

CYANIDE MOBILITY IN SOILS

by

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ABSTRACT

Three solutions of cyanide, KCN in de-ionized water (simple form), $K_3Fe(CN)_6$ in de-ionized (complex form) and KCN in natural landfill leachate (mixed form) were each leached through five soils (Ava, Kalkaska, Mohave (calcareous), Molokai and Nicholson) of varying physical and chemical properties to evaluate which soil characteristics govern the movement of the various cyanide forms in soils. The effluent from each column was collected and analysed for total cyanide each day. In general, KCN and $K_3Fe(CN)_6$ in water were both found to be very mobile in soils, while KCN in leachate was found to be less mobile. Soil properties such as low pH, presence of free-iron oxide and kaolin, chlorite and gibbsite type clay (high positive charge) tended to increase attenuation of cyanide in the three forms. High pH, presence of free $CaCO_3$ (high negative charges), low clay content and montmorillonite clay tended to permit greater mobility of the three cyanide forms.

INTRODUCTION

The frequency of groundwater pollution by hazardous toxic elements is steadily on the increase in the United States. With existing air and surface water pollution regulation forcing an ever increasing volume of toxic chemical waste to the land for ultimate disposal, particularly in heavily populated and industrial regions of the United States, many aquifers in various regions of the United States may be in danger of serious water quality degradation if soils are not effective in preventing hazardous waste from migrating downward to the ground water table, when they are recharged by rainfall infiltrating through the land surface. The need for research in this area seems clear. Presently there is a great deal of interest in evaluating soils as a filter for a variety of hazardous chemicals. Cyanide is one of the chemicals of concern.

Definition of Cyanide

Because cyanide appears in a variety of forms in nature and industry a strict definition is required so that one may proceed in an understandable manner. Taras (1971) refers to cyanide in industrial waste as all the CN^- groups present in the cyanide compound that can be determined as cyanide ion, CN^- , by the Liebig distillation method.

Cyanide are further classified into (a) simple and (b) complex groups.

Simple Form: $A(CN)_x$

Where A = an alkali (Na, K, NH_4) or a metal

X = the valence of A and the number of CN groups

CN is present as CN^-

Complex Form: $A_yM(CN)_x$

Where A = the alkali metal present y times

M = the heavy metals (Fe^{2+} , Fe^{3+} , Cd, Cu, Ni, Ag, Zn and others)

x = the number of CN^- group and equal to the valence of A taken y times plus that of the heavy metal.

The anion radical in the complex cyanide appears as $M(CN)_x^-$. When the simple cyanide comes in contact with acids HCN forms.

The metal cyanides vary widely in their decomposition to HCN in acid. As a matter of convenience they may be further grouped, on a basis of rate of decomposition, as

(1) Readily Decomposable--metallic forms of Ag, Au, Cd, Cu, Ni, Pt and Zn.

(2) Slowly Decomposable--Fe and Co.

The stability of alkali metallic cyanide also vary in aqueous solution alone, many remain rather stable in water. Because of the toxicity of CN^- , the formation of a more stable cyanide has been a significant factor in the activity of biological system.

Sources of Cyanide in Soils

Cyanide is introduced into the soil by natural means and through the activity of man. It is produced naturally by several fungi (Bach, 1956), at least one bacterium (Michael and Corpe, 1965) and many members of the higher plant community (Robinson, 1962). The amount produced by these organisms is rather insignificant when compared to the quantity which is discarded each day as a waste product of some modern day industries, having been used extensively since the 1800's for extracting precious metals and for stripping undesirable foreign substances from metal surfaces before plating. The cyanide bearing waste is introduced into the waste system by drag-out losses, leakage and accidental tank spills. Rudolph (1953) estimated the cyanide (CN^-) concentration in the waste stream of cyanide plants varied from 100 mg/liter to 50,000 mg/liter depending on the efficiency of the plant and the process involved.

Cyanide Reaction in the Soil

Virtually no organic compound is left immune to degradation by the highly versatile microbial population. Cyanide is no exception, despite the fact that it is highly toxic to the biological system as CN^- . Cyanide is also utilized as an energy source and/or source of nitrogen by plants and microorganism. Ware and Painter (1955) isolated

an anaerobic autotrophic actinomycete contained in sewage which is capable of growing on silica gel containing only KCN as a source of carbon and nitrogen. Allen and Strobel (1966) found several fungi which assimilated or transformed CN^- in synthetic media. Strobel (1967) went as far as to say that a cyanide cycle exists in the soil similar to that of carbon and nitrogen. In fact cyanide and related compounds such as cyanamids, dicyanodiamids and granidine nitrite have long been regarded by agriculturalists as potential nitrogen fertilizers. As early as 1918 Cowie (1919) recognized that cyanamids can serve as a valuable fertilizer because it forms ammonia readily in soils. Cyanide (CN^-) added to soil in modest amounts (up to 200 ppm $NcCN$) appears to be readily transformed and/or degraded depending on oxidation/reduction conditions. McCool (1945) suggests it is only slightly less effective as a N-fertilizer for corn, tobacco and mustard than $NaNO_3$ when applied to nitrogen deficient soils. Cyanide as KCN was shown by Strobel (1967) to yield CO_2 and NH_3 in the presence of non-sterile soils. He suggested that cyanide is fixed by various soil organism in several ways, all of which give rise to some organic nitrile. The nitriles yield ammonia plus the corresponding organic acids as a result of nitrilase activity. Many microorganisms of the soils can utilize ammonia and fix the nitrogen in the form of living cells.

Cyanide Toxicity

Although numerous organisms exist which can degrade CN^- , the CN^- is still one of nature's most toxic compounds. The highly toxic nature of it has been well documented. Simple alkali cyanides and many alkali-metallic cyanides, which form CN^- in aqueous solutions may decompose slowly to form varying degrees of toxicity. The level of toxicity of the more stable cyanide depends on the metal present and the proportion of CN^- groups converted to simpler alkali cyanides. Ludzack, Moore, Krieger and Ruchhoft (1951) and Schant (1939) found cyanide (CN^-) to have an inhibitory effect on fish life at concentration of 0.3 ppm. The Advisory Committee on Revision of U. S. Public Health Service (1961) suggested .01 mg of HNC/liter to be the limiting concentration of cyanide in drinking water. These figures seem to give a clear indication of the harmful effect uncontrolled dumping of cyanide waste could have on aquatic life and human health. Fortunately, cyanide waste is treated in most plants. Methods range from chemical treatment (alkaline chlorination, acidification, ponding, complexation, ozonation and electrolytic oxidation) to biological treatment using activated sludge (Murphy and Nesbitt, 1964). Although some of these processes (alkaline chlorination and activated sludge) are quite effective none of them completely remove the cyanide.

Objectives

Despite the fair amount of information on cyanide in the soil there still exists a number of critical gaps which need filling before predictions concerning the fate of CN^- in the soil can be made. The object of this investigation is (1) to measure cyanide mobility in soils and (2) to determine those soil properties which govern the mobility and/or attenuation of cyanide in the soil.

MATERIALS

Soils

The soils used for this study were collected from various parts of the United States, covering four major soil orders.

- (1) Ava (Illinois)--is an Alfisol, a Typic Fragiudalf. Ava was collected from the B (argillic) horizon. It has a yellowish brown color, a silty clay loam texture with an acidic pH. Ava developed under a deciduous forest.
- (2) Kalkaska (Michigan)--is a Spodosol, a Typic Haplorthods. Kalkaska was collected from the B (spodic) horizon. It has a reddish brown color, a sand texture with an acidic pH. Kalkaska developed under a forest environment.
- (3) Mohave, Limey (Arizona)--is an Aridisol, a Typic Haplarigid. Mohave was collected from the B (argillic) horizon. It has a reddish brown color, a clay loam texture with an alkaline pH. Mohave is a desert soil formed under an arid environment. It differs from other soils of the Mohave series in that it contains free CaCO_3 .
- (4) Molokai (Hawaii)--is an Oxisol, a Typic Torrox. Molokai was collected from the B (oxic) horizon. It has a dark red color, a clay texture with a slightly acidic pH. Molokai formed under a tropical environment.

(5) Nicholson (Kentucky)--is an Alfisol, a Typic Fragiudalfs. It was collected from the B (argillic) horizon. It has a yellowish brown color, a silty clay texture with a slightly acid pH. Nicholson was formed under a forest environment. Other characteristics of the soils are shown in table 1. The pH of the soils was determined on a saturated paste of the soil with a pH glass electrode meter. The electrical conductivity was measured with an electrical conductivity bridge on the saturated soil paste extract. Textural size was determined by the conventional pipet method. Surface area was determined by method in Heilman, Carter and Gonzales (1965). Free ion oxide was determined by the method of Kilmer (1960). The predominant clay minerals present were identified by X-ray diffraction.

Leachate

The natural municipal solid waste leachate was produced by packing representative solid waste (table 2) in a 3800 liter commercial septic tank situated above ground, saturating it with water and letting it digest for several months. Concentration levels of some constituents in the leachate appear in table 3.

Cyanide Compounds

Three cyanide solutions were used (table 4) in this study.

Table 1. Characteristics of soils

Soil	Order	pH	CEC	EC	Surface Area	Free Iron Oxide	Bulk Density	Sand	Silt	Clay	Texture Class	Predominant Clay Mineral
			meq/100g	$\mu\text{mhos/cm}$	m^2/g	%		%	%	%		
Ava (Illinois)	Alfisol	4.4	19	207	61.5	4	1.36	6	67	27	Silty Clay Loam	Vermiculite Kaolinite
Kalkaska (Michigan)	Spodosol	4.6	10	75	8.9	1.8	1.6	95	3	2	Sand	Chlorite Kaolinite
Molokai (Hawaii)	Oxisol	6.2	14	1262	67.3	23	1.33	23	25	52	Clay	Kaolinite Gibbsite
Nicholson (Kentucky)	Alfisol	6.7	37	176	120.5	5.6	1.34	3	47	49	Silty Clay	Vermiculite
Mohave _{Ca} (Arizona)	Aridisol	7.8	12	510	127.5	2.5	1.53	32	28	40	Clay Loam	Mica Montmorillonite

Table 2. Type of material in the municipal waste-type landfill used to generate leachate

Solid Waste Material	Amount Loaded in 1000 gal.	Generator
	lbs.	%
Paper (mostly news-paper)	1,400	45.7
Food waste	450	14.7
Garden waste	376	12.3
Plastic	34	10.2
Rubber	109	
Leather	60	
Textiles	109	
Metal (mostly cans)	187	6.1
Glass	177	5.8
Ash #59	127	4.1
Soil 68		
Calf manure	35	1.1

Table 3. Analysis of the natural leachate from a municipal waste-type landfill used in this study.

pH	6.7
EC ($\mu\text{mhos/cm}$)	2500
K (ppm)	240
Na (ppm)	60
Ca (ppm)	160
Mg (ppm)	33
Zn (ppm)	.3
Si (ppm)	14
Cl (ppm)	200
Fe (ppm)	90
Mn (ppm)	1.3

Table 4. Characteristics of cyanide solution

Cyanide Solution	pH of Solution	Concentration of Cyanide in Solution	Type of Ion Present
KCN in de-ionized water	10.0	ppm ~ 97	CN [⊖]
K ₃ Fe(CN) ₆ in de-ionized water	8.5	~ 98	Fe(CN) ₆ [⊖]
KCN in landfill leachate	7.0	~ 80	Unknown

- (1) KCN in de-ionized water (simple form) defined in page 1.
- (2) $K_3Fe(CN)_6$ in de-ionized water (complex form) defined on page 2.
- (3) KCN in natural landfill leachate (mixed form). The mixed form contains both the simple and complex form of cyanide. The formation of a blue precipitate occurred when KCN was added to the leachate, tests indicated the precipitate to be Prussian blue $[Fe(CN)_6]_3Fe_4$. Other compounds of cyanide were present but their actual identities are unknown.

Leaching Apparatus--(figure 1):

- (1) Three PVC columns, 5 cm. in diameter and 10 cm. in length
- (2) One Peristaltic pump with its accessories
- (3) Plastic tubing (nylon)
- (4) Collecting bottles (125 ml. volume)
- (5) 1N NaOH solution

Distillation Apparatus--(figure 2):

- (1) Heating jacket
- (2) Boiling flask
- (3) Condensation column
- (4) Transport tubing
- (5) Fisher-Milikan gas scrubber
- (6) Suction flask (vacuum)
- (7) 1N NaOH solution

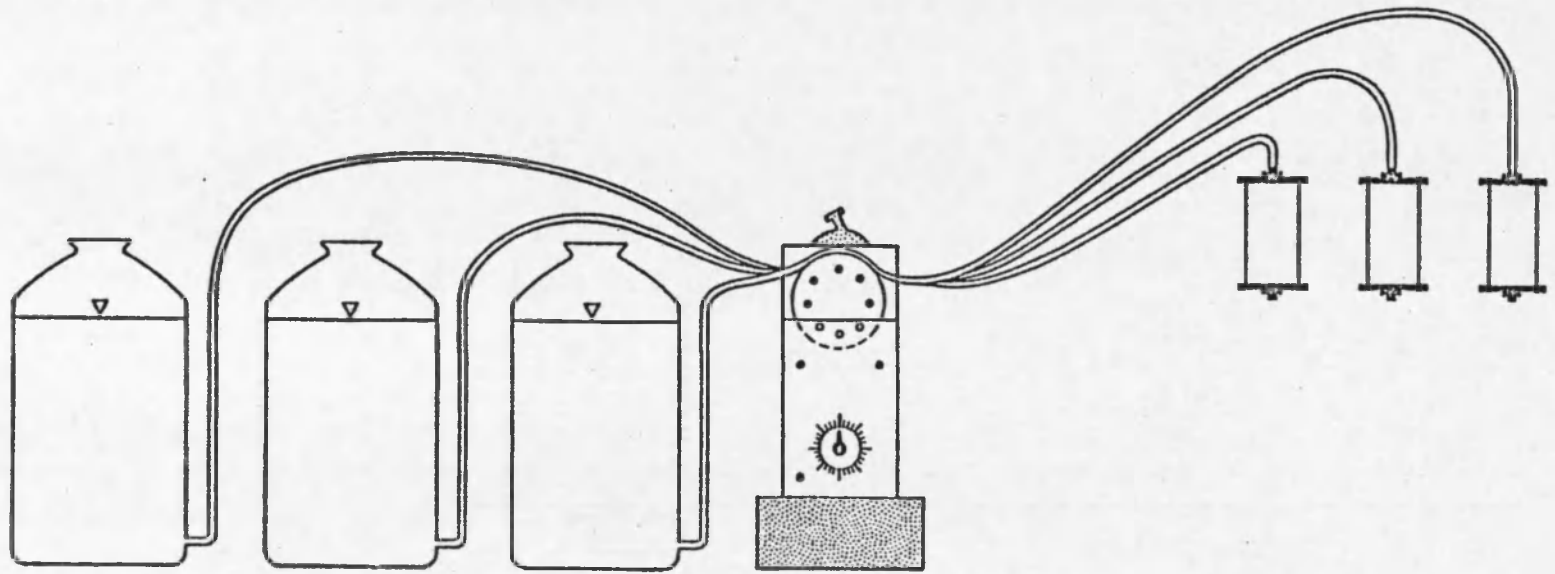


Figure 1. Leaching apparatus

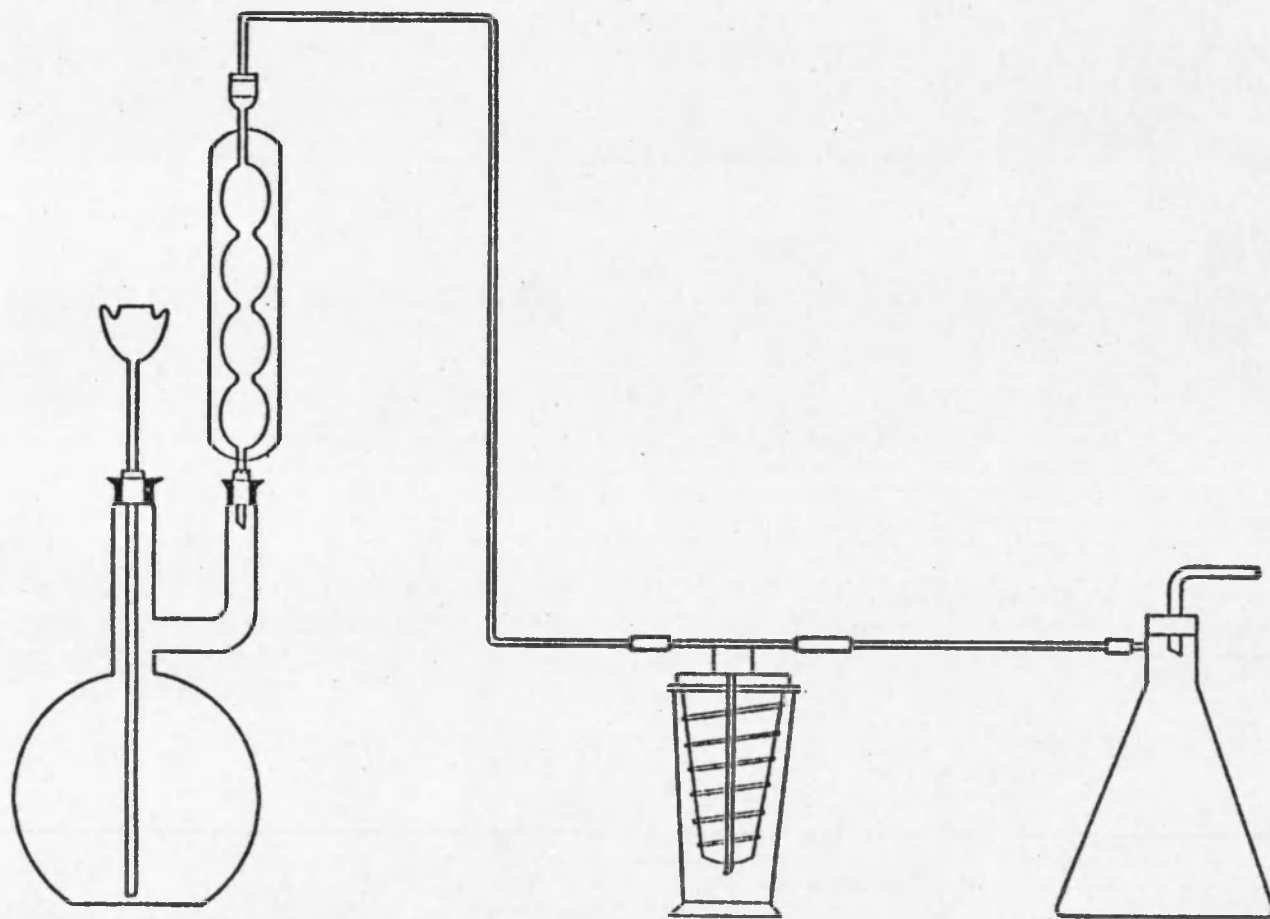


Figure 2. Distillation apparatus

Titration Apparatus:

- (1) Silver Nitrate solution
- (2) Buret (10ml)
- (3) Stirrer and magnet bar
- (4) Rhodanine indicator solution
- (5) Glass ware

METHODS

Three solutions of cyanide (table 4) were each leached through five soils (table 1) for approximately 15 days. The soils were dried in air and passed through a two millimeter sieve. Each soil was uniformly packed in a PVC column 5 cm in diameter and 10 cm in length to a specified bulk density greater than the usual natural condition to avoid column side effect and insure uniform flow throughout.

Aeration of the leachate and boiling of the water was required before adding cyanide to prevent carbon dioxide from degrading the cyanide, Miller (1906).

The columns were saturated slowly in an inverted position to insure uniform wetting and to exclude air. The cyanide solutions were pumped through the soil using a Pharmacia (Piscataway, NJ) Peritaltic pump (figure 1). The flow rate was regulated to deliver approximately one pore volume of effluent per day. The eluate was collected in 125 ml. plastic bottles containing 5 ml of 1N NaOH solution. The NaOH stabilizes any cyanide coming through the column.

The eluate was analyzed for total cyanide (CN^-) each day by the Liebig distillation method figure 2 (Taras, 1971). The distillation process proceeds in this fashion. An aliquot of the sample is placed in the boiling flask.

Its volume is brought up to about 300 ml with de-ionized water, the suction is initiated at this point. To the boiling flask, 25 ml. of concentrated sulfuric acid is added and the sample is heated to 80 C with a heating cup from one and one-half hours for simple cyanide to three hours or more for the complex cyanide. The cyanide in the sample reacts with the sulfuric acid and is transformed to HCN gas. The HCN is transported to a Fisher-Milikan gas scrubber by suction. The gas scrubber contains 50 ml of 1N NaOH plus another 50 ml of de-ionized water to bring the level of the solution to a descent height so that proper mixing can occur. Upon entering the scrubber the HCN reacts immediately with the NaOH to form NaCN plus water. After proper distillation time has elapsed the sample is collected and brought up to 250 ml volume with 1N NaOH. A 25 ml aliquot is drawn off and analyzed for CN^- by using silver nitrate as the titrant and rhodanine as the indicator.

A statistical analysis was performed using two types of variables: first the parameter characterizing the attenuation of cyanide, mass absorbed per gram of soil per milliliter of added leachate and secondly, the soil properties: % clay, pH, CEC, surface area and free ironoxide.

RESULTS

The relative mobility of the three cyanide solutions is best illustrated in Mohave_{Ca} clay loam and Kalkaska sand (figures 3 and 4). All data are plotted as pore volume versus C/C_{max} , where C/C_{max} is the ratio of effluent concentration to influent concentration, KCN and $K_3Fe(CN)_6$ in de-ionized water were both found to be very mobile in soils, with 90% or more of the cyanide being leached out on or before the fourth pour volume. KCN in landfill leachate was the least mobile of the three solutions.

The influence of soil type on the movement of the three cyanide solutions is illustrated in figures 5, 6 and 7. Figure 5 shows the amount of KCN in de-ionized water that was leached through four soils, Mohave_{Ca}, Ava, Nicholson and Molokai. The figure indicates KCN leached most rapidly in the soil having the highest pH and free $CaCO_3$ (Mohave_{Ca}). The negative charges on the clay surface of Mohave_{Ca} tends to repel the CN^- , causing it to be leached out more rapidly than in soils having a higher concentration of positive charges. The CN^- was retained most by soils having a high concentration of hydrous oxides of iron (Molokai and Nicholson). Korte, Skopp, Fuller, Niebla and Alesii (1975) found similar results while

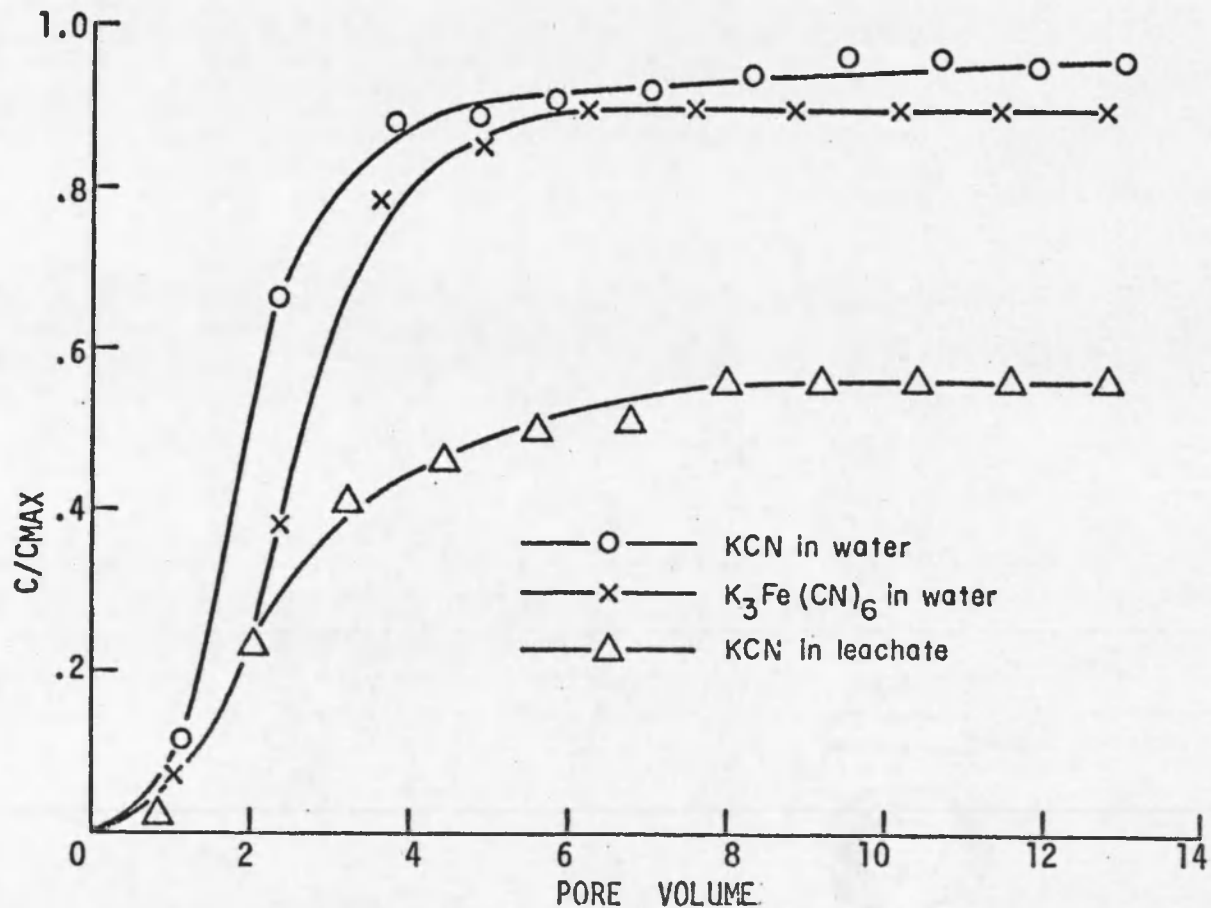


Figure 3. Relative mobility of KCN in de-ionized water, $K_3Fe(CN)_6$ in de-ionized water and KCN in leachate through Kalaska sand

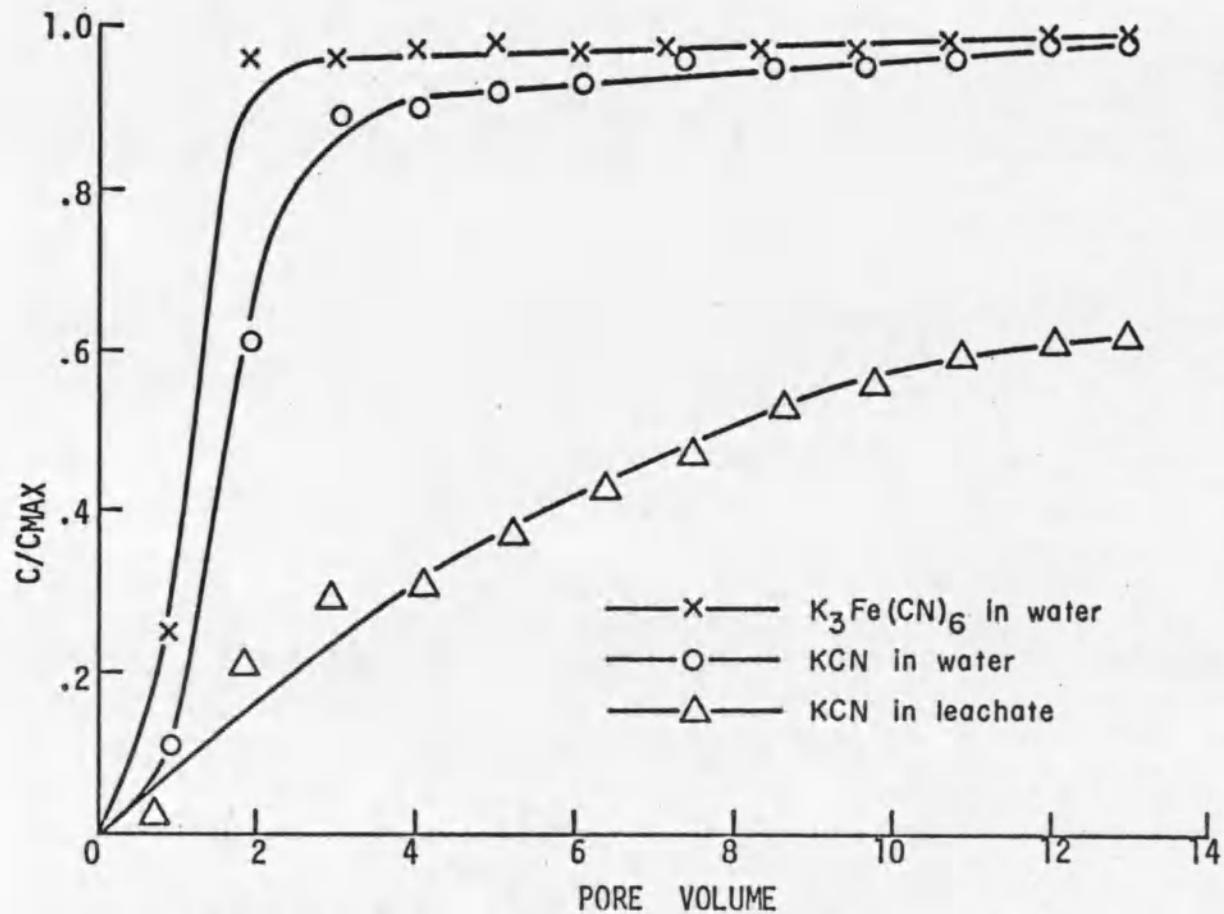


Figure 4. Relative mobility of KCN in de-ionized water, $K_3Fe(CN)_6$ in de-ionized water and in leachate through Mohave_{Ca} clay loam.

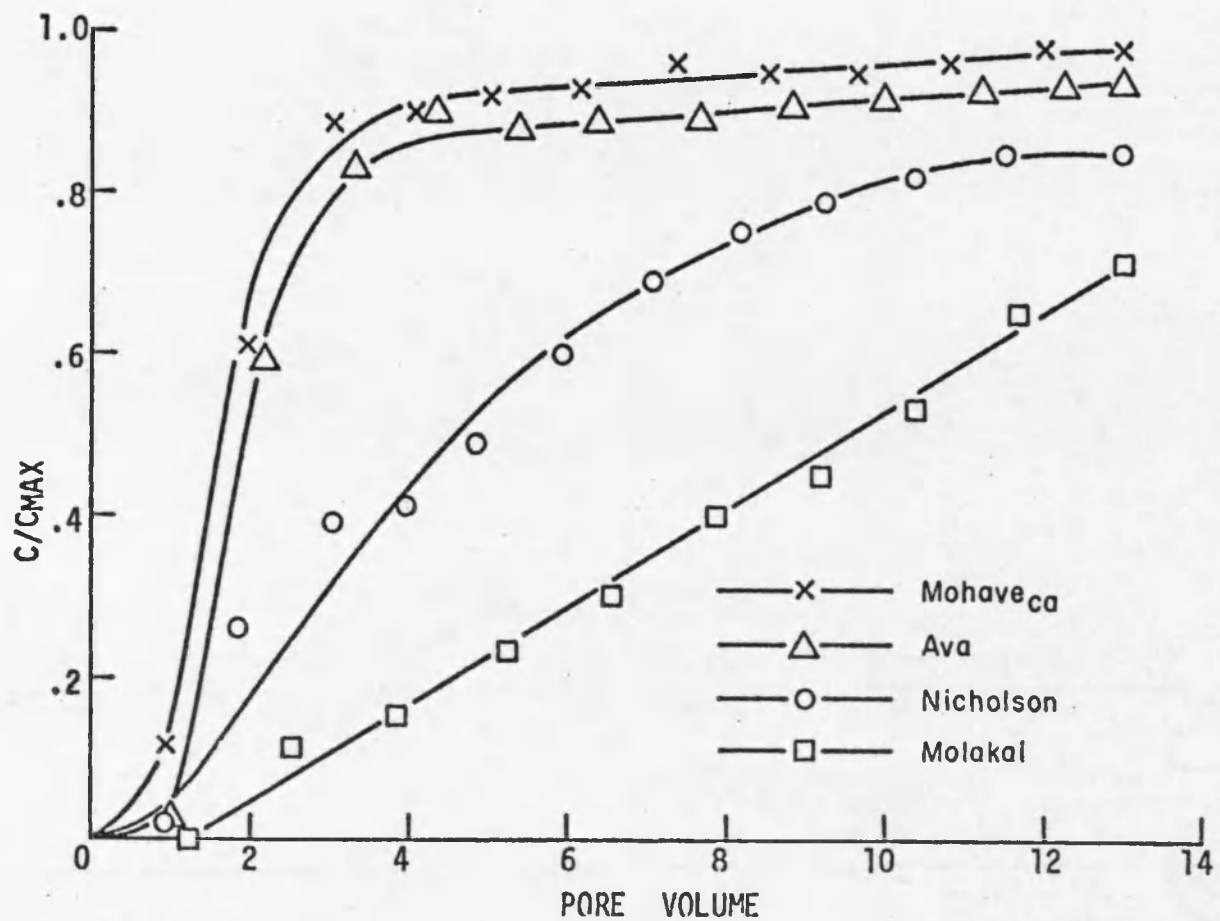


Figure 5. Relative mobility of KCN in de-ionized water through four diverse soils.

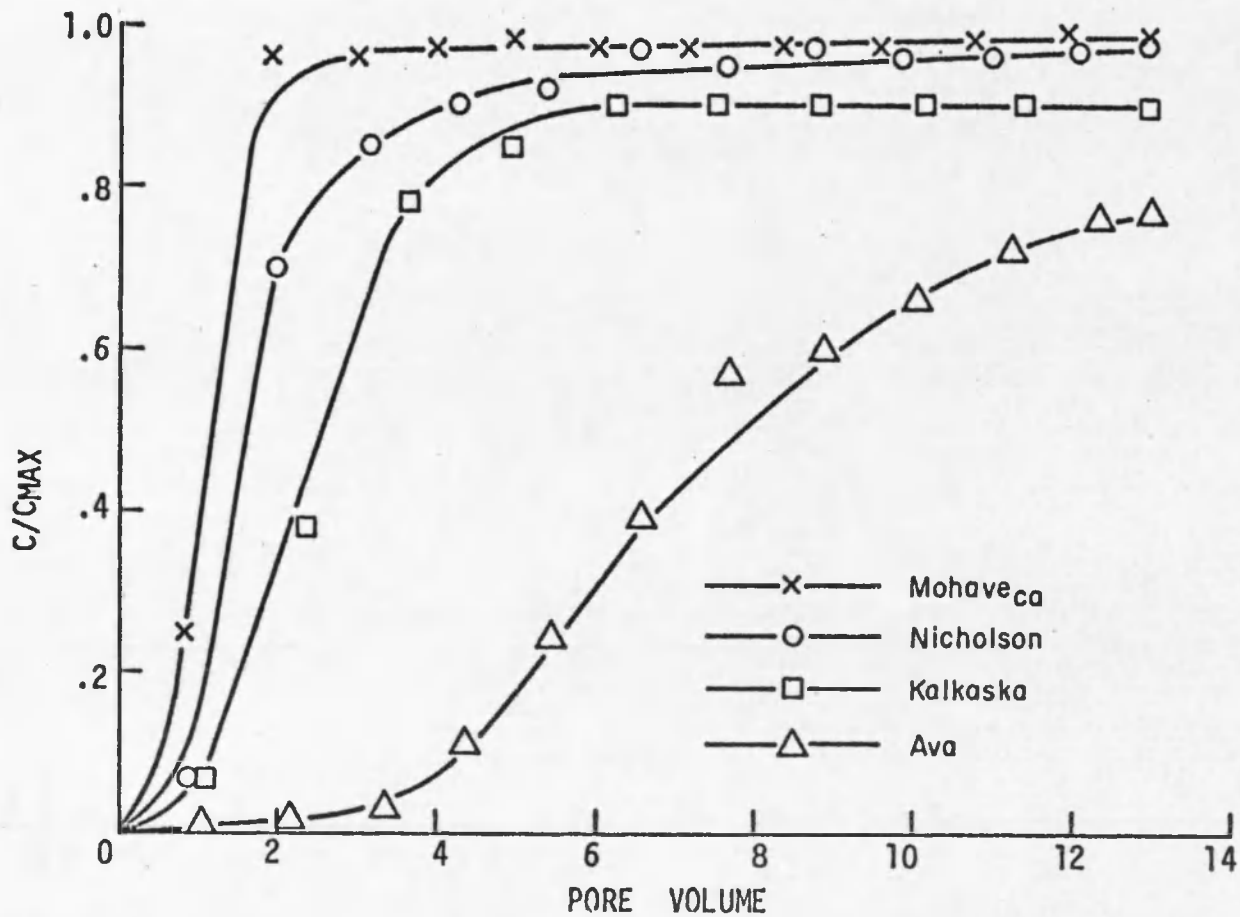


Figure 6. Relative mobility of $K_3Fe(CN)_6$ in de-ionized water through four diverse soils.

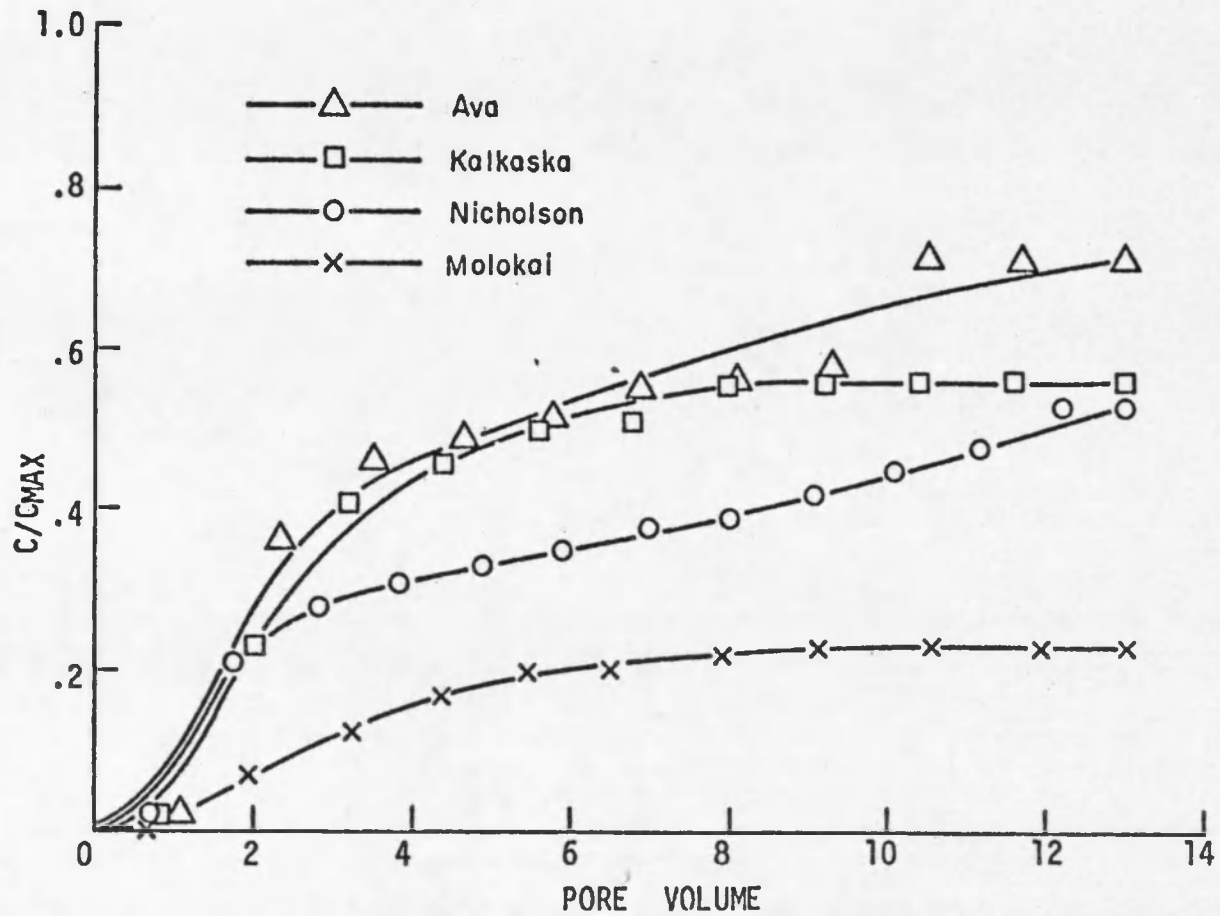


Figure 7. Relative mobility of KCN in leachate through four diverse soils.

working with the anion forms of As, Cr, Se and V. This conclusion is further supported by data from Berg and Thomas (1959). They found Cl^- which is similar to CN^- in its absorption behavior, attenuated in soils having a high percentage of kaolin clay, iron and aluminum oxide. Schofield (1939) also reported that soils high in these oxides have a high anion exchange capacity. Acid soils such as (Ava) proved, surprisingly, to be a poor attenuator of CN^- . Texture seemed to have some influence on the attenuation of KCN but its effect was masked by the presence of iron oxide and CaCO_3 .

Figure 6 illustrates the movement of $\text{K}_3\text{Fe}(\text{CN})_6$ in de-ionized water through four soils Mohave_{Ca}, Ava, Nicholson and Kalkaska. The Ferricyanide ion also migrated most rapidly through soils having a high pH and the presence of free CaCO_3 (Mohave_{Ca}) for the same reason given for KCN in water. Ferricyanide ion moved slowest in soils having a low pH with kaolin type clay being the dominant clay minerals (Ava and Kalkaska). A low pH would indicate the clay surface to have a high percentage of positive exchange site, which would attract the $\text{Fe}(\text{CN})_6^{3-}$ ion and retain it. Kamprath, Nelson and Fitts (1956) found good retention of SO_4^{2-} by an acidic soil high in oxides and kaolin, whereas, the 3-layer minerals appeared to have poor retention of SO_4^{2-} . Although iron-oxide had an affinity for $\text{Fe}(\text{CN})_6^{3-}$ its presence was not as effective as soil pH in governing

the movement of this form of cyanide. Texture seems to play a more important role in the retention of $\text{Fe}(\text{CN})_6^{3-}$ when accompanied by low soil pH. The higher clay content soil (Ava) retained more of the Fe than the sandier soil of similar pH (Kalkaska).

Figure 7 portrays KCN in landfill leachate migrating through four soils (Ava, Kalkaska, Nicholson and Molokai). KCN in leachate behaved very similarly to KCN in water. This solution moved most rapidly through soils with low pH (Ava and Kalkaska). Cyanide was retained the most by soils having a high concentration of iron-oxide (Molokai and Nicholson). Of the three solutions, KCN in leachate was found to be attenuated the best. This can be partly explained by the precipitation of prussian blue when KCN was added to the leachate (Robine, Lenglen and LeClere, 1906). The blue precipitate was found permeating the top 4 cm of the soil column. The accumulation indicates prussian blue to be quite immobile in soils. The form of cyanide that eluted through the soil column probably was the CN^- that had not reacted with the iron in solution to form prussian blue and other cyanide compounds.

Very little cyanide if any was lost to biological degradation in the soil columns this can be supported by the fact that the anaerobic state of the soil columns inhibited any microbial degradation of cyanide. Microorganisms responsible for degrading cyanide under anaerobic

conditions are very sensitive to high cyanide concentration. Coburn (1949) found 2 ppm in the wastestream to be the limit for effective anaerobic degradation of cyanide. This concentration is much less than that which was passed through the soil columns.

DISCUSSION

Shown in Table 5 are the correlation coefficients of five soil parameters: % clay, pH, CEC, surface area and % free iron oxide, with the mass of cyanide compound absorbed per gram of soil, per milliliter of effluent added. KCN in water correlated very high with % iron oxide. It also gave a fair correlation to the % clay. There was no correlation to either CEC, pH or surface area. The absence of a correlation to pH seems to again support the conclusion that low pH has little or no effect in attenuating this form of cyanide.

KCN in leachate gave similar correlation to KCN in water. It correlated very high with % iron oxide. It also gave a fair correlation to the % clay. These similar correlations between KCN in water and KCN in leachate for both % iron oxide and % clay while giving no correlation to either soil pH, CEC or surface area would lead one to believe the mechanism controlling the attenuation of these two solutions are very similar.

$K_3Fe(CN)_6$ in water gave a good negative correlation to pH, indicating that as the pH drops, attenuation increases. It also gave a fair correlation to % iron oxide. There was no correlation to either % clay, CEC or surface area.

Table 5. Correlation coefficients of mass absorbed per gram of soil with soil properties

Cyanide Compounds	Clay	pH.	CEC	Surface Area	Fe ₂ O ₃
KCN In Water	.68	.20	.24	.10	.94
KCN In Leachate	.69	.42	.08	.19	.91
K ₃ Fe(CN) ₆ In Water	.12	-.77	-.20	-.40	.64

RECOMMENDATIONS

The evidence gathered from this experiment showed cyanide to be quite mobile in a variety of soils. Due to its mobile nature and its potentially high toxicity to the biological system it is imperative that cyanide be disposed of in a safe manner in the environment. A number of recommendations have been arrived at to prevent contamination of cyanide to the ground water system;

(1) The maintenance of the cyanide plant should be kept in tip-top condition so as to reduce spills and leakage. This will greatly reduce the level of cyanide in the waste stream.

(2) A good treatment system should be established. Preferably one that would remove the majority of cyanide in its toxic state. A variety of treatment systems are listed on page 4. Of these complexation and activated sludge seems to lend themselves to the best use. Complexation simply changes the state of the cyanide to a very stable non-toxic compound. Activated sludge is extremely effective in detoxifying CN^- by using microorganisms to break down CN^- into ammonium and carbon dioxide.

(3) Any cyanide remaining in the waste stream after treatment can be filtered effectively enough by the soil. Here again certain soils are better for this task than others.

Results from this experiment reveal the preferred soil should be deep, have a clay texture high in iron oxide and kaolinitic type clay with a low pH.

(4) The soil should be inoculated if possible by cyanide degrading organisms, this will help break down the cyanide trapped by the soil.

CONCLUSION

Cyanide as $\text{Fe}(\text{CN})_6^{3-}$ and CN^- in water were both found to be very mobile in soils. Cyanide as KCN in natural landfill leachate was found to be less mobile. Soil properties such as low pH, percentage free-iron oxide and kaolin, chloride and gibbsite type clay (high positive charges), tended to increase attenuation of cyanide in the three forms. High pH, presence of free CaCO_3 (high negative charge), low clay content and montmorillonite clay tended to increase the mobility of the cyanide forms. Evidence from this investigation shows cyanide could possibly contaminate the ground water if proper methods of handling the cyanide are not used.

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