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**Adsorption and Desorption of Atrazine from Various Lake
Sediments in Texas**

Report

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LITERATURE REVIEW¹

Atrazine (2-chloro-4-ethylamino-6-isopropylamino-s-triazine) has been used extensively throughout the United States for broadleaf weed control in corn, sorghum, and sugarcane production, as well as in several minor crops. In 2002, 32.0 million ha of corn and 3.9 million ha of sorghum were planted in the U.S (NASS 2003). In 2002, 0.8 million ha of corn and 1.3 million ha of sorghum were planted in Texas (TASS 2002). Approximately 34.7 million-kg of atrazine are applied yearly in the U.S., making it one of the most commonly used pesticides in this country. Eighty-six percent of this quantity is used in corn production, 10% is used in sorghum production, and 4% on remaining crops (EPA 2002).

Due to this extensive use, atrazine has been one of the most detected herbicides in U.S. surface water (Ma et al. 1997; Seybold et al. 1999). It has been estimated that 0.5% to 6.0% of the atrazine applied in agricultural systems is removed by surface runoff (Hall et al. 1972; Hall 1974; Triplett et al. 1978; Wauchope 1978). This leads to the main source of pesticide contamination in lakes (Spalding et al. 1994). Approximately 20% of the 50,000 community drinking water systems in the U.S. are supplied by surface water. Approximately 3,600 drinking water systems of the 20% supplied by surface water have had atrazine detections. The maximum contaminant level (MCL) of 3 ug L⁻¹ for atrazine has been met or surpassed in 200 of these 3,600 water systems. These 200 water systems are located in Alabama, Ohio, Illinois, Indiana, Iowa, Kansas, Kentucky, Louisiana, Michigan, Missouri, and Texas (EPA 2003).

¹ This proposal is in the format of Weed Science.

Due to the detections in Texas, some monitoring programs have been initiated. Eight lakes around the Dallas area of North Texas, were analyzed by the Texas A&M Pesticide Fate Laboratory in College Station, TX for the presence of atrazine in the lake water. These eight lakes consisted of Big Creek Lake, Joe Pool Lake, Lavon Lake, Bardwell Lake, Lake Waxahachie, Lake Aquilla, Lake Tawakoni, and Richland-Chambers Reservoir. Concentrations have primarily ranged from 0.3 ug L^{-1} to 0.5 ug L^{-1} through out the year in the source water of these lakes. Occasionally, concentrations have ranged from 30 to 50 ug L^{-1} atrazine during the spring months following atrazine applications. Higher concentrations of pesticides typically have been found in both ground and surface water following initial herbicide application (Pionke et al.1990; Spalding et al. 1989; Triplett et al.1978; Wauchope 1978; Wu et al. 1983). All eight lakes have been placed on the Texas 2000 Clean Water Act Section 303(d) list because the atrazine concentrations in finished drinking water indicated contamination of source water. Seven of these lakes were classified as “threatened” lakes meaning that the finished drinking water from these lakes had a concentration of 1.5 ug L^{-1} atrazine or 50% of the MCL. Aquilla Lake was classified as an impaired body of water due to the fact that its finished drinking water exceeded the MCL of 3 ug L^{-1} (TNRCC 2002).

Although not deemed a human carcinogen, atrazine has become a growing concern regarding human and wildlife health due to its presence in surface water. Sexual abnormalities in frogs and a decrease in length and weight have been linked to atrazine contamination (Carr et al. 2003; Diana et al. 2000; Morgan 1996; Reeder et al. 1998; Tavera-Mendoza 2002). Lab studies on frogs have shown observed sexual abnormalities at atrazine concentrations of $>20 \text{ ug L}^{-1}$. Above 200 ug L^{-1} of atrazine, both frog length

and weight decreased (Diana et al. 2000). High atrazine concentrations had no effect on survival (Diana et al. 2000). It was concluded in these studies that atrazine concentrations in natural water were too low to cause a threat to wildlife (Carr et al. 2003; Diana et al. 2000; Morgan 1996; Reeder et al. 1998; Tavera-Mendoza 2002). Recently it was reported that concentrations as low as 0.1 ug L^{-1} were shown to cause sexual abnormalities in frogs in the laboratory (Hayes et al. 2002). This concentration is below the EPA's MCL. The same study reported seeing frog abnormalities in natural water at concentrations ranging from 0.2 ug L^{-1} and greater although no other contaminants were studied (Hayes et al. 2002). A study on cricket frogs (*Acris crepitans*) concluded that 2.7% of frogs collected from different sites in Illinois during a 3-year period were intersexed. Along with atrazine, polychlorinated biphenyl (PCB) and polychlorinated dibenzofuron (PCDF) were found in the samples collected (Reeder et al. 1998). More research is needed to determine if atrazine is the sole cause of such abnormalities.

Atrazine, a symmetrical triazine, has several different fates in lake water including hydrolysis, photolysis, adsorption, and desorption (Gao et al. 1998; Goswami et al. 1971; Konstantinou et al. 2001; Mersie et al. 1998a; Moreau et al. 1997; Wauchope et al. 1985). Atrazine has a relatively low water solubility (33 mg L^{-1}) and moderate adsorption (average $K_{oc} = 100 \text{ ml g}^{-1}$), but little is known about the interaction of atrazine and sediment (Mersie et al. 1998b). Along with atrazine, three of its metabolites have been found in lake water (EPA 2003). These metabolites are desethyl atrazine (DEA), desisopropyl atrazine (DIA), and diaminochlorotriazine (DACT).

Often sediment has been questioned as a potential storage reservoir, transport vehicle, and ultimately a long-term contamination source of organic compounds like atrazine. Adsorption and desorption of organic compounds to soil depends on several different factors, including sand, silt, and clay contents, organic matter, pH, and aerobic and anaerobic environment. Soil and water pH plays a primary role in the adsorption behavior of atrazine. As pH decreases, the cationic fraction is increased, increasing atrazine adsorption to soil (Clay et al. 1990a; Gao et al. 1998; Rae et al. 1998; Weber 1970). Likewise, atrazine tends to desorb as pH increases (Jenks et al. 1998).

Atrazine adsorbs to both organic matter and clays. Organic matter has a greater affinity to atrazine compared to clay (Gao et al. 1998; Laird et al. 1994; Ying et al. 2000). Atrazine affinity to soil increases as organic matter increases (Patakioutas et al. 2002). This increase in organic matter decreases the amount desorbed (Jenks et al. 1998).

A decrease in soil particle size increases the adsorption of atrazine to soil and decreases the amount susceptible to desorption. Its high affinity to clay and silt particles is greater due to greater surface area compared with coarser textures (Sonon et al. 1995). However, if pH decreases and organic matter increases, or a combination of the two, the affinity of a sandy soil to atrazine increases (Gao et al. 1998).

In several studies, it has been shown that the total amount of atrazine adsorbed to a soil or sediment is not completely desorbed. Normally small fractions (5.5 to 40 %) of the amount adsorbed were desorbed demonstrating that relatively large amounts of atrazine potentially are adsorbed or degraded (Clay 1990b; Gao 1998; Jenks et al. 1998; Pignatello et al. 1991; Seybold 1996).

As in soils, sediments may have different adsorptive characteristics and, therefore, may have different affinities for organic compounds like atrazine. Unlike soil, sediment is found in an anaerobic environment and often contains a greater amount of organic matter, which may affect adsorption capacities of sediments (Seybold 1999).

A classification system for adsorption isotherms was designed by Giles et al. (1960). This design classified adsorption patterns of soils in four different categories. The different isotherms included the S, C, L, and H-type isotherms. The S-type occurs where lower concentrations of the pesticide remain in water, mid-level concentrations adsorb, and high concentrations reach a maximum adsorption. The C-type isotherm, demonstrates constant partitioning of a compound across a range of concentrations and a maximum adsorption is never reached. The L-type isotherm (Langmuir) demonstrates a relatively high affinity at lower concentrations, but then reaches maximum adsorption at higher concentrations. The H-type isotherm (high affinity) is similar to the L-type but has a higher affinity at low concentrations and reaches a maximum (Weber 1970).

The difference between the amount of a herbicide adsorbed to a soil or sediment and the amount of the herbicide desorbed from the same soil or sediment is known as hysteresis. No one mechanism influences the adsorption and desorption process of a compound in the same way. Several types of bonds, varying in strength between the sediment and herbicide, can cause adsorption. The same bonds influences the herbicides desorption. Bonds range from weak bonds such as London-van der Waals forces to strong bonds such as covalent bonds. In desorption, the weaker bonds are broken, but the stronger ones may not be over come. Without the release of these stronger bonds, equilibrium during the desorption process is never met. This in part explains the amount

of certain herbicides, which remain adsorbed even after several desorption cycles (Koskinen et al. 1990).

Another concern in the possible sediment storage and recontamination factor is the degradation and persistence of atrazine once adsorbed to the sediment. Past studies, have shown that the half-life of atrazine is variable among soils and sediments. The average field half-life is 60 d, but the half-life has ranged from 39 d to 261 d (Vencill 2002). In sediments collected from estuarine water, atrazine's half-life of 15 to 20 d was reported (Jones et al. 1982), and a half-life of 145 d was reported from Wisconsin lake sediments (Armstrong et al. 1967; Jones et al. 1982).

Many different factors contribute to atrazine degradation once adsorbed to soil. Atrazine is susceptible to breakdown by UV light (photolysis) after application into hydroxyatrazine and *N*-desethylated atrazine (Vencill 2002; Jordan et al. 1970). Photolysis is faster in soil systems than in water because photolysis is hindered by the presence of organic matter in surface water (Konstantinou et al. 2001). In soils at a moderately acidic pH ranging between 5.5 to 6.5, atrazine degradation to hydroxyatrazine is due to chemical hydrolysis, and at moderately neutral pH in soils, hydrolysis is partially driven by an increase in microbial activity (Vencill 2002; Blumhorst et al. 1994). Hydrolysis is slower at more basic values ranging from 7.5 to 8 in soils (Vencill 2002; Blumhorst et al. 1994). In anaerobic environments, pH is typically higher than aerobic situations and has a low rate of atrazine hydrolysis and also atrazine dealkylation by microbes (Goswami et al. 1971; Jones et al. 1982; Klint et al. 1993).

Soil bacteria can drive microbial hydroxylation and mineralization. Some *Pseudomonas* species have been observed to mineralize atrazine in both aerobic and

anaerobic environments (Shapir et al. 1998). In one study, over 30 atrazine-degrading bacteria cultures were found in soil samples. In this study, it was concluded that hydrolysis of atrazine mediated by bacteria is wide spread in soils (Mandelbaum et al. 1993).

Soil characteristics also play a role in degradation of atrazine. Soils with higher organic matter typically degrade atrazine more efficiently through microbial degradation (McCormick et al. 1965). Soil characteristics can influence the sequestration of atrazine. Organic carbon, soil texture, and cation exchange capacity play a large role in holding atrazine unexcessable for microbes. (Radosevich et al. 1997). Estimated rates of atrazine degradation in soil and sediment have differed substantially depending on the study. Atrazine degradation was slower in submerged soils than in aerobic soil (Goswami et al. 1971). In this study, actual soil samples treated with atrazine were submerged. Jones et al. (1982) found that atrazine degradation was faster in sediment/water systems than in soil systems. In this study both sediment and soil were studied. Although some work has been documented regarding atrazine behavior, it is not known what effect differing sediment characteristics have on atrazine contamination in surface water.

HYPOTHESIS

Lake sediments differ in characteristics and, therefore, differ in their ability to adsorb and desorb atrazine.

OBJECTIVE

To determine relative adsorption and desorption of various Texas lake sediments to atrazine.

MATERIALS AND METHODS

Eight lake sediments were collected from sites where atrazine was detected during an earlier study. All eight lakes were located in northeast Texas and were used as community water use systems.

Sediment collection and preparation.

Sediment samples were collected using an Eckman dredge from a bridge or boat depending on site availability at each individual lake. Samples were placed in 3.75-L Ziploc bags and placed in a cooler for transport back to the laboratory. Once back at the laboratory, samples were dried on paper in the greenhouse. The sediment samples were then ground with a soil grinder and then a mortar and pestle as needed to get uniform particle size. After grinding, sediments were passed through a 2-mm sieve. Subsamples were characterized by the Texas A&M Soil Characterization Laboratory for texture, percent organic carbon, pH, CEC, extractable bases, and % base saturation.

Batch equilibrium adsorption and desorption.

Four 2-g samples were weighed out for each collection and placed into separate 35-ml centrifuge tubes. Initial standard solutions were made using a ring-labeled atrazine standard combined with an analytical grade atrazine standard in methanol. One hundred μL of the initial standard solutions were added to each centrifuge tube along with 5-ml of 0.01M CaCl_2 solution to provide a constant ionic strength during the adsorption-desorption experiment. This provided a concentration of approximately $8.41 \text{ Bq mL}^{-1} \text{ }^{14}\text{C}$ material in all samples, and 0.5, 1.0, 2.0, and 5.0 $\mu\text{g mL}^{-1}$ of analytical grade, non-radio labeled atrazine.

The samples were placed on a table shaker and shaken for 24 h. Tubes were then removed and centrifuged for 10 min. A 2-mL aliquot were then removed from the supernant of each sample and placed in glass scintillation vials along with 10 ml of scintillation fluid for the adsorption study. The remaining solution (1ml) was removed from the samples and replaced with fresh 0.01M CaCl₂ solution. Samples were then shaken again for 24 h. Another 2-ml aliquot were removed and placed in glass scintillation vials along with 10 ml of scintillation fluid for analysis of atrazine desorption. The procedure was repeated two more times in a 48-h period. The aliquots in scintillation fluid were then placed on a Beckman LS6500 Multipurpose scintillation counter. Each sample was counted for 20 min.

The relative affinity of atrazine to a sediment was determined by the Freundlich equation.

$$X/M = KC^{1/n} \quad (1)$$

$$\log X/M = \log K + 1/n \log C \quad (2)$$

Where, X/M equals the amount adsorbed per amount adsorbent, C represent the solution concentration at equilibrium, and K and n are constants. K represents the affinity of the herbicide to the sediment, and 1/n is the slope. If n is approximately 1, K represents the distribution coefficient, K_d.

Isotherms were characterized, by graphing X/M on the y-axis and C on the x-axis. Hysteresis was determined, by graphing the adsorption amounts using log X/M on the y-axis and log C on the x-axis. Desorption of two initial concentrations of 1.0 ug mL⁻¹ and 5.0 ug mL⁻¹ were chosen to determine desorption behavior of atrazine from the selected sediments. These desorption amounts were subtracted from the total amount adsorbed at

the specified concentration. The result was the amount of atrazine remaining adsorbed after the consequent desorption steps. The difference between adsorption and desorption showed the amount of atrazine that was desorbed. This also provided a way of determining the degree of hysteresis.

The experiment was executed in a completely randomized block design and replicated four times. Linear regression was used to determine K_d . Percent desorption was determined using total amount atrazine adsorbed and total amount of atrazine remaining adsorbed after desorption process. The general linear model (GLM) procedure was used to determine differences between K_d values and percent desorption between the different lake sediments (SAS 1985). Tukey's studentized range was used for mean separation of K_d values and percent desorption to determine differences between adsorption and desorption of the various sediments.

RESULTS AND DISCUSSION

The texture of the sediments varied from a loamy fine sand at Richland-Chambers Reservoir to a clay at both Big Creek Lake and Aquilla Lake (Table 1). The organic carbon content ranged from a 0.45% at Richland-Chambers Reservoir to a 2.98% at Big Creek Lake (Table 2). This is approximately a difference of 2.5%. The pH of the sediments ranged from a 6.4 at Big Creek Lake to a 7.8 at Lake Tawakoni (Table 3). Both Big Creek Lake and Aquilla Lake are both clays and high in % organic carbon, but the sediment at Aquilla Lake has a pH of 7.4, a one-point increase in pH from the sediment of Big Creek Lake. It has been stated, as pH decreases, it increases atrazine adsorption (Clay et al. 1990a; Gao et al. 1998; Rae et al. 1998; Weber 1970).

On the graphical representation of the Freundlich Equation for Big Creek Lake, Lake Waxahachie, Richland Chambers Reservoir, and Aquilla Lake, log of X/M is found on the y-axis and log of C is found on the x-axis (graph 1, 2, 3, &4). The solid black line represents the adsorption curve with points located for initial concentration 0.5 ug ml⁻¹, 1.0 ug ml⁻¹, 2.0 ug ml⁻¹, and 5.0 ug ml⁻¹. The y-intercepts for Big Creek Lake, Lake Waxahachie, Richland Chambers Reservoir, and Aquilla Lake were 0.65, 0.48, 0.26, and 0.40 respectively. The y-intercept is used to determine K adsorption values for each lake. All adsorption curves were linear and had n values ranging from 0.98 to 1.02. Since n was approximately 1.00, K = K_d can be assumed. Initial concentrations of 1.0 ug ml⁻¹ and 5.0 ug ml⁻¹ were chosen to draw desorption curves. Desorption curves (dashed lines) for Big Creek Lake, Lake Waxahachie, Richland Chambers Reservoir, and Aquilla Lake show that 78%, 84%, 92%, and 88% of the atrazine initially adsorbed remains adsorbed to the sediment respectively (Graphs 1, 2, 3, &4). Hysteresis is present due to the difference in adsorption and desorption (Graph 1, 2, 3, &4). If hysteresis did not occur, the amount initially adsorbed would then have been desorbed, causing the desorption curves to fall on top of the adsorption curve.

K_d values ranged from 1.8 at Richland Chambers Reservoir to a 4.5 at Big Creek Lake (Table 4). Sediment from Richland Chambers Reservoir was a loamy fine sand, low in organic carbon, and had a pH of 7.7. Sediment from Big Creek Lake consisted of clay, high organic carbon and a pH of 6.4. Sediment from Aquilla Lake had a K_d value of 2.5, which differed by 2 points from Big Creek Lake. Aquilla Lake was also a clay, high in organic carbon, but had a pH of 7.4.

The K_d value of Big Creek Lake was significantly different than all other sediments. The K_d values of Richland Chambers Reservoir and Lake Tawakoni were not significantly different from each other but were significantly different than all other sediments. There was a +.84 correlation between percent organic carbon and K_d , there was a -.75 correlation between pH and K_d , and a +.74 correlation between percent clay and K_d .

Percent desorption ranged from 7.6% at Richland Chambers Reservoir to 21.6% at Big Creek Lake, a difference of 14% (Table 4). Lake Tawakoni and Richland Chambers Reservoir consisted of fine, loamy, sandy type materials low in organic carbon. The percent desorption of these two lakes were significantly different than the percent desorption of Joe Pool Lake, Lavon Lake, and Big Creek lake, which consisted of higher clay content and higher organic carbon content.

A correlation of +.81 is seen between K_d values and percent desorption (Graph 5). The more atrazine a sediment adsorbs, the more atrazine a sediment desorbs. The less atrazine a sediment adsorbs, the less atrazine a sediment desorbs.

Conclusion

There were differences in atrazine adsorption and desorption to the various sediments. This suggests that availability in water once sediment has been contaminated depends on characteristics of individual sediments.

Sediment Data

Texture

Big Creek Lake	Clay
Joe Pool Lake	Silty clay
LavonLake	Silty clay
Bardwell Lake	Silty clay
LakeWaxahachie	loam Silty clay
Aquilla	loam Clay
Lake Tawakoni	Very fine sandy
Richland-Chamber Reservoir	loam Loamy fine sand

Table 1. Texture characteristics of 8 various lake sediments. Samples were characterized by the Texas A&M Soil Characterization Laboratory, College Station, TX

Sediment Data

% Organic Carbon

Big Creek Lake	2.98
Joe Pool Lake	1.87
Lavon Lake	2.07
Bardwell Lake	1.37
Waxahachie Aquifer	1.45
Lake	2.08
Tawakoni Reservoir	0.58
Richard Chamber Reservoir	0.45

Table 2. Organic Carbon content of 8 various lake sediments. Samples were characterized by the Texas A&M Soil Characterization Laboratory, College Station, TX

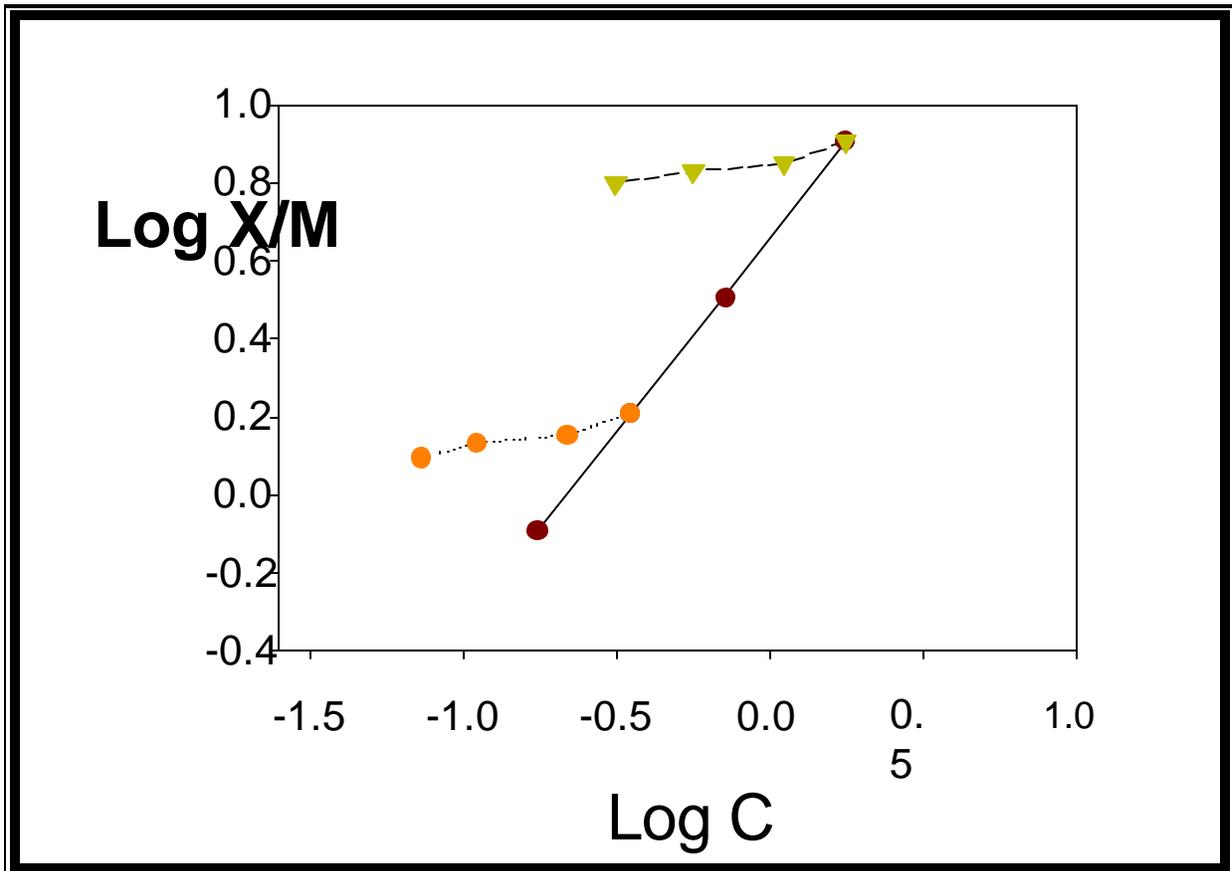
Sediment Data

pH

Big Creek	6.4
Joe Pool Lake	7.5
Lavon Lake	7.4
Bardwell Lake	7.7
Lake Waxahachie	7.5
Aquilla	7.4
Lake Tawakoni	7.8
Richland-Chamber Reservoir	7.7

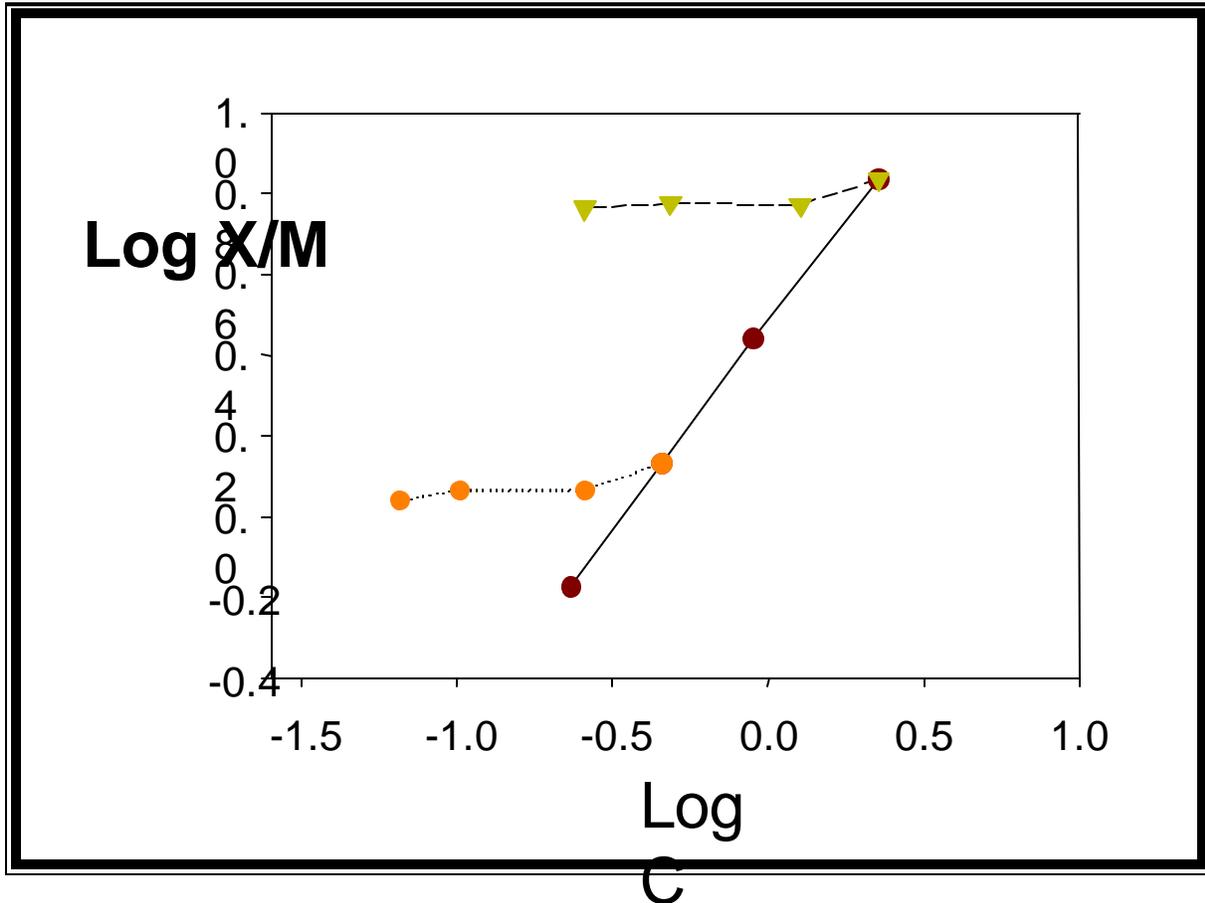
Table 3. pH of 8 various lake sediments. Samples were characterized by the Texas A&M Soil Characterization Laboratory, College Station, TX

Big Creek



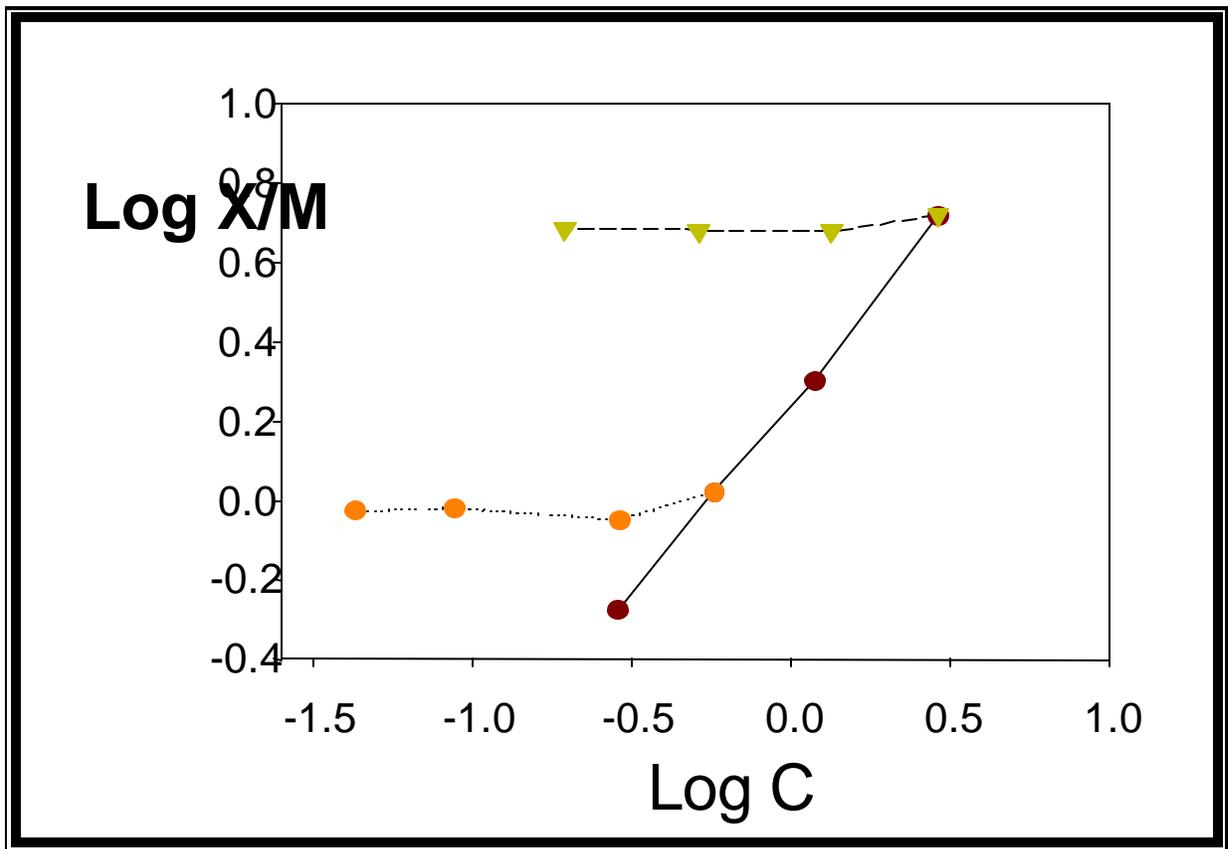
Graph 1. Graphical representation of the Freundlich equation.

Waxahachie



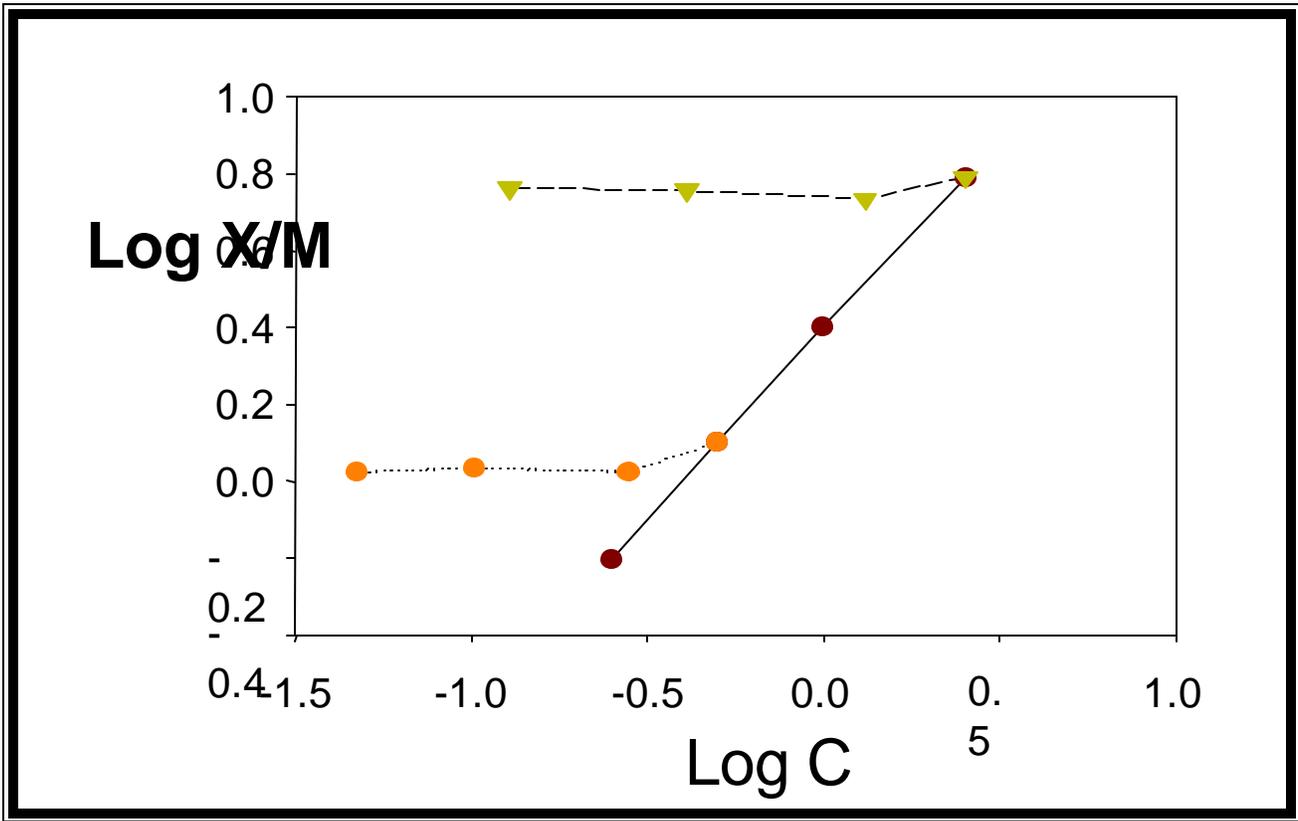
Graph 2. Graphical representation of the Freundlich equation.

Richland Chambers



Graph 3. Graphical representation of the Freundlich equation.

Aquilla

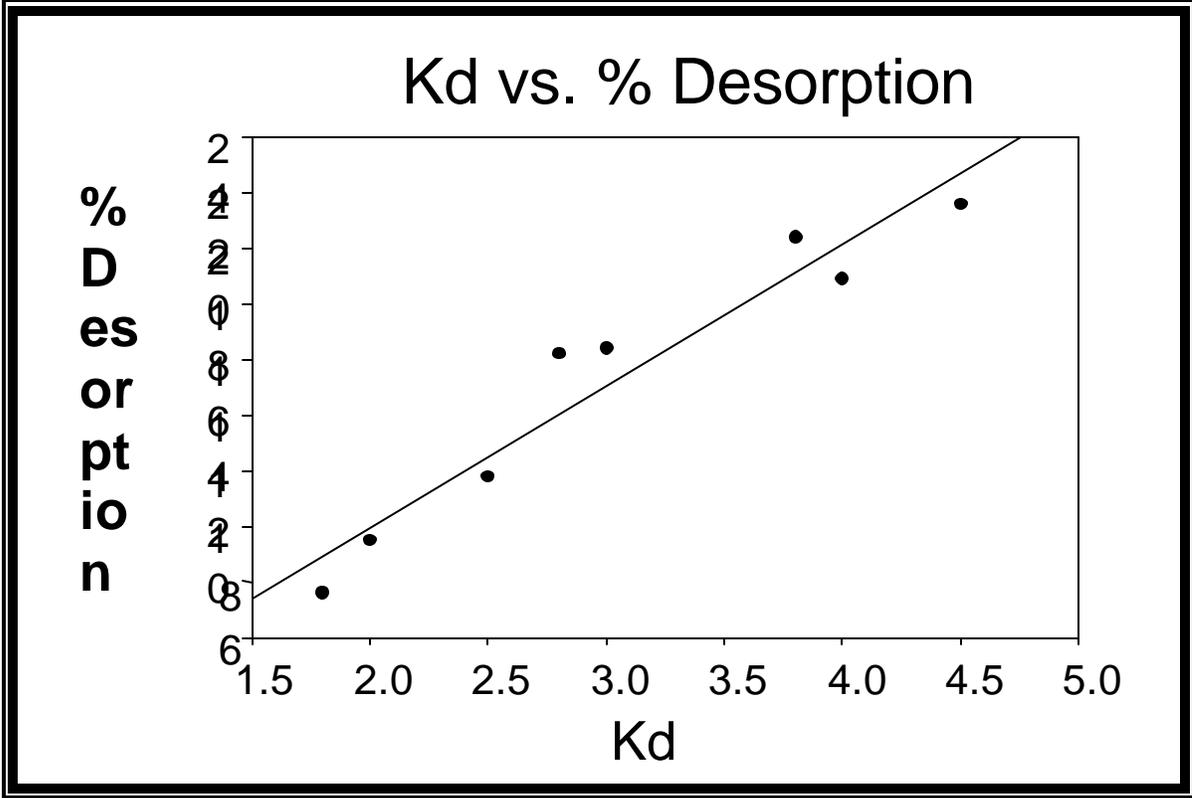


Graph 4. Graphical representation of the Freundlich equation.

Adsorption/Desorption

<u>Collection Site</u>	<u>Kd</u>	<u>n</u>	<u>% Desorption</u>
Richland Chambers Reservoir	1.8	0.98	7.6
Tawakoni Lake	2.0	0.98	9.5
Aquilla Lake	2.5	0.99	11.7
Lake Waxahachie	2.8	1.00	16.2
Bardwell Lake	3.0	1.02	16.4
Lavon Lake	3.8	0.97	20.4
Joe Pool Lake	4.0	0.99	18.9
Big Creek Lake	4.5	0.99	21.6

Table 4. pH of 8 various lake sediments. Samples were characterized by the Texas A&M Soil Characterization Laboratory, College Station, TX



Graph 5. Relationship between Kd and % Desorption.

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