DECONTAMINATION

OF RADIOACTIVELY CONTAMINATED WATER

BY SLURRYING WITH YAZOO AND ZILPHA CLAYS

By

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and

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INTRODUCTION

The level of radioactivity of most radionuclides discharged into watercourses must be reduced before the water can be consumed. Various attempts have been made to determine the most economical method for this activity reduction. These attempts have included slurrying with powdered metal and clays, chemical coagulation, and ion exchange on synthetic resins.

One of the most practical methods of activity reduction is that of slurrying with clay. Good results have been obtained by this method and by the addition of clay in conventional water treatment processes such as chemical coagulation and lime-soda ash softening.

A majority of the clays under investigation or in use today require pretreatment before they are suitable for use. The clays chosen for this experiment were Yazoo and Zilpha clays. These clays are abundant throughout most of the state of Mississippi and may be easily mined near the surface. If these untreated clays have decontamination properties comparable with those of other treated clays, the cost per gallon of removing radioactive materials could be materially reduced.

As the literature concerning clay slurries is reviewed, it may be observed that each particular clay must be slurried with each radioisotope

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before any conclusion concerning the overall effectiveness of the clay may be inferred. This study was limited to three radioisotopes: Co^{60}, Sr^{90}, and Cs^{137}. These radionuclides were selected because of their presence in most nuclear waste discharges and the latter two are abundant in fallout from nuclear detonations.

LITERATURE REVIEW

The processes by which most radioisotopes are removed from solution have been described by Lacy (1), Spitsyn and Gromov (2,3), Tamura and Struxness (4), and Straub, et al. (5) as ion exchange and surface adsorption. Each clay has a limited number of ion exchange and adsorption sites. Since radioisotopes behave the same chemically as their stable counterparts, any stable isotopes present in the waste being treated will compete with the radioisotopes present for the available exchange sites. This competition will reduce the effectiveness of removal by adsorption or ion exchange of any radioisotope by any clay.

Co^{60} is considered to be primarily an external radiation hazard because it is principally a gamma emitter. Co^{60} may also be considered an internal hazard, because it also emits beta particles and is concentrated by the lower large intestine.

Cobalt is generally easily removed from wastes in which the pH is greater than 6 and the waste is free of complexing agents. This statement is supported by the results obtained by Straub and Kreiger (6). They observed that optimum removal occured at a pH of 6 or greater, with little additional removal being obtained as the pH was raised. Perhaps an even more noteworthy finding of these two authors was the effect that the chemistry of the solution had upon the removal efficiency. They noted that when acetic, citric, or phthalic acids were present, lower removals were obtained than with other substrates of comparable pH values.

The theory that cobalt forms anionic complexes with the salts of certain acids was further verified by Hawkins (7). He reports that of the wastes discharged to the soil by the Naval Reactor Facility, Co^{60} constituted 60 percent of the activity which seeped through the soil into the water of the monitoring well. Since these wastes contained ammonium citrate, Hawkins determined that the cobalt was migrating as an anionic citrate complex and could be removed by an anion exchange resin.

The results obtained by Hawkins from laboratory clay column studies demonstrated that clay can effectively remove uncomplexed Co^{60} from solution. The results of the operation of the process waste treatment plant at Oak Ridge National Laboratory reported by Cowser and Tamura (8) also indicated the effectiveness of clay for removing Co^{60}.

Sr^{90} is considered as one of the greatest internal hazards since it is a beta emitter and is concentrated in the skeleton of the body. The daughter of Sr^{90}, Y^{90}, is also a beta emitter. The level of activity of Sr^{90} would be almost constant for a human life time, resulting in extensive damage to bone and bone marrow tissue.

Since the chemical behavior of strontium is similar to that of calcium, it may be expected that optimum removals of Sr^{90} would occur at high pH values. The results presented by Culp (9) demonstrate that high pH is necessary to precipitate Sr^{90} either by lime-soda ash softening or by phosphate coagulation. The latter method proved effective as 97.8 percent of the strontium in the test water was removed by a dose of 240 mg/l phosphate. A disadvantage of this process is that accurate control of pH and of the ratio of phosphate to lime dosage is required. McCauley and Eliassen (10), however, have reported that lime-soda ash softening results in a removal of about 50 percent for Sr^{90} with the softening plant operating at maximum efficiency.

Clay was added to radioactive water as turbidity and coagulated by Straub (11) and Morton and Straub (12) using common chemical coagulants. The results of these jar tests showed that with clay dosages of 5000 mg/l, removals of up to 52 percent for strontium were obtained. This was nearly 9 times the maximum removal of 6 percent for chemical coagulants alone.

The presence of $CO_3^=$, $SO_4^=$, and $C_2O_4^=$ anions in solution was found to be detrimental to the adsorption of Sr^{90} by clay by Spitsyn and Gromov (2); however, McHenry (13) states that Sr^{90} removal by clay was greatly improved by the addition of $C_2O_4^=$ ions.

McHenry also states that Sr^{90} removal was optimum at a pH of approximately 10. The results obtained by Cheng (14) show that optimum Sr^{90} removal by Chinese clays is obtained at pH values greater than 8, with 2 clays having a maximum removal at pH 10. Although the maximum pH was 14 for these 5 clays, little additional removal was obtained beyond pH 10 or 11. However, the results of Takizawa (15) show that for sodium saturated clays, the optimum removal of Sr^{90} occurs in the pH range of 6.4 to 9.2. Another demonstration of the importance of pH for optimum strontium removal has been provided by Tamura (16), who states that for an aluminum saturated montmorillonite a pH of 10 provided maximum strontium removal.

Since Cs^{137} is chemically similar to sodium, ingestion of this radioisotope will result in its deposition in the soft tissues all over the body. The distribution of Cs^{137} within the body and the emission of beta particles by this radioisotope create an internal hazard. The most important soft tissues of the body are the reproductive organs which are particularly sensitive to damage.

The normal water treatment processes, such as chemical coagulation and lime-soda ash softening, should not be effective for removing highly soluble Cs^{137} salts. This assumption was verified by the results of Culp (9). When the process of chemical coagulation was modified by adding 5000 mg/l of clay as turbidity, Straub (11) and Morton and Straub (12) report that 98 percent of the initial Cs^{137} concentration was removed.

The effects of pH on Cs^{137} adsorption by clays are described by Cheng and Hamaguchi (17). They state that the range for optimum removal of Cs^{137} by most clays is from pH 3 to pH 8. Cheng further states that below pH 3 the large number of hydrogen ions present compete with the Cs^{137} ions for adsorption by the clay, whereas the excess of cations present at high pH values also offer considerable competition to the Cs^{137} ions.

Another important parameter in the process of adsorption of Cs^{137} by clays is the structure or mineralogy of the clay involved. Oi and Ohashi (18) stated that their results indicated that at low Cs^{137} concentrations (less than 10^{-6} *M*) a preferential structural adsorption occurs, creating an increase in Cs^{137} removal. An explanation of this structural adsorption is described by Tamura and Jacobs (19, 20, 21), Schulz, *et al*. (22), and Jacobs (23).

The property of ion exchange of a clay results from a positive charge deficiency within the lattice of the clay mineral. The structural causes of these charge deficiencies have been fully described by Grim (24). Since the clay structure generally prohibits the penetration of cations into the crystal lattice, Lacy (25) states that the charge deficiency is satisfied by the formation of an electric double layer at the interface between the particle and the solution.

The process of selective adsorption of one ion from an electrolyte in which the clay particle is suspended is reported by Weiser (26) to primarily account for the formation of the double layer. The adsorbed ion is retained in the double layer primarily by Coulombic forces, with van der Waals forces contributing very little to the retention.

The relative replacing power or exchange ability of different cations has been reported by Kelley (27) to be a function of the structure and chemistry of the clay, the nature of the exchangeable cations, and the chemistry of the solution in which the exchange occurs.

The replacing power of cations in solution has been reported by Straub (28) and Weiser (26) to be a function of the valence of the exchanging ion. At low concentrations in water, the affinity of cations for the clay follows the Schulze-Hardy rule. This rule states that a quadravalent ion will be adsorbed more easily than a tetravalent ion, a tetravalent ion more easily than a divalent ion, and divalent ion more easily than a monovalent ion. For ions of the same valence, Lacy (1) and Straub (28) state that the affinity increases according to the lyotropic series. That is, the order of affinity for the alkaline metals is

Cs > Rb > K > Na > Li ;

for the alkaline earth cations, it is

Ba > Sr > Ca > Mg ;

for the transition metals (divalent state),

Zn > Cu > Ni > Co > Fe ;

and for the trivalent cations,

La > Ce > Pr > Nd > Sa > Eu > Y > Sc > Al.

Straub further states that molecular adsorption takes place between clay and weak electrolytes in solution. Lacy (1) states that the mechanism of this surface adsorption is obscure, and suggests that the clay enters into a loose combination with the material adsorbed by means of inactive valences of atoms in the surface layers.

EQUIPMENT AND PROCEDURE

The gamma detection equipment consisted of a well type Thallium activated NaI scintillation crystal and a photomultiplier tube connected to a six decade scalar. The beta detection equipment consisted of a thin window (1.4 mg/sq. cm) Geiger-Muller tube connected to another six decade scalar. The input voltage of both scalars was stabilized by a constant voltage transformer.

The slurry equipment consisted of blade type laboratory stirrer. A vacuum filter assembly using cellulose nitrate filters was used to separate the slurried clay from the effluent.

The clays were initially ball milled for 24 hours to insure a high percentage of colloidal sized particles. The clays were then oven dried at 103 to 105 degrees Centigrade to enable measurement of the clay on a dry weight basis. An analysis of each clay is shown in Table 1.

An aliquot of radioisotope was added such that initial concentrations of 3000 cpm/ml of Co^{60} and Cs^{137} and 8000 cpm/ml of Sr^{90} were obtained for the "spiked" solution. These concentrations were approximately 2×10^{-2} μc/ml for the "spiked" solution of each radioisotope. Tap water was used as the substrate for each of the radioisotopes. A chemical analysis of the water is shown in Table 2.

For Sr^{90} removal, additional samples were prepared using a substrate consisting of tap water with enough 1.0 N NaOH added to increase the pH to approximately 10. Culp (19) and Straub (11) have shown that phosphate coagulation will precipitate strontium; therefore, the use of phosphate buffers to increase the pH may result in erroneously high removals being attributed to the clay. For the same reason, it was thought to be improper to use $Ca(OH)_2$ for raising the pH. Since sodium ions have not been shown to be detrimental to strontium removal, it was decided that 1.0 N NaOH would be used to raise the pH for the strontium samples.

As Tamura and Jacobs (20) stated that heat treatment of montmorillonite clays improved their ability to adsorb Cs^{137}, each of the two clays was heat treated prior to its use in slurries. This pretreatment consisted of heating the clays to 600 to 650 degrees Centigrade for six days.

After a 90 minute slurrying period, the effluent was separated from the clay by vacuum filtration. Samples of the effluents were then taken for purposes of counting. Sufficient time was allowed for Sr^{90} and Cs^{137} to reach radioactive equilibrium with their daughters, Y^{90} and Ba^{137m} respectively, before the final count was made.

RESULTS AND DISCUSSION

For the tap water substrate used, Yazoo and Zilpha clays provided very good removal of Co^{60} as may be seen in Figure 1. A percentage removal of 90 percent was obtained using 500 mg/l of Yazoo or 700 mg/l of Zilpha. It may be noticed in Figure 1 that any clay dosage higher than 1500 mg/l results in very little additional removal.

A decontamination factor of approximately 14 was required for reduction of the concentration of the radioisotopes used in this investigation (2×10^{-2} μc/ml) to a concentration suitable for exposure to the general public. This decontamination factor was obtained by a dosage of approximately 1100 mg/l of Yazoo clay as may be seen in Figure 2. In this same figure it may be noted that a dosage of approximately 2800 mg/l of Zilpha clay produced the same decontamination factor of approximately 27 as did a dosage of 1500 mg/l. Thus, it may be seen that for this particular radioisotope, Yazoo clay was the superior clay.

Zilpha clay, as shown in Figures 3, 4, 5, and 6, was far superior to Yazoo clay as an adsorber of Sr^{90}. As shown in Figure 4, a dosage of 4000 mg/l of Zilpha clay resulted in a percent removal of approximately 97 percent for tap water substrate (pH 8), whereas the same dosage of Yazoo clay had a result of approximately 57 percent removal. Lacy (1) reports that he obtained 95 percent removal for a dosage of 4000 mg/l of a clay with an exchange capacity of 29 me/100g using a comparable substrate. However, Lacy does not specify that the effluent samples were allowed sufficient time for radioactive equilibrium to be reestablished.

The much higher percentage removals obtained by Zilpha clay compared to Yazoo clay may possibly be attributed to the lack of $CaCO_3$ in Zilpha clay. The $CaCO_3$ present in Yazoo clay could possibly have been ionized in the tap water substrate. Spitsyn and Gromov (2) have stated that the presence of $CO_3^=$ ions is detrimental to strontium removal. As shown in Figure 4, the difference between Yazoo and Zilpha clay was much less at pH 10. Calcium carbonate is only very slightly soluble at this higher pH.

The effect of pH at dosages higher than 3000 mg/l, as shown in Figure 3, is not as marked for Zilpha clay as for Yazoo clay. This also may be due to the absence of $CaCO_3$ in Zilpha clay.

The point marked Yazoo at pH 2.7 appearing in Figures 3 and 5 was obtained when it was necessary to use 2.25 ml of Sr^{90} in 1.0 $\underline{N}$ HCl solution to approach the desired activity concentration. This lower pH is detrimental to Sr^{90} removal, as shown in these figures.

The decontamination factor for Sr^{90} required to lower the initial concentration of this radioisotope to a concentration which is considered safe for public exposure was approximately 20,000. As shown in Figures 4 and 5, the highest decontamination factor for Sr^{90} obtained in this investigation was approximately 54 for about 6000 mg/l of Zilpha at pH 10. Although the decontamination factor obtained was insignificant compared to the factor required, a series of batch slurries, such as proposed by Vesely, et al. (31), using this dosage and pH may produce the required decontamination.

Both Yazoo and Zilpha clays provided very good removal of Cs^{137} as shown in Figure 7. As shown in this figure, both clays provided approximately 90 percent removal of Cs^{137} at a dosage of only 250 mg/l. Cowser and Tamura (8) report that 86 percent removal of Cs^{137} is obtained at the process waste treatment plant at Oak Ridge National Laboratory. This treatment involves the addition of lime and soda ash based on the stoichiometric requirements of the waste with an addition of 200 mg/l excess soda ash and 200 mg/l of Grundite clay (65-75 percent illite, 10-20 percent kaolinite, and 5 to 15 percent quartz).

A dosage of 1000 mg/l of either clay may appear to be the optimum dosage as shown in Figure 7, as little increases in percentage removal is obtained for increased clay dosage. However, the initial concentration of Cs^{137} required a decontamination factor of approximately 100. As may be seen in Figure 8, the highest dosage of each clay used approaches this required decontamination factor. A dosage of Zilpha clay of approximately 2600 mg/l attains a decontamination factor of 85; whereas a dosage of approximately 2800 mg/l of Yazoo attains a factor of approximately 91.

As stated in the literature review, Oi and Ohashi (18) have shown that a preferential structural adsorption for cesium occurs at cesium concentrations less than 10^{-6} M. The concentration of cesium in the water used in this investigation was approximately 8×10^{-10} M. Therefore, a preferential adsorption of cesium could have accounted for the high removals of Cs^{137} obtained in this investigation.

The results of pretreating each clay by heating to 600-650 degrees Centigrade for 6 days are shown in Figures 7 and 8. The slurry time for these pretreated clays was 90 minutes, the same as for the normal clays. The percentage removals obtained for the heat treated clays were generally the same as or lower than the percentage removals obtained for the same dosage of normal clays. One advantage of the heat treated clays was their marked settling ability. As soon as the slurry mixers were shut off the majority of the clay in the slurry settled out of suspension.

Figures 9 and 10 show the percent removal of each radioisotope obtained with each type clay and the decontamination factor for each type clay is shown in Figures 11 and 12.

CONCLUSIONS

A dosage of approximately 1100 mg/l of Yazoo clay effectively decontaminated the Co^{60} contained in the substrate used in this investigation; whereas the desired decontamination factor was not obtained with any dosage of Zilpha clay. Yazoo clay was ineffective for decontaminating Sr^{90} in a tap water substrate, perhaps because of its $CaCO_3$ content. Zilpha clay was effective for removing small amounts of Sr^{90} from the substrates used. Yazoo and Zilpha clays were approximately equally effective for removing micro quantities of Cs^{137} from the substrate. Pretreatment of Yazoo and Zilpha clays by excessive heating for a prolonged period appears to reduce the effectiveness of these clays for removing Cs^{137} from the substrate. However, the time required for these pretreated clays to settle out of suspension is much less than the time required for normal clays. The decontamination factors and percent removals obtained for the clay dosages used in this investigation, except Yazoo clay for Sr^{90}, exceeded the values obtained from comparable experiments involving other clays reported in the literature. These encouraging results indicate that Yazoo and Zilpha clays may prove to be more economically feasible than the clays now used commercially as adsorbers of radioisotopes. Therefore, the results of this investigation appear to justify a further, much more detailed study of the capabilities and limitations of these clays as adsorbers of radioisotopes.

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Table 1. Chemical and Mineralogical Properties of Yazoo and Zilpha Clays.

Property	Zilpha Clay	Yazoo Clay
Total cation exchange capacity	52.1 me/100g	51.5 me/100g
Exchangeable Ca^{++}	18.1 me/100g	38.6 me/100g
Exchangeable Mg^{++}	6.0 me/100g	11.0 me/100g
Exchangeable K^+	2.8 me/100g	2.3 me/100g
Exchangeable Na^+	0.2 me/100g	3.2 me/100g
Percent $CaCO_3$	--	17.3
Percent $SO_4^=$	0.50	0.92
pH	3.8	7.5
Percent montmorillonite	75-85	70
Percent Mica & Quartz	15	10
Percent Kaolinite	--	15-20

This table was adapted from an analysis by Glenn (29).

Table 2. Chemical Analysis of State College, Mississippi Tap Water.

Chemical Constituent	Concentration (mg/l)
Dissolved solids	114.40
Alkalinity (as $CaCO_3$)	84.04
Total hardness (as $CaCO_3$)	26.00
Calcium (Ca^{++})	8.20
Magnesium (Mg^{++})	1.69
Sodium (Na^{+})	29.30
Potassium (K^{+})	3.55
Silicon Dioxide (SiO_2)	11.34
Iron (Fe)	0.13
pH	7.80

This analysis was adapted from Analysis No. 359,511, State Chemical Laboratory (30).

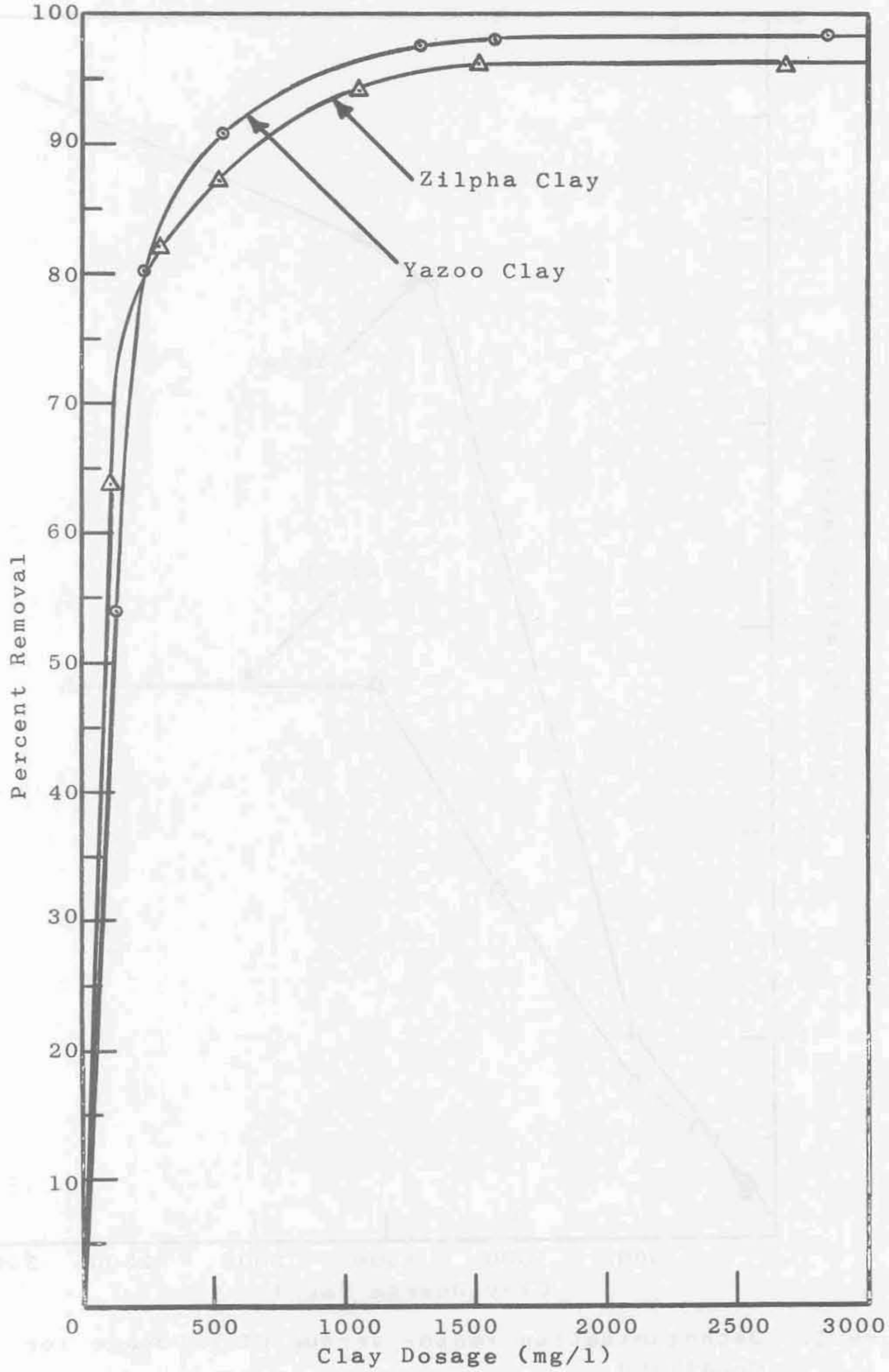

Figure 1. Percent Removal Versus Clay Dosage for Cobalt-60.

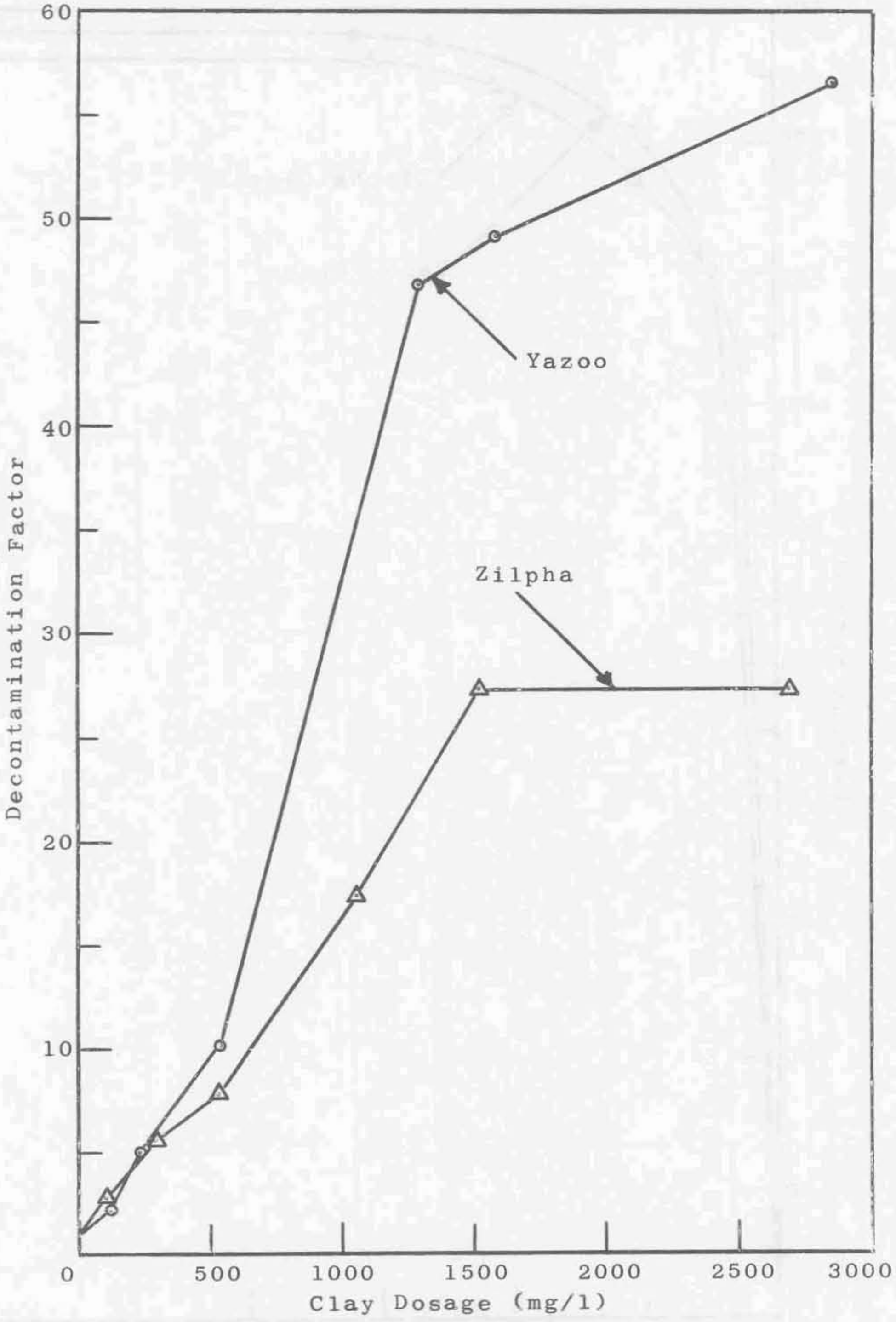

Figure 2. Decontamination Factor Versus Clay Dosage for Cobalt-60.

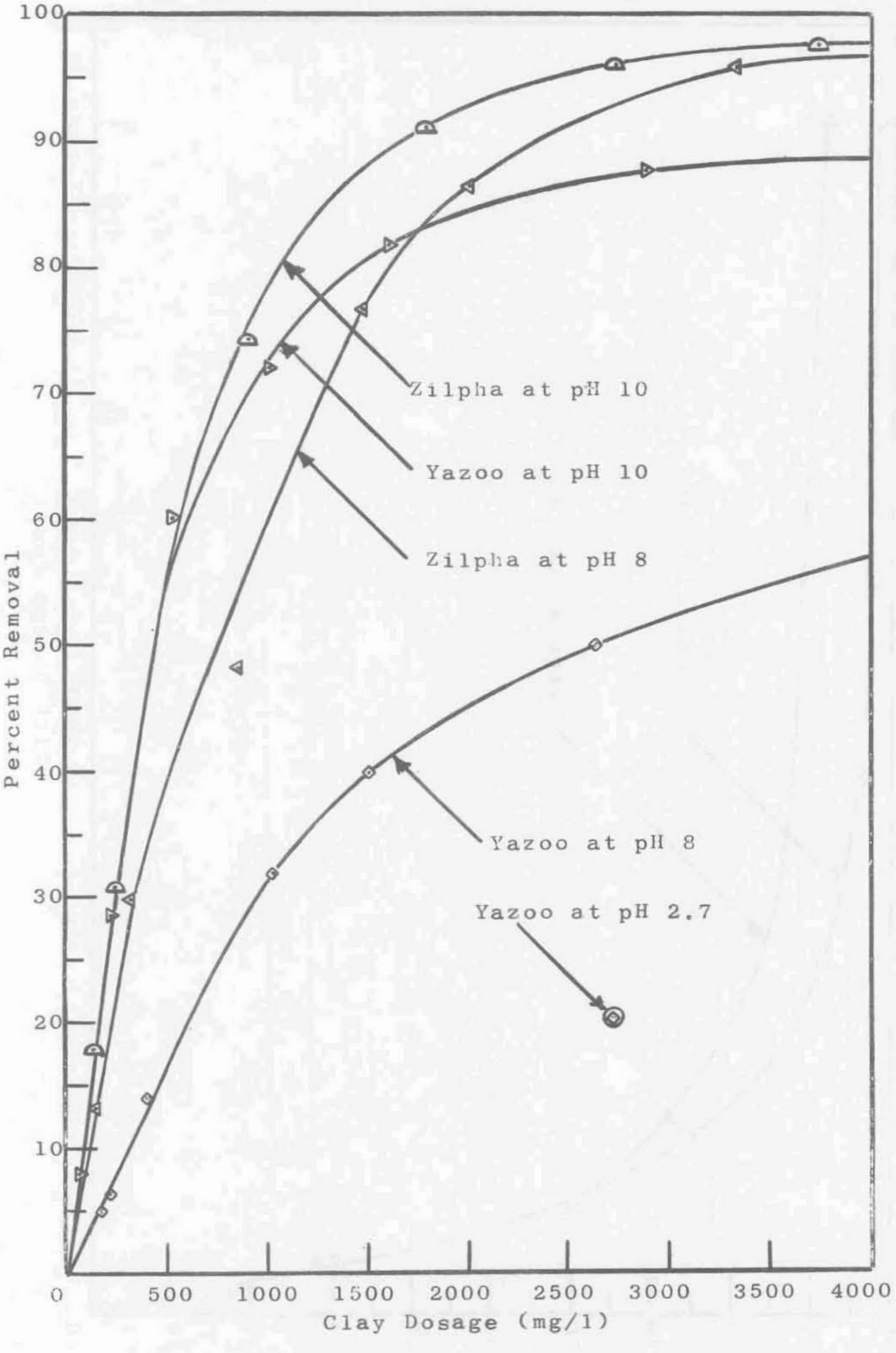

Figure 3. Percent Removal Versus Clay Dosage for Strontium-90.

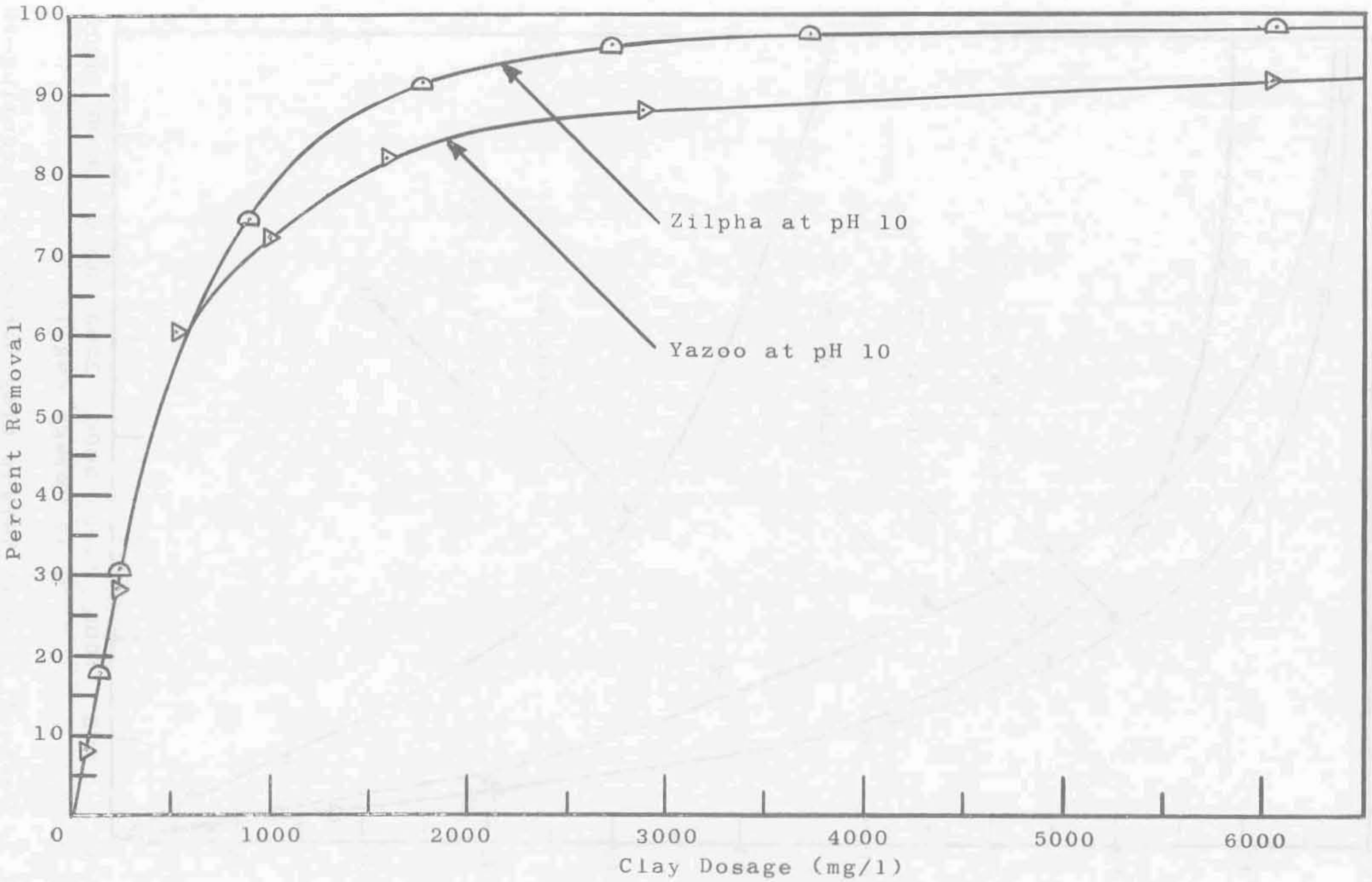

Figure 4. Percent Removal Versus Clay Dosage for Strontium-90.

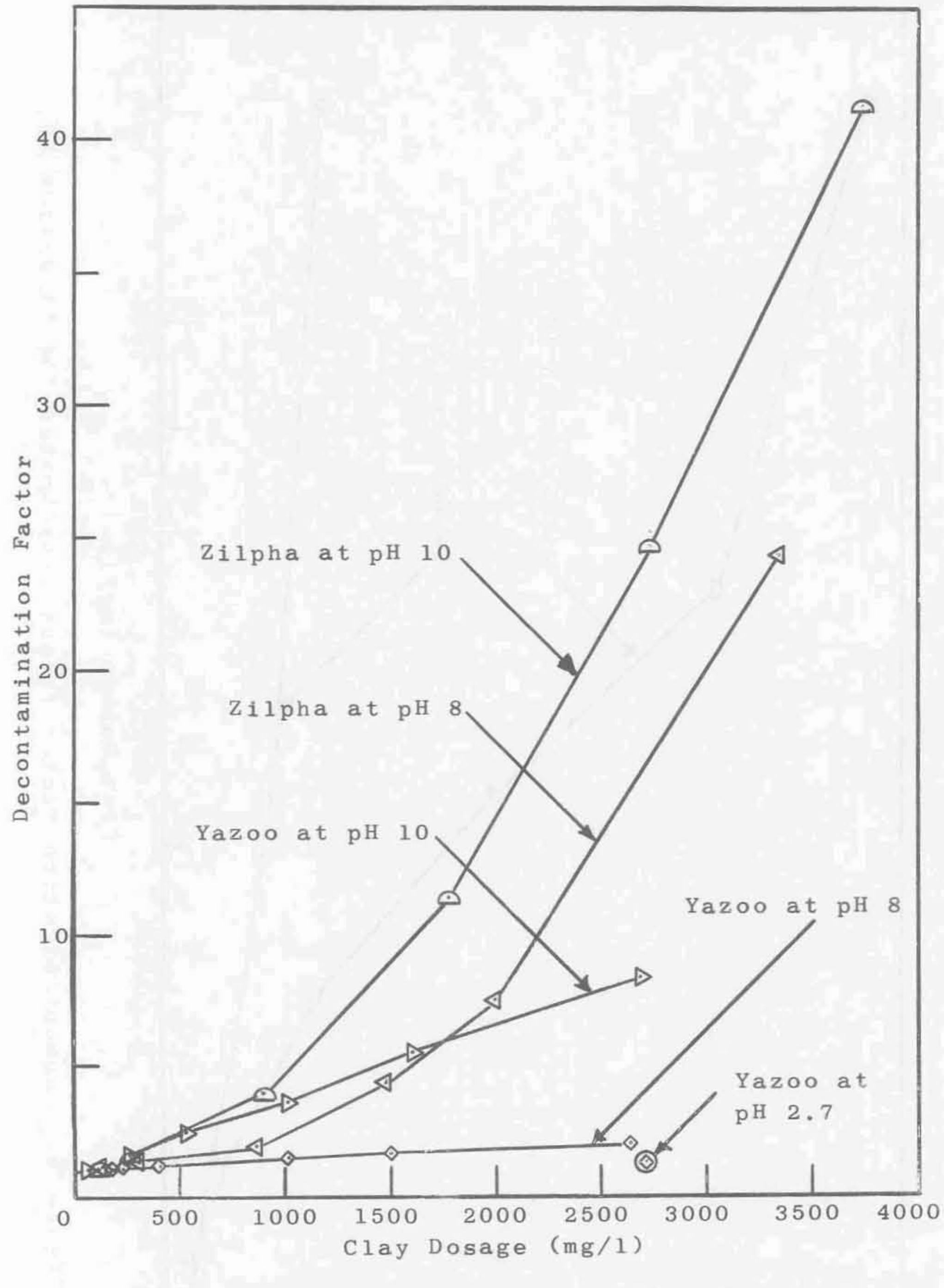

Figure 5. Decontamination Factor Versus Clay Dosage for Strontium-90.

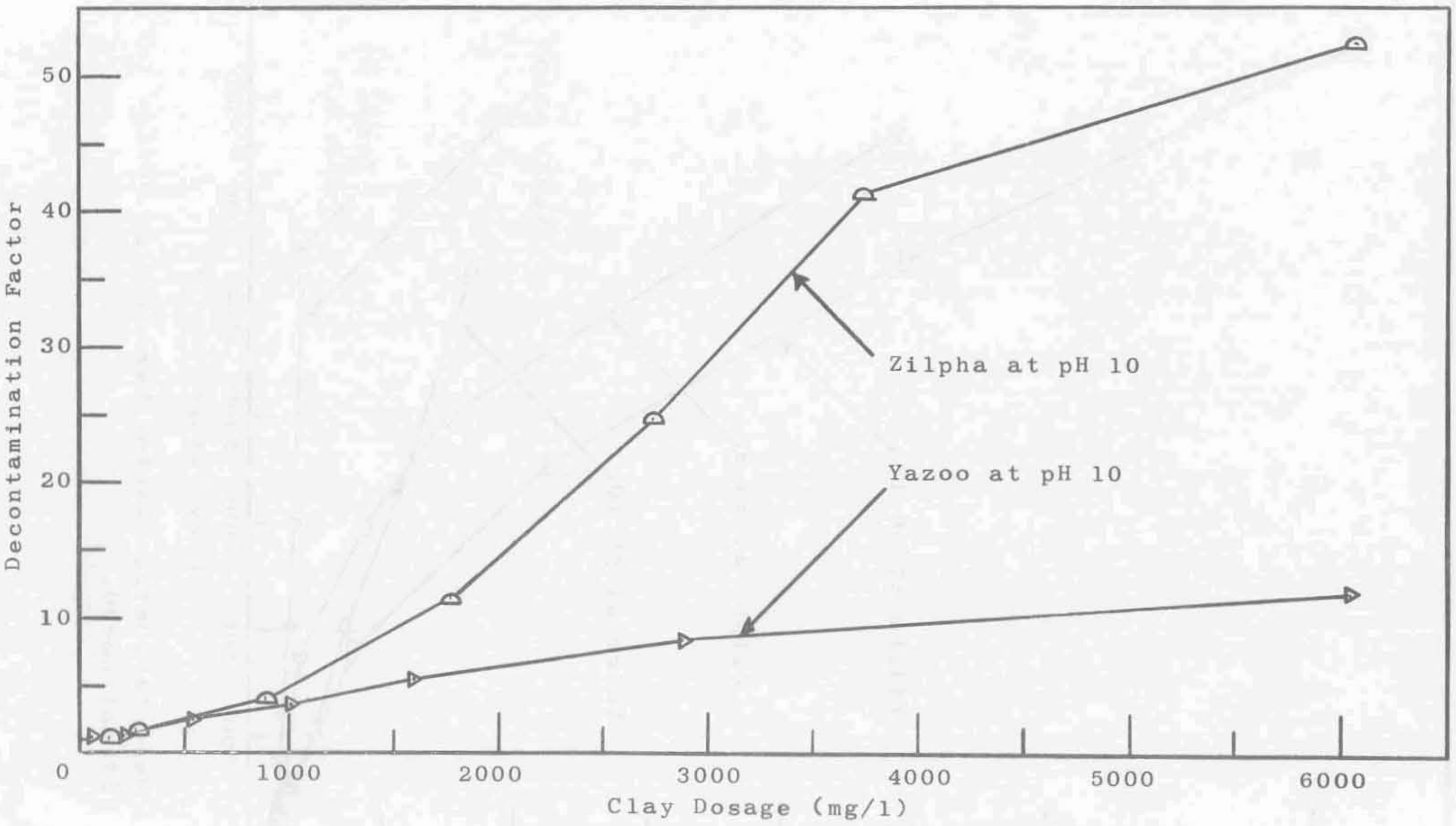

Figure 6. Decontamination Factor Versus Clay Dosage for Strontium-90.

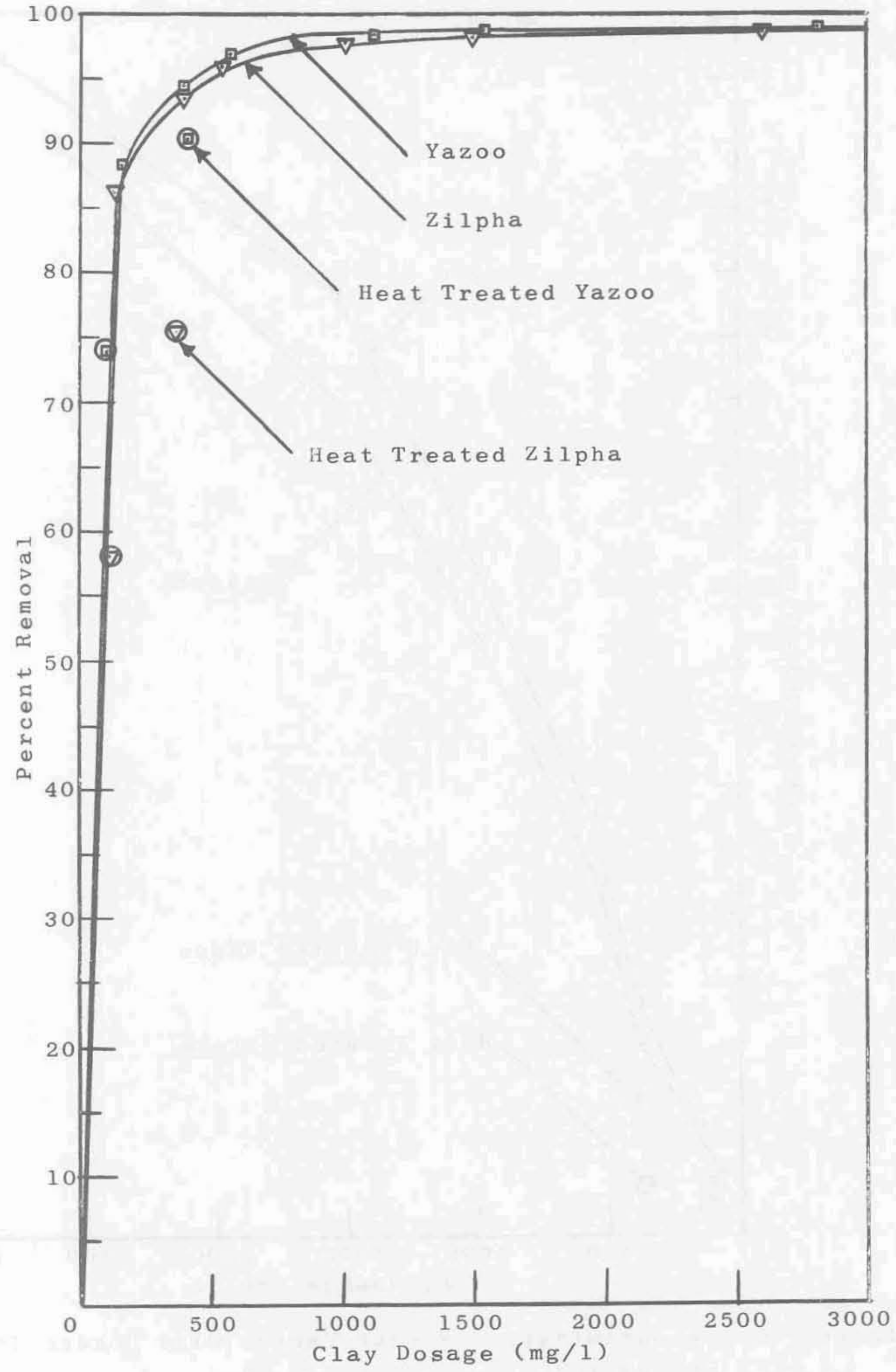

Figure 7. Percent Removal Versus Clay Dosage for Cesium-137.

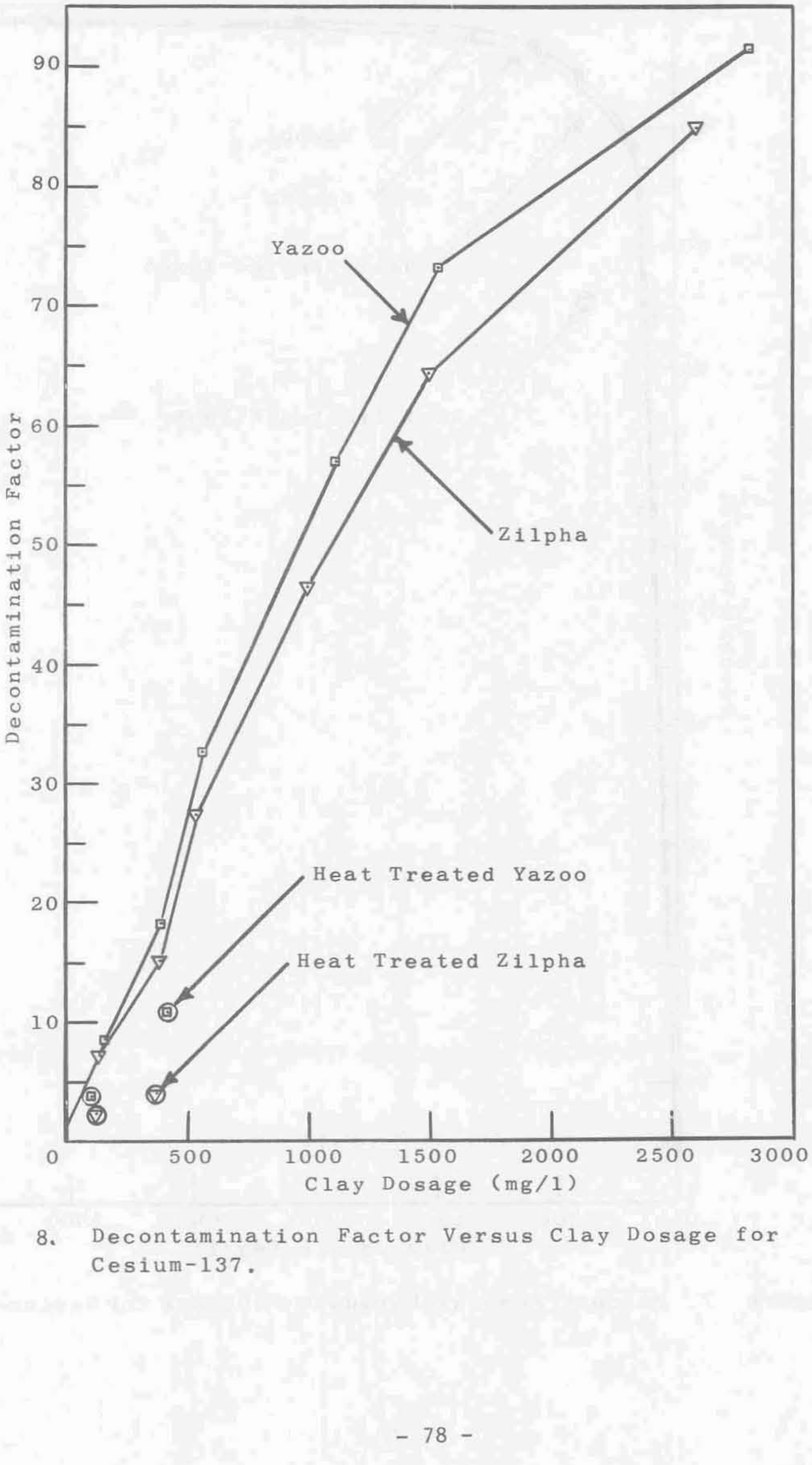

Figure 8. Decontamination Factor Versus Clay Dosage for Cesium-137.

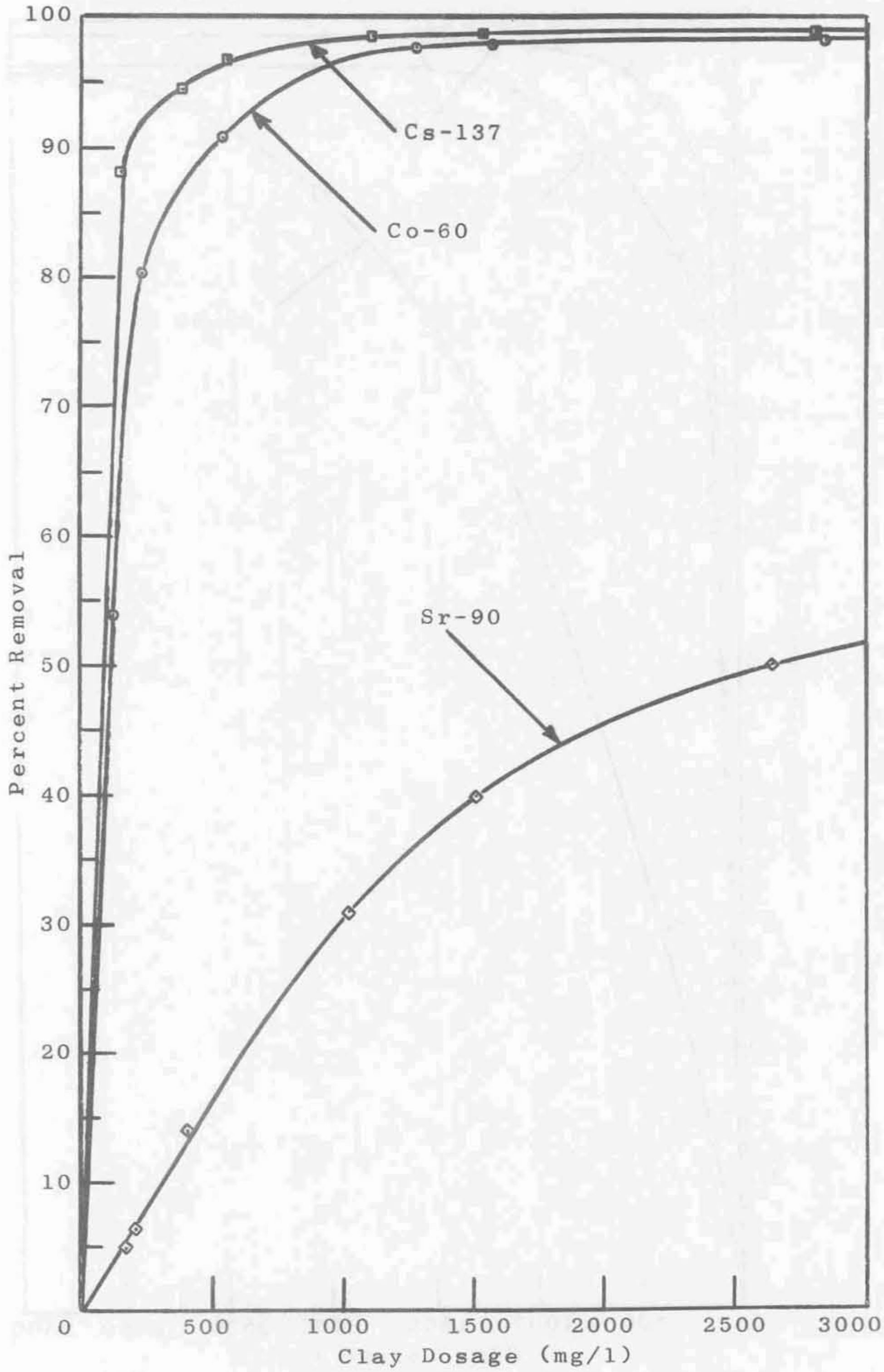

Figure 9. Percent Removal Versus Clay Dosage for Yazoo Clay.

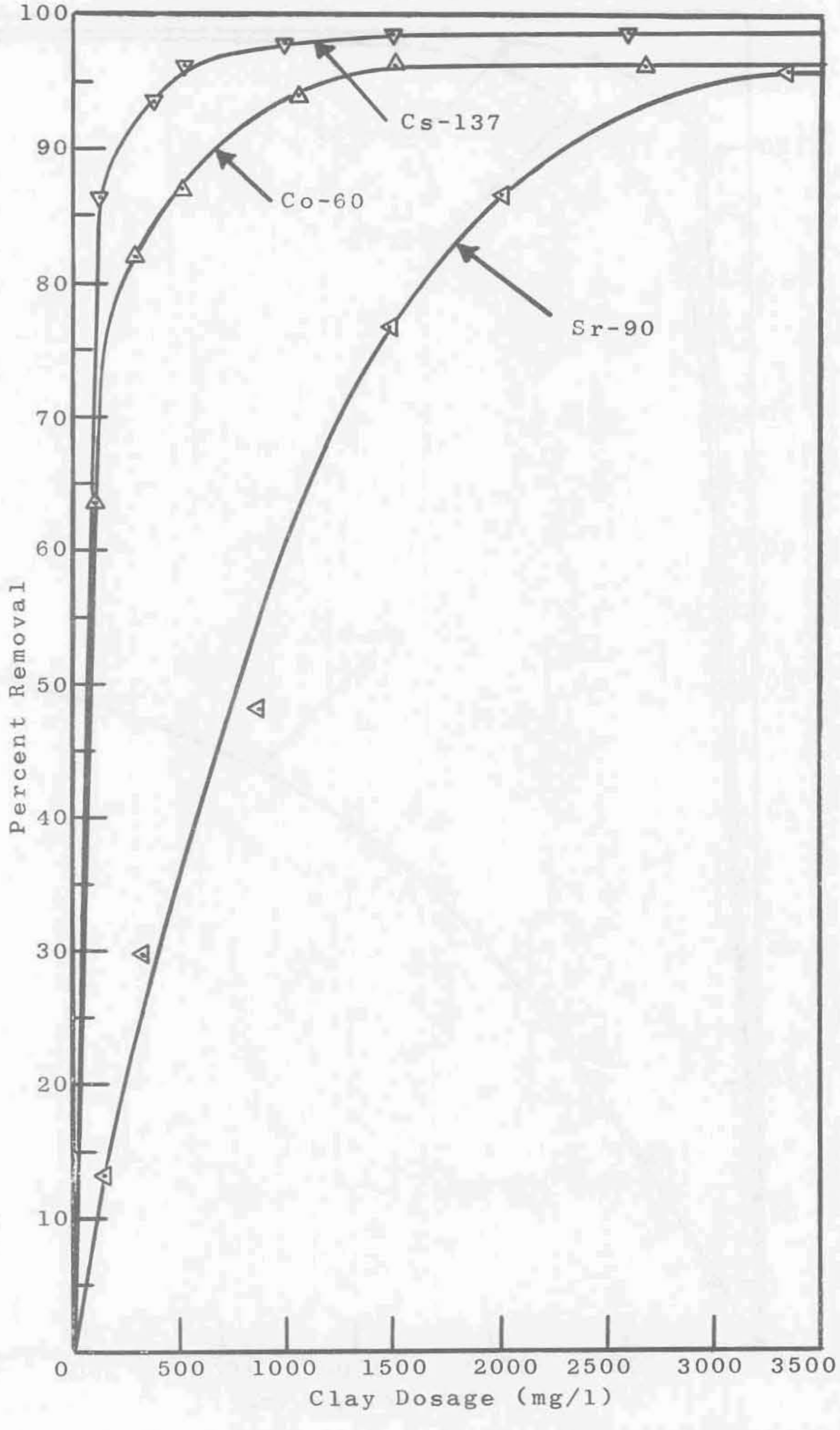

Figure 10. Percent Removal Versus Clay Dosage for Zilpha Clay.

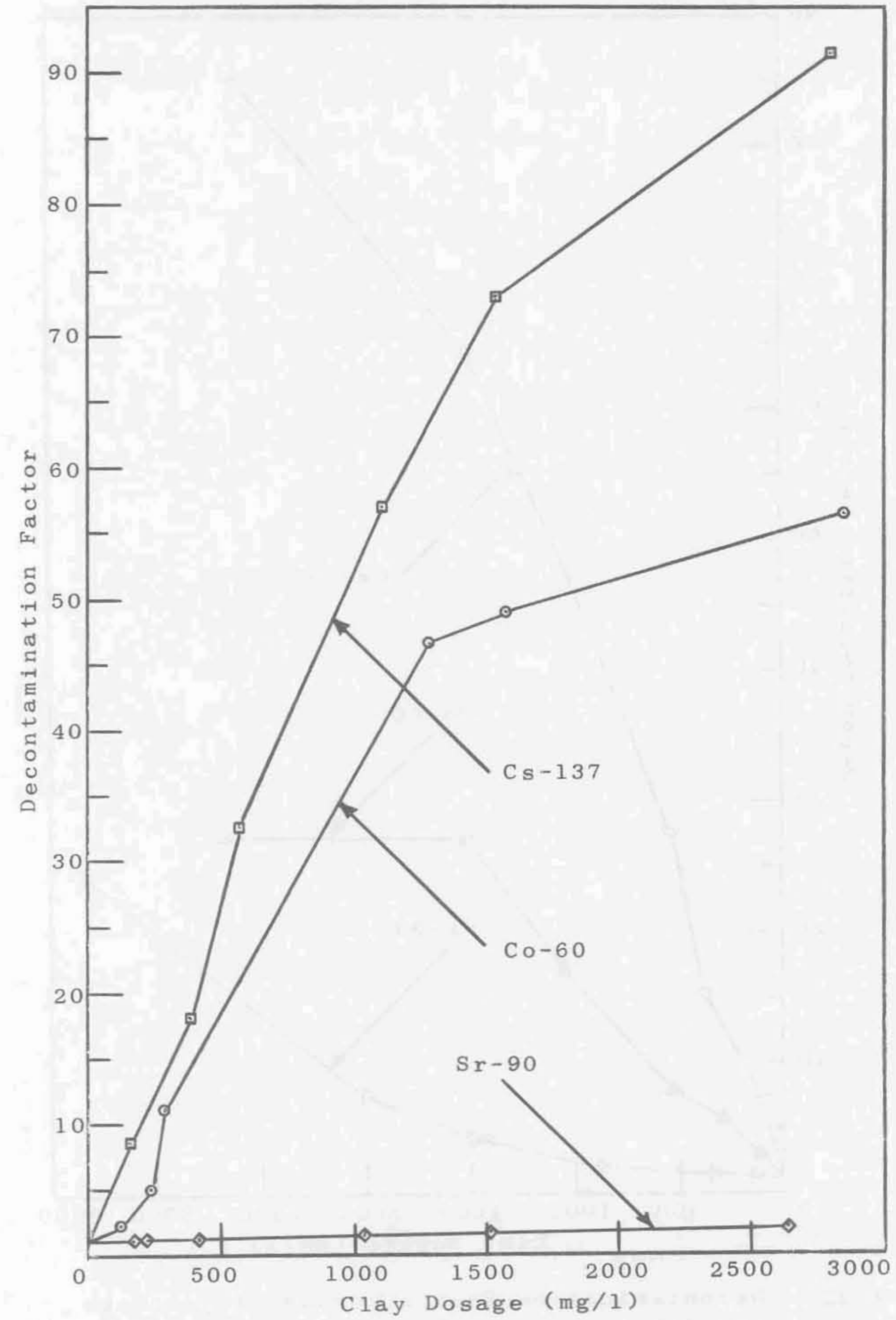

Figure 11. Decontamination Factor Versus Clay Dosage for Yazoo Clay.

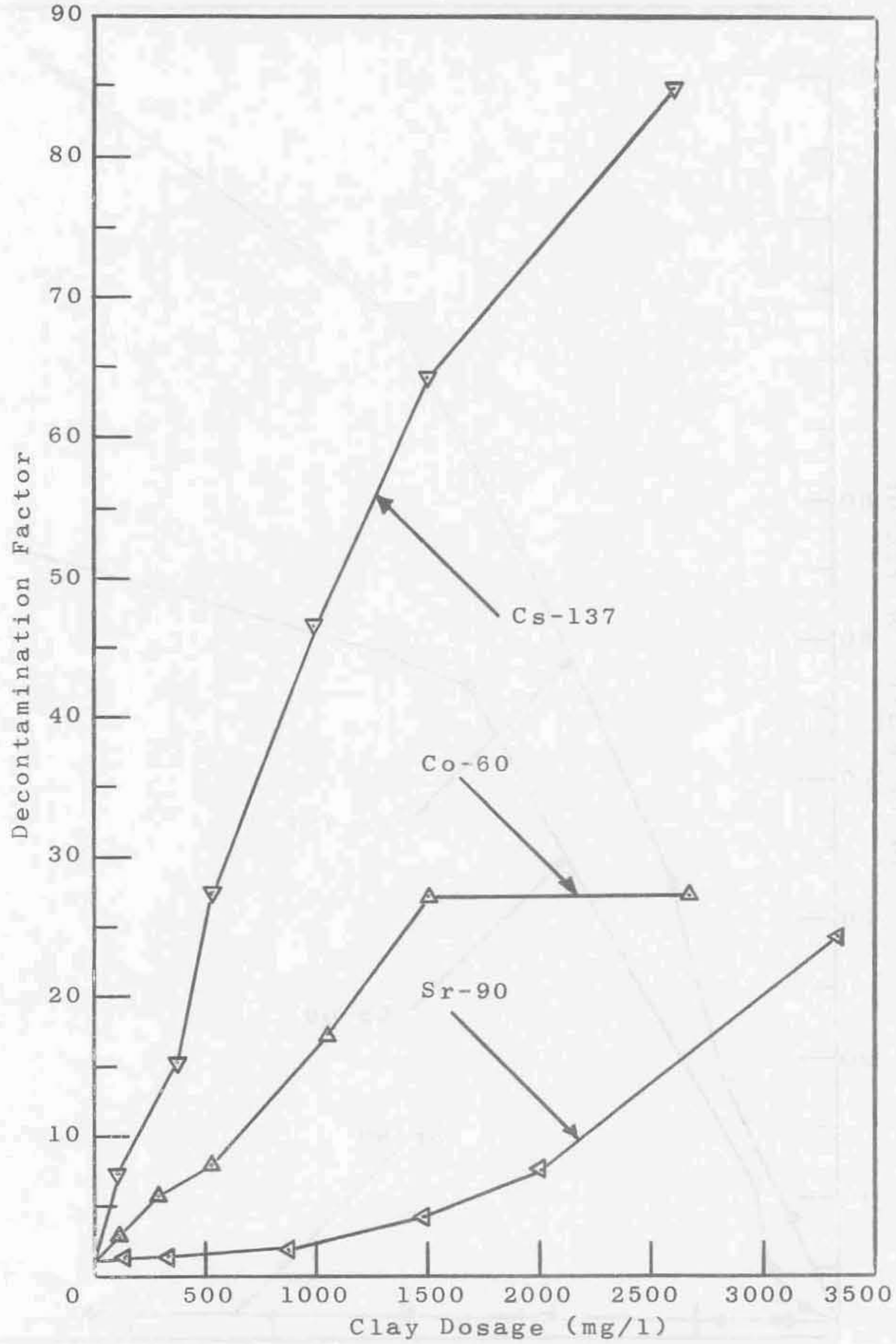

Figure 12. Decontamination Factor Versus Clay Dosage for Zilpha Clay.