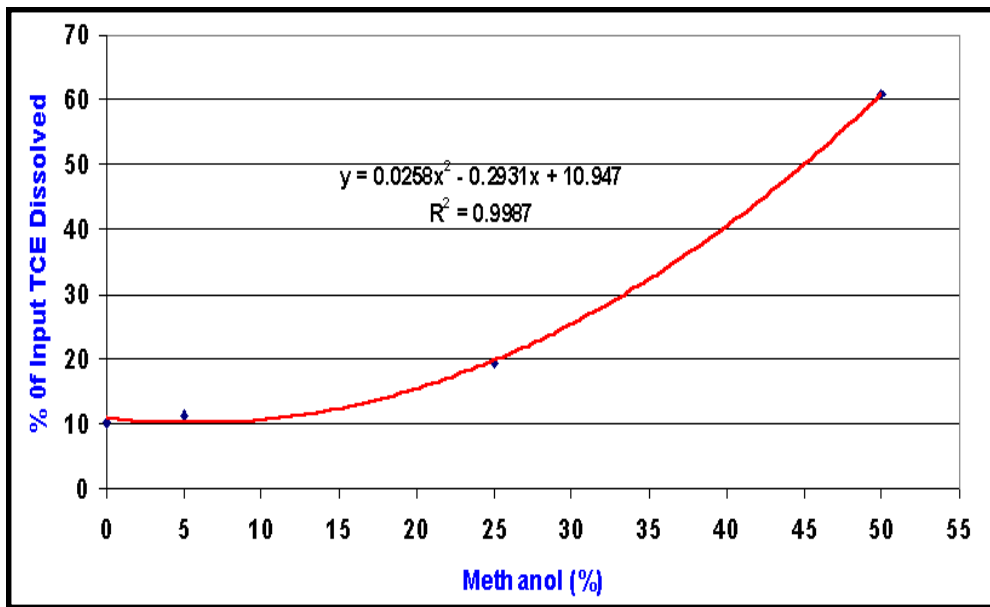
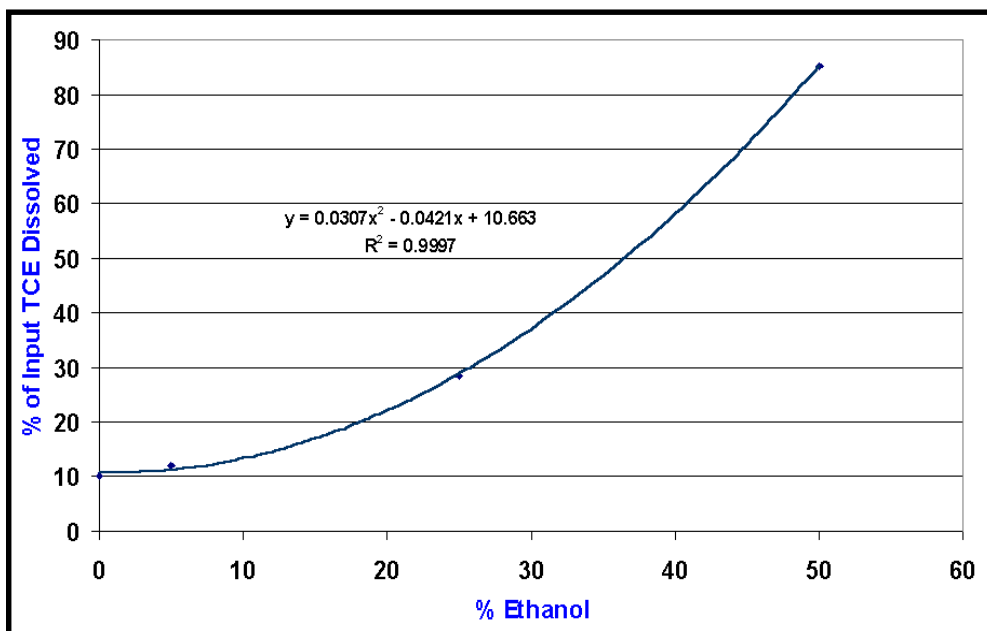**Figure 9.** TCE Solubility in Methanol-Water System.

Figure 10. TCE Solubility in Ethanol-Water System.

Russell, H.H., J.E. Matthews, and G.W. Sewell, 1992. TCE Removal from Groundwater, Groundwater Issue, EPA/540/S-92/002.

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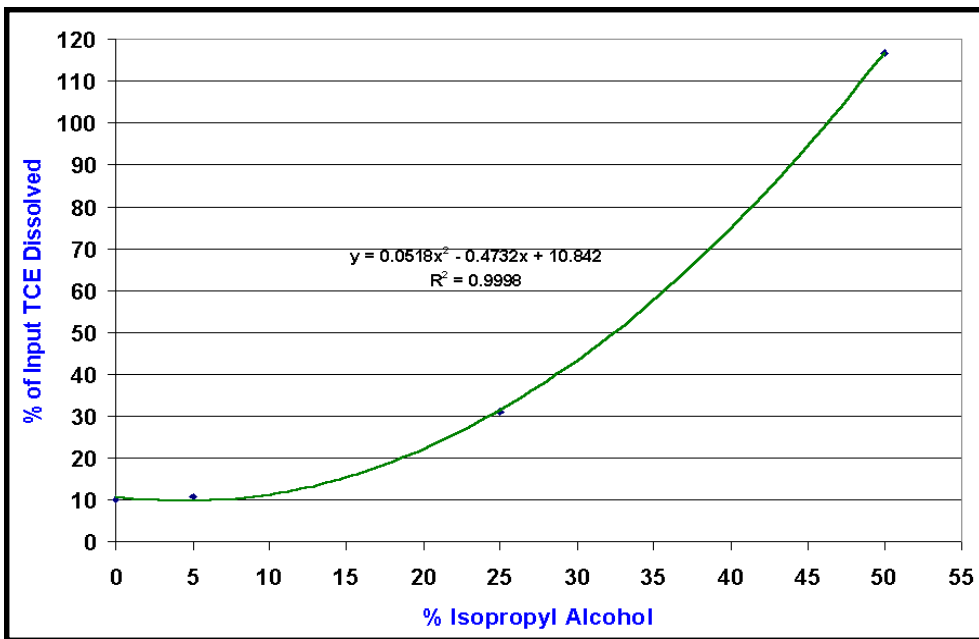
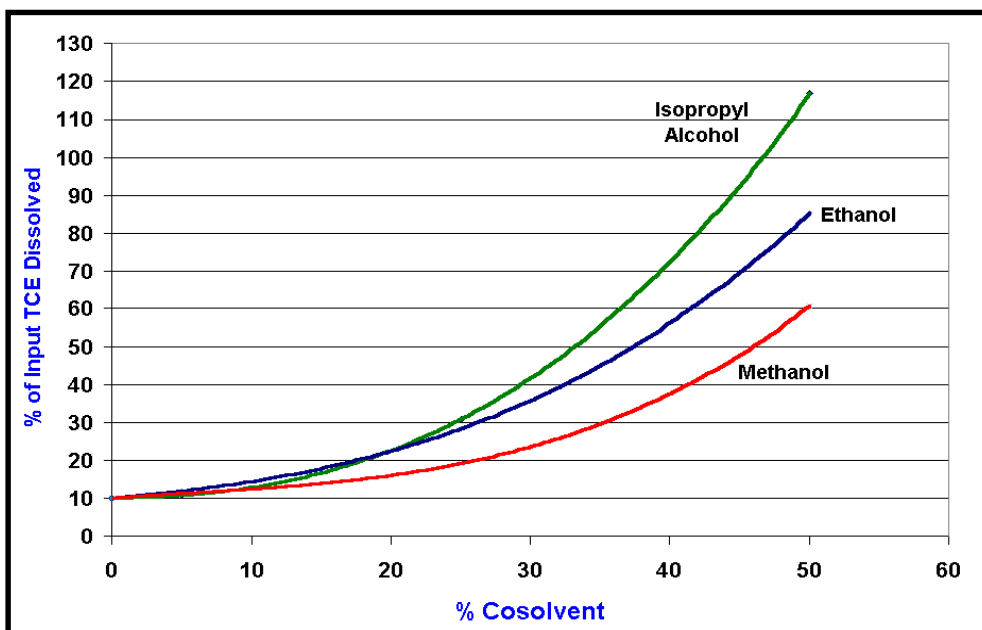**Figure 11.** TCE Solubility in Isopropyl Alcohol-Water System.

Figure 12. TCE Solubility in Cosolvent-Water Systems for Methanol, Ethanol, and Isopropyl Alcohol.
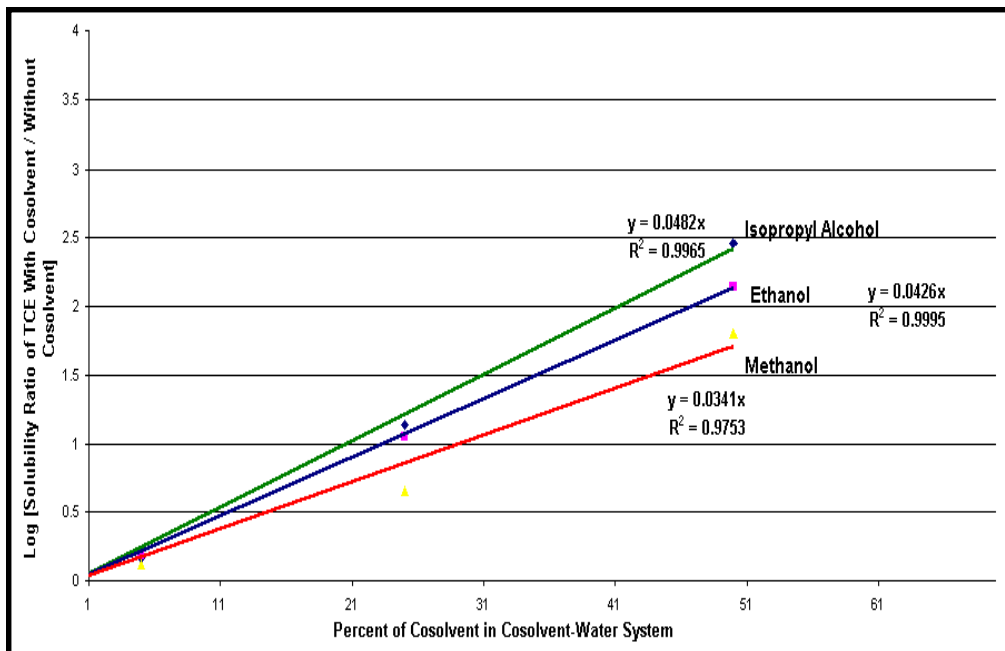
Figure 13. Log-Linear Plot of TCE Solubility Ration (With/Without Cosolvent) Vs. Cosolvent Percentage According to the Exponential Model.