EFFECT OF pH ON ALUMINUM, IRON AND CALCIUM

PHOSPHATE FRACTIONS OF SEDIMENT

IN LAKE CARL BLACKWELL

By

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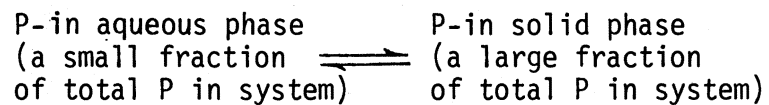
CHAPTER I

INTRODUCTION

Phosphorus is not only an essential element for plant growth, it also has the distinction of being the limiting element for aquatic plant growth as well (1)(2). Eutrophication, or over-enrichment, is defined as the natural process of maturation of a lake from a nutrient poor to a nutrient rich body of water. The discharge of domestic sewage and agricultural drainage into lakes has accelerated the eutrophication process.

There is a great deal of literature emphasizing the importance of phosphorus inflows in accelerating eutrophication. Addition of excess nutrients can stimulate production of organic matter, algal blooms, and aquatic plant growth in excess of the oxidative capacity of a body of water, thereby making it aesthetically unpleasing and reducing the recreational quality of the lake (3).

Phosphorus is available in a body of water as anions of phosphoric acid which are orthophosphate, complex inorganic phosphate, and organic phosphate. According to Pomeroy et al.(4), orthophosphate seems to be the most easily utilized form of the nutrient for algae or higher aquatic plants. However, not all of the phosphorus entering a body of water is immediately available for algal growth. Phosphorus is taken up by bacteria, and sediments also trap incoming phosphorus.

Hayes and Phillips (5) show the dynamic interaction in water and

1

sediment systems as:

$$\text{P-in aqueous phase} \rightleftharpoons \text{P-in solid phase}$$
$$\text{(a small fraction} \quad \text{(a large fraction}$$
$$\text{of total P in system)} \quad \text{of total P in system)}$$

They further state that regulation of the phosphorus concentration in the water by this interaction is controlled by phosphate mineral solubilities and by adsorption or ion exchange equilibrium at the sediment water interface.

The ability of sediment to adsorb and release phosphorus is one of the most important factors determining the nutrients' concentration in lakes. Work by Williams et al. (6) shows that there are many factors which affect the cycling of phosphorus in the sediment. Soil type and size are two important factors. For example, non-calcareous sediments sorb more phosphate than calcareous sediments. Mixing is another important parameter because it may be rate determining for phosphorus exchange between lake sediments and overlying water (5).

Possibly one of the most important factors affecting phosphorus exchange between the sediment and the water is pH. The pH of the overlying water and sediment is a dominating factor because it controls the concentrations of available iron, aluminum, and calcium (4) as well as affecting sorption-desorption reactions, precipitation-solubilization reactions, and oxidation-reduction reactions (6). Because of its direct and indirect effects on all aquatic biological and chemical reactions, pH ultimately becomes a major controlling factor in the availability of sediment bound phosphorus for algal utilization and the subsequent enhancement of eutrophic conditions. The purpose of this investigation was to study the effects of pH on the aluminum, iron, and calcium phosphate fractions of sediments obtained from Lake Carl Blackwell, Oklahoma.

CHAPTER II

LITERATURE REVIEW

It is generally agreed by ecologists and biologists that phospho-
rus is one of the most important nutrients required to support growth
of aquatic plants (7)(8)(9). Even though it is difficult to clearly
define the influence of a single element on the growth rate of aquatic
organisms, a body of literature does exist showing that there is a
minimum level of nutrient content below which there is little or no
growth of aquatic plants. Above this minimum, experiments have shown
that there is a second level at which there is an accelerated growth
of aquatic plants like algae and phytoplankton. When nutrient contents
exceed these levels, the eutrophication process may proceed rapidly
(10)(11).

In general, it is found that the minimum phosphorus content needed
for growth of algae is below 0.01 mg/l, and phosphorus concentrations
in excess of 0.1 mg/l often cause accelerated growth of various types
of aquatic plants (12). The nutrient sources contributing to eutrophi-
cation of a body of water consists of agricultural run-off from pasture
land and fertilized fields, sewage effluent, and effluent from indus-
tries. Internal nutrients are derived from the death and decomposition
of algae, plankton, and other aquatic plants and organisms like zoo-
plankton, fish, bacteria, and also from bottom sediments (13).

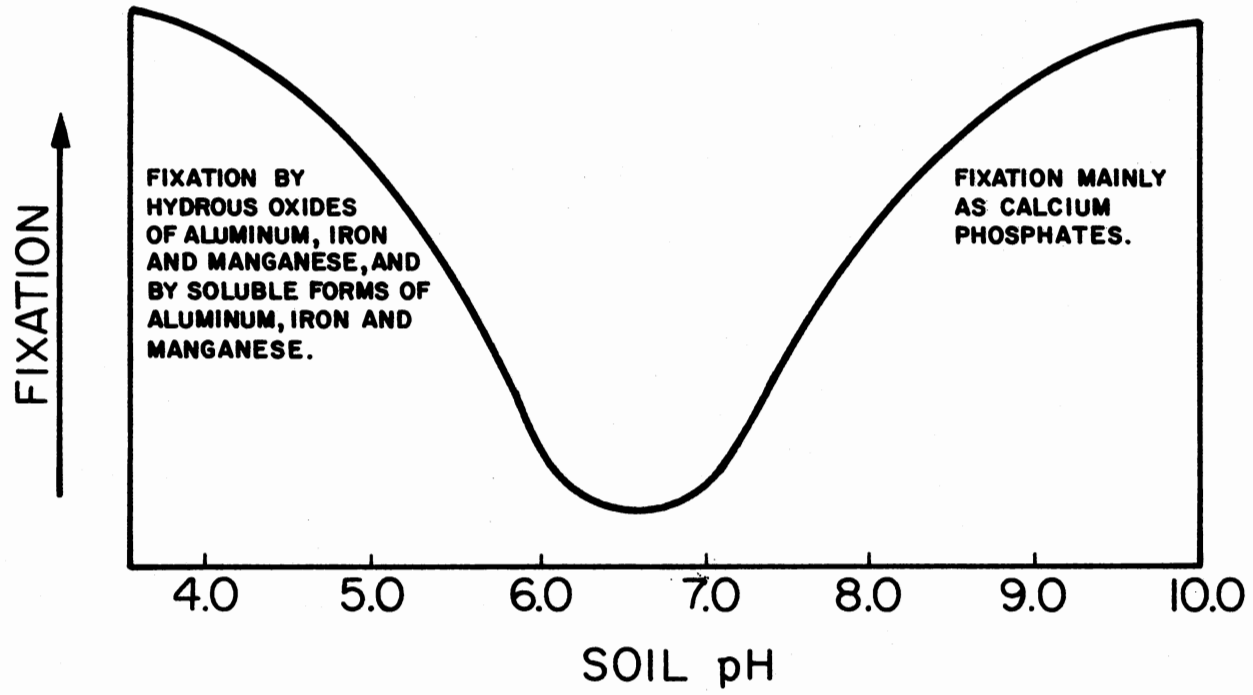According to Lee (14), lake sediments generally contain one to

two milligrams of phosphorus per kilogram of dry sediment. The sediments of a lake can provide a buffer system for many elements. The effect of this buffer system could be to keep the concentration in the overlying water constant even if concentrations of the element in the inflowing waters vary widely. Pomeroy et al. (4), working with estuarine sediment, states that these sediments act as a buffer to keep the concentrations of phosphate in water constant. Harter (15), in a study of phosphorus adsorption sites in soils, suggests that the lake sediment may act as both a sink and a reservoir. If the sediment is continually enriched with phosphorus by adsorption and then by precipitation as Al and $Fe-PO_4$, it acts as a sink. If the phosphorus in the water decreases, the sediment may release the phosphorus which is in the anion exchange sites and consequently act as a reservoir (15).

Phosphorus fixation in sediment depends on two important factors—soil type and pH (16). According to Buckman et al. (17), most phosphorus in soil falls under two groups—those which contain calcium and those which contain aluminum and iron. Soils that essentially consist of silica fix none or very little phosphorus. In a study on twenty-five sediments from Wisconsin lakes by Shukla et al. (18), lake sediments sorbed between 20 and 100% of inorganic phosphate which was added at a level of 0.83 mg/l to oven dried sediment. He reports that calcareous sediments fixed less phosphate than noncalcareous sediment and sorption of phosphorus tended to be inversely related to the calcium carbonate content of the calcareous sediment. These results are also supported by Murrmann and Peech (19) on the study of the effect of pH on labile and soluble phosphate in soils and by McAllister and Logan (20).

The pH of the sediment is a dominating factor in phosphorus fix-
ation since concentrations of available aluminum, iron, and calcium
are controlled by this parameter (16). Inorganic phosphorus in sedi-
ment consists as salts of orthophosphatic acid. These salts are
classified as oxy-, fluoro-, and hydroxy phosphates of iron, aluminum,
calcium and a few other minerals (14)(16). One or more of the ionic
forms of orthophosphatic acid might be present in all fixation reac-
tions where phosphorus is involved. Over the pH range of 4.0 to 8.0
the principal ionic species in the sediment are $H_2PO_4^-$ and HPO_4^{-2}. The
monovalent ion, $H_2PO_4^-$ is dominant (16)(17). Therefore, in a highly
acidic environment only $H_2PO_4^-$ ions are present. If the pH is increas-
ed, first the HOP_4^{-2} and finally PO_4^{-3} dominates. HOP_4^{-2} is not as readily
available to aquatic plants as $H_2PO_4^{-1}$. In lake sediment this relation-
ship is complicated by the availability or absence of other compounds
or ions such as soluble iron and aluminum under acidic conditions or
calcium under basic conditions, which is responsible for affecting the
availability of phosphorus. Therefore, it is apparent that the effect
of the sediment's pH on phosphorus availability is determined to a
large degree by various cations present (17). Sediments in acidic
environments are characterized by iron and aluminum phosphate but in
an alkaline environment, calcium phosphate would be expected to pre-
dominate (24)(25). This relationship is expressed in Figure 1 (16).

According to Black (21) the clay fraction of the sediment is the
most active portion of the sediment in phosphate fixation. This phos-
phate fixation ability is attributed to reactions of phosphate with
iron and aluminum which are present in forms of hydrous oxides or ex-
changeable cations. The strong sorption of phosphorus to clays is

Figure 1. Form of Phosphorus Fixation as a Function of Soil pH.
Above Illustrates Average Conditions and no
Inference Should be Made to any Particular Soil (16)

FIXATION BY
HYDROUS OXIDES
OF ALUMINUM, IRON
AND MANGANESE, AND
BY SOLUBLE FORMS OF
ALUMINUM, IRON AND
MANGANESE.

FIXATION MAINLY
AS CALCIUM
PHOSPHATES.

FIXATION

SOIL pH

caused by specific chemical interactions of the phosphate group with Al^{+3} on the plate edges of the clay surface. The works of Harter (22), Pomeroy et al. (14) and MacPherson et al. (23) with different lake sediments show that phosphate is sorbed readily by sediments, and this process is reversible and is dependent on pH with maximum sorption occurring in the slightly acidic environment.

Chen et al. (26), in a kinetic study of phosphate reaction with aluminum oxide and kaolinite, found that a minimum of phosphate fixation occurs when soil is "neutral" (pH 6 to 7). This is also supported by other authors (16)(17)(19).

In a study of the fixation of phosphate by kaolinitic clay, Black (21) found a maximum fixation at pH 3 to 4 regardless of the concentration of phosphate or the time of contact. However, when the dilute phosphate solution was used maximum fixation was shown to be a pH 5 to 7. Black believes that there is exposure of silica groups at the edges of the crystals, and available valences are created by breakage of a crystal into two parts which react with water to form hydroxyl groups which in turn might account for the fixation observed at approximately a neutral pH. Murphy (27), in a study of the fixation of phosphate by soil clay, has also suggested that the fixation at pH 5 to 7 is due to an exchange reaction between the phosphate and the hydroxyl groups on the clay.

In order to predict phosphorus exchange between sediments and water resulting from changes in Ph, it is necessary to recognize and understand what aquatic processes can induce pH changes. Microbial activity, photosynthesis-respiration of aquatic plants, and oxidation-reduction reactions can all ultimately affect or be affected by pH and

may cause the release or uptake of phosphorus from the lake sediments (15).

Even though chemical and physical factors affect nutrient equilibria, the bottom organisms and aquatic plants have a significant effect. Purcella (28) states, "Biological effects cannot be separated from the physical and chemical relationships which control nutrient availability because increased nutrient concentrations can lead to increased biomass and therefore increased utilization of nutrients". Generally it is believed that microorganisms such as bacteria play a dominant role in biological transformation. Usually these microorganisms are pH dependent. Microbial metabolic by-products and death and decomposition of these bottom organisms will ultimately affect the pH of the lake sediment (28).

According to Lee (14) organisms are directly or indirectly responsible for the kinetics of exchange reactions. Direct evidence found by Pomeroy et al. (4) indicates that phosphate is released from disintegrating particles as they pass through zooplankton or other organisms. The indirect effects involve the biochemical reactions which bring about a change in pH and other chemical characteristics of the water which alter exchange reactions. These biochemical reactions include primary photosynthesis and respiration reactions which both tend to alter the pH of the overlying water and influence the amount of organic matter present (14). Due to the increased biochemical respiration of organics present in the lake sediments, hypolemnetic waters might show decreases in several pH units during periods of summer stratification. Lee (14) states that in Lake Mendota, just before fall over-turn, the hypolemnetic waters have a pH of about 6.5 while epilemnetic waters

have a pH of about 8.5 to 9. This high pH in surface water has been attributed to photosynthesis which causes a removal of CO_2. This pH change could have a significant effect on exchange reactions of bottom sediments and the overlying water, especially if the pK for the $HOP_4^=$ and $H_2PO_4^-$ is near 7. Therefore, the investigator (14) proposes that, if these two species are markedly different in the sorption tendencies, a change in pH could change distribution of phosphorus between sediments and overlying water.

Oxidation-reduction potential of sediment plays a major role in controlling many exchange reactions including phosphorus exchange (5). Redox systems present in water are, to a large extent, controlled by the growth and death of photosynthetic organisms (30). It is well known that very often reducing conditions in the lake sediments cause the release of some compounds such as phosphorus, iron and aluminum to overlying water. Hayes and Phillips investigating interaction of lake water and sediment repeatedly found that the removal of oxygen either over the lake bottom or in laboratory containers will allow inorganic phosphorus to come up out of the sediments. Various studies show that ferric iron in lake sediments influences and controls the exchange of phosphorus from the lake sediment to the overlying water. Mortimer (31) states that under reducing conditions the phosphate is released because ferric iron is reduced to ferrous iron, and ferrous phosphates will be solubilized in the overlying waters. Work by Botch (32) studying the transformation of iron sediment as influenced by redox potential and pH shows that at pH 6 to 7 the conversion of reducible iron to the water soluble and exchangable form took place at redox potential between +300 mV and +100 mV, while a redox potential

of -100 mV had to be attained at pH 8 before iron was released. Therefore, he concluded that a combination of low redox potential and low pH had a distinct effect on the increased solubility of at least some of the iron phosphate (32).

CHAPTER III

MATERIALS AND METHODS

The sediments studied in this investigation were collected from
Lake Carl Blackwell, located approximately seven miles west of Still-
water, Oklahoma. It is a warm monomitic lake, characterized as
eutrophic, and subject to hypolimnetic oxygen depletion in the summers.

The sediments were obtained by dredging the bottom of the lake to
a depth of about 10 cm in an area where the depth of the water approxi-
mated 5 to 6 feet. The sediment was then brought to the lab and was
air dried in thin layers in shallow porcelain pans at room temperature.
After complete drying, the sediments were ground and sieved to attain
uniform size. The samples were then stored in covered containers. A
portion of such sample was sent to the Oklahoma State University Soils
Laboratory for determination of the amount of silt, sand and clay
present. The pH of the sample was found by first moistening the dry
sediment with distilled water until it reached the consistency of a
thick paste, and then allowing 30 minutes for equilibration. The pH
was determined with an Orion Research Digital Ionalyzer model 601A,
and a Sargent-Welch glass pH electrode.

Experimental Approach and Set-up

This experiment was set up to measure the amount of phosphorus
bound in the aluminum, iron and calcium fractions of the sediment at

various pH values. A determination of the "saloid" bound phosphorus
was also made. Saloid-bound phosphates are easily soluble and loosely
bounded phosphorus. A 250 gram sample of dried sediment was placed
into each of ten 600 ml Pyrex beakers and to these containers approx-
imately 300 ml of distilled water with pH ranging from 1 to 12 (pH of
1, 2, 3, 4, 6, 7, 9, 10, 11, and 12) was added. HCl and $Ca(OH)_2$ were
used to decrease or increase pH of the distilled water in order to
arrive at these exact pH values. To minimize evaporation these beakers
were covered with aluminum foil and were left to equilibrate. By
measuring the pH of the sediments daily the optimum equilibration time
was found to be two weeks, even though most of the soil's pH change
occurred in the first two days and continued to increase or decrease
slowly depending on the acidity or alkalanity of the overlying water
during the next 12 days.

Experimental Procedures

In order to determine the amount of aluminum, iron and calcium
phosphate of each of the 12 samples after two weeks of equilibration,
50 ml of the aqueous sediment samples were taken and after filtration,
the collected sediment was dried in an oven at 90°C.

After approximately 25 hours the sediments were removed from the
oven and ground. Approximately one gram of each sample was fraction-
ated by a modified Chang and Jackson procedure (33) as outlined by
Peterson and Corey (34). All fractionations were done in duplicate.

The order of extraction of inorganic phosphate was as follows:
"saloid-bound" phosphate, soluble in 1 N NH_4 Cl, after shaking for
30 minutes; aluminum phosphate, soluble in 0.5 N NH_4F with pH of 8.2,

after shaking for 24 hours; iron phosphate, soluble in 0.1 N NaOH, after shaking for 17 hours; calcium phosphate soluble in 0.5 N H_2SO_4, after shaking for one hour. After each extraction, the sediment sample was washed two times with 25 ml of saturated NaCl centrifuged at 2,000 rpm for 5 minutes, and the NaCl removed by decantation. This centrifugation speed was also employed for ten minutes in the separation of NH_4F for aluminum extraction and fifteen minutes for extraction of iron and calcium with NaOH and H_2SO_4 respectively.

After each extraction a 3 ml aliquot of the extract was dispensed into a colorimeter tube and 3 ml of chloromolybdic boric acid was added. After mixing, one drop of chlorostannous reductant was added to produce a blue color. Percent transmittance was measured at 660 mμ within 15 to 20 minutes.

CHAPTER IV

RESULTS

Some of the chemical and physical properties of the lake sediments studied in this investigation are shown in Table I. These results indicate that silt is the major component of the sediment in Lake Carl Blackwell. The initial pH of the collected sediment was 6.93.

Table II shows the pH values of overlying water which were experimentally fixed from pH 1 to 12 by the addition of hydrochloric acid for acidic pH values and calcium hydroxide for basic pH values. The initial and final pH values of the lake sediments after equilibration for two weeks with experimental overlying water are also shown in this table.

The effects of sediment pH on the saloid bound phosphorus fraction are shown in Figure 2. Plotted data points in this and following figures represent an average of the values obtained from duplicate fractionations. The saloid bound fraction predominates in a very acidic environment, and as the pH increased, the amount of phosphorus fixed in this fraction rapidly decreased until pH 7.34 at which time the quantity of phosphorus bound remained relatively constant.

Figure 3 represents the data collected from the study of the effects of Ph on the aluminum phosphate fraction of the sediment. Minimum fixation occurs at a pH of 7.33. Aluminum phosphate is slightly more prevalent in the sediment in the acidic pH range than in the alkaline range.

TABLE I

SEDIMENT COMPOSITION WITH RESPECT
TO SILT, SAND AND CLAY

Constituent	Amount Present
Silt	41%
Sand	34%
Fine clay	17%
Coarse clay	4%

TABLE II

EXPERIMENTAL OVERLYING WATER pH VALUES AND
FINAL SEDIMENT pH ACHIEVED AFTER
TWO WEEKS OF EQUILIBRATION

Experimental Overlying Water pH	Initial Sediment pH	Final Sediment pH
1	6.93	1.55
2	6.93	4.21
3	6.93	6.92
4	6.93	7.19
6	6.93	7.31
7	6.93	7.33
9	6.93	7.34
10	6.93	7.36
11	6.93	7.37
12	6.93	8.95

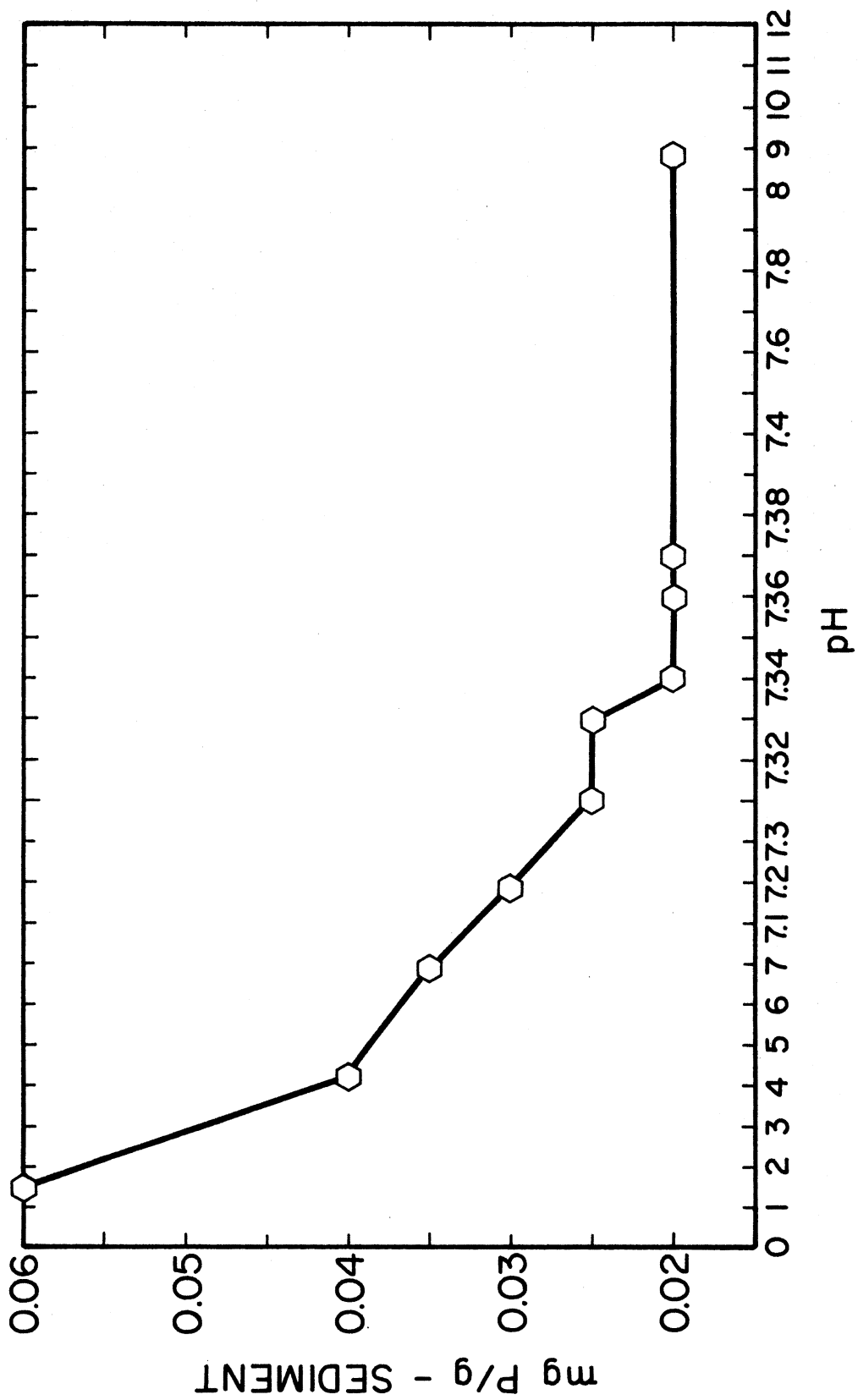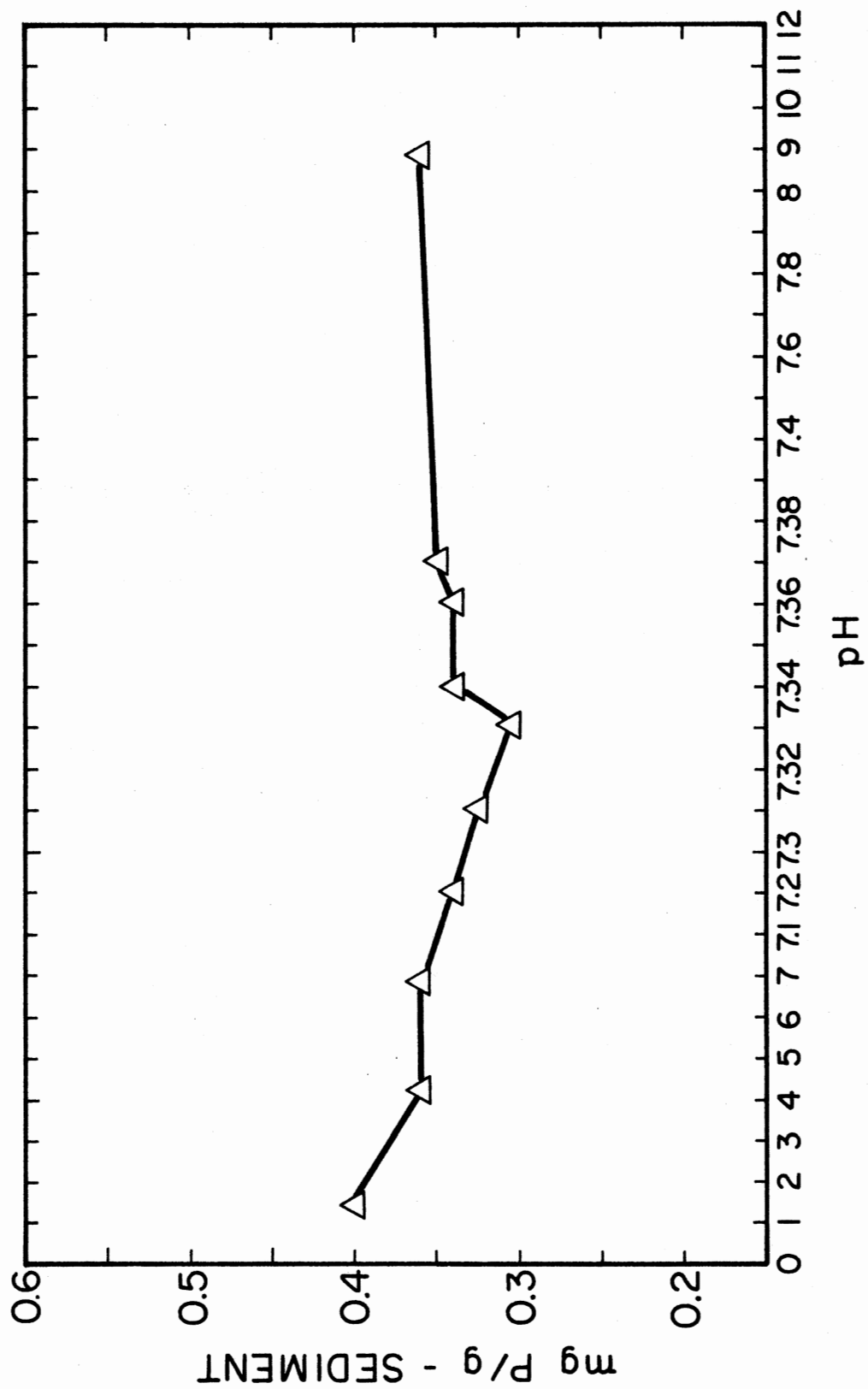Figure 2. Saloid-Bound Phosphorus Versus Sediment pH

Figure 3. Aluminum Bound Phosphorus Versus Sediment pH

Iron phosphate concentrations in the sediment versus pH are shown in Figure 4. From this figure, it can be seen that iron, like aluminum phosphate predominates in the acidic and alkaline range and is at a minimum of pH 7.33.

Figure 5 shows phosphorus fixation by calcium versus pH. The minimum amount of phosphorus present in the calcium fraction occurred at a pH of 1.55. There was a rapid increase in the amount fixed until pH 7.31 was reached. From pH 7.31 to 7.33 there was a rapid decrease in calcium bound phosphate and a slight decrease occurred from pH 7.33 to 8.9.

Concentrations of saloid-bound, aluminum, iron and calcium phosphates in one gram of sediment under different sediment pH values are shown in Figure 6. Total sediment phosphorus concentrations in one gram of sample under different sediment pH values are shown in Figure 7. As can be seen from this figure minimum phosphate fixation occurred at a neutral pH of 7.33.

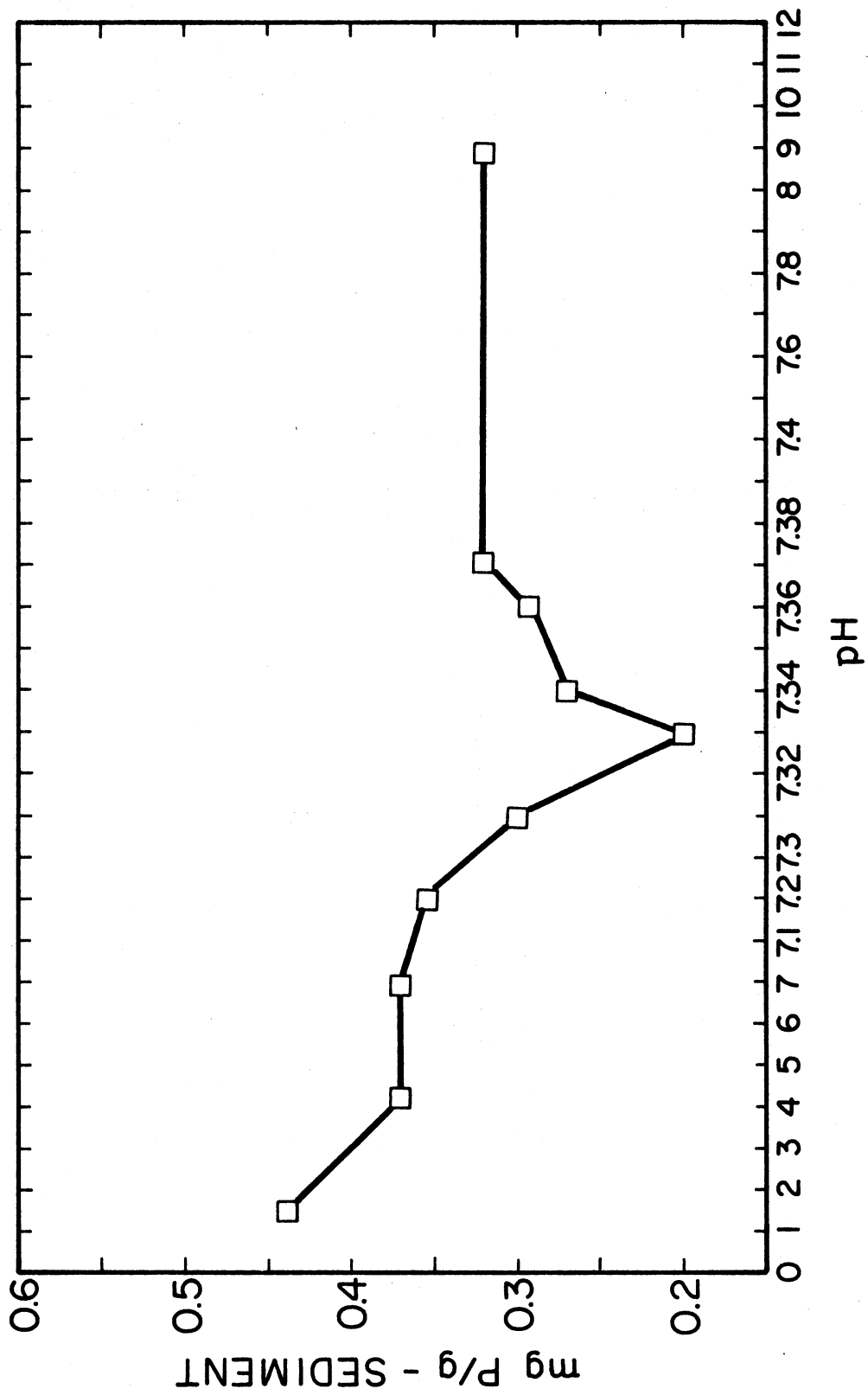Figure 4.   Iron Bound Phosphorus Versus Sediment pH

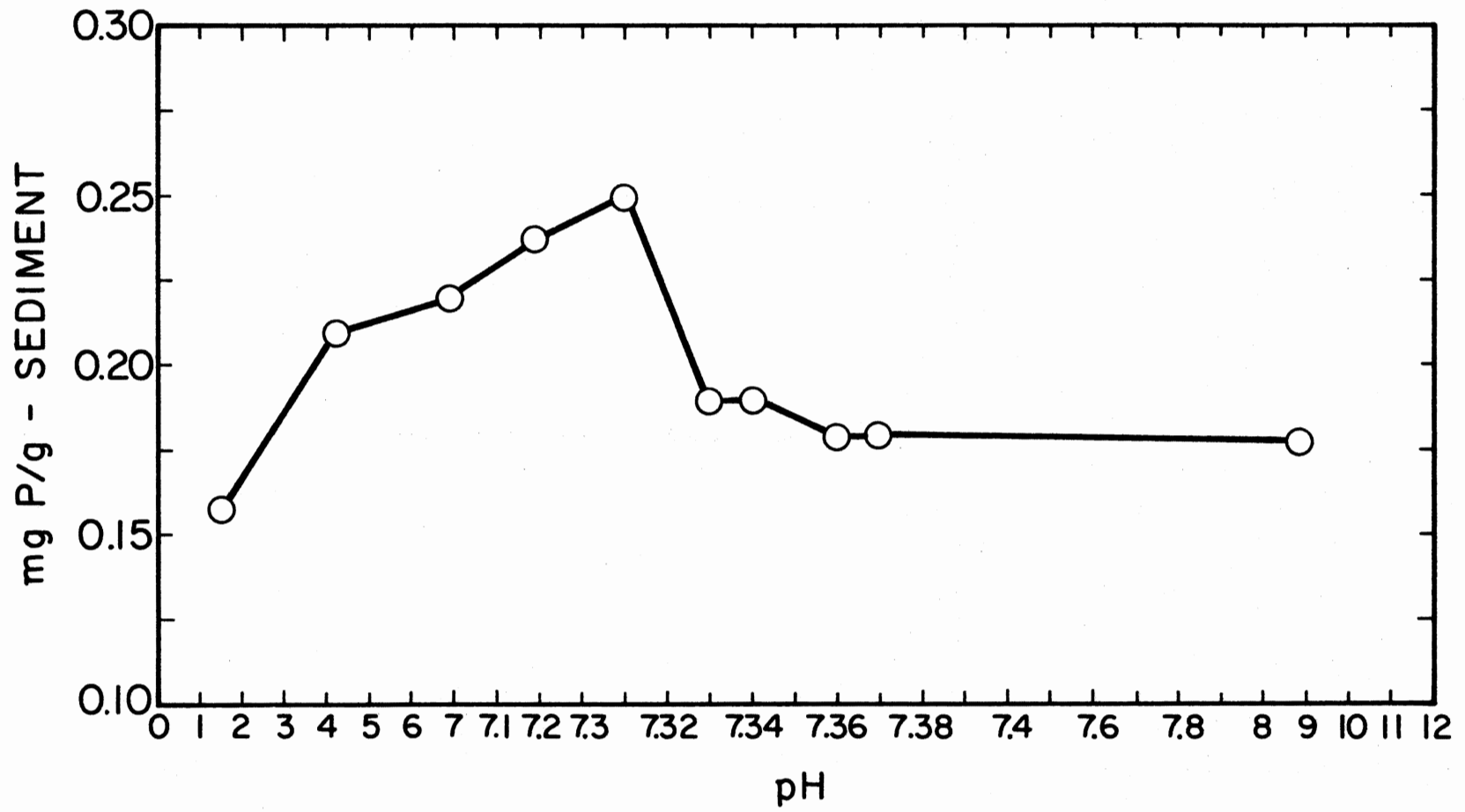Figure 5. Calcium Bound Phosphorus Versus Sediment pH

Figure 6.   Aluminum-Iron-Calcium- and Saloid Bound Phosphorus
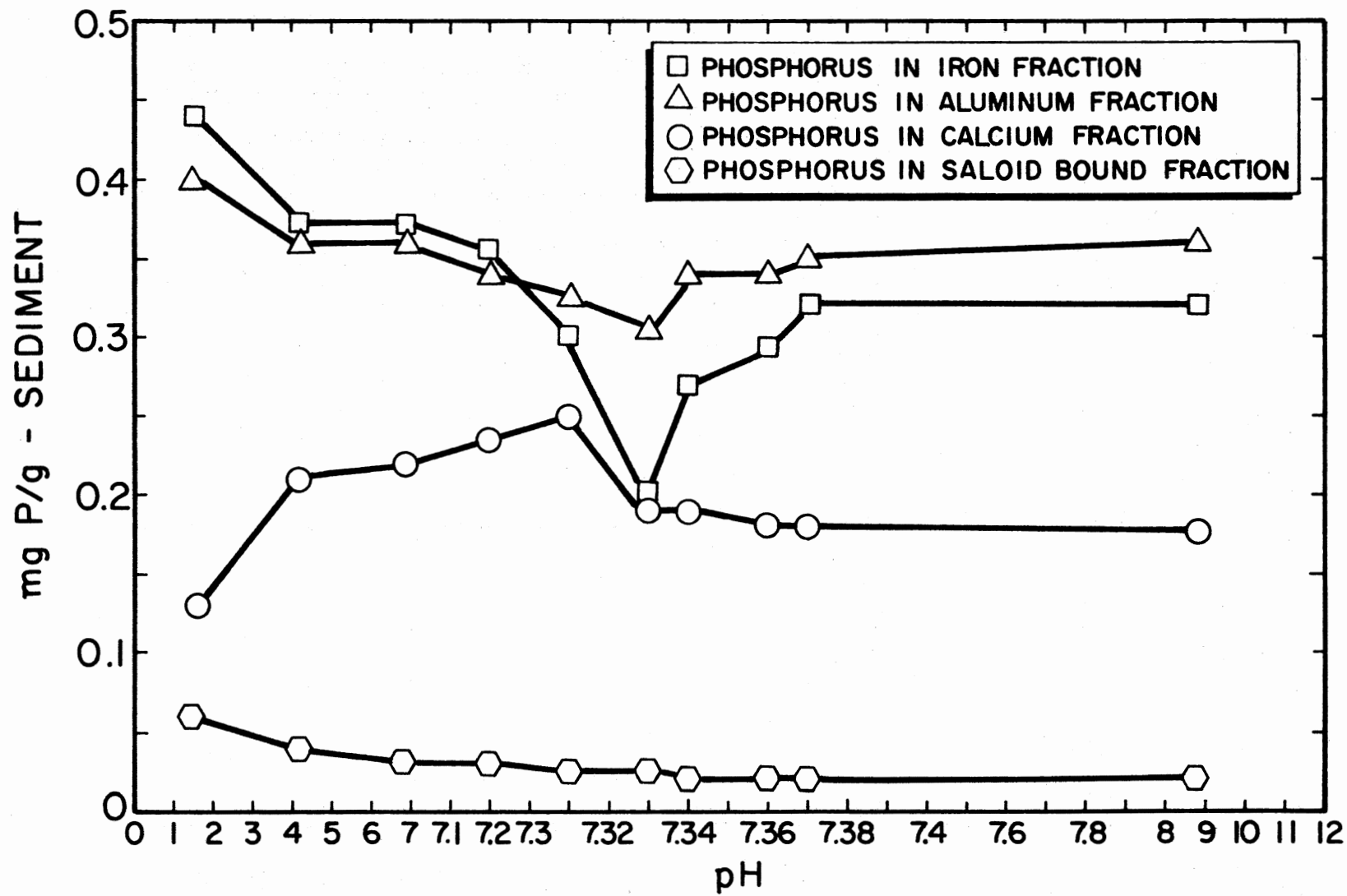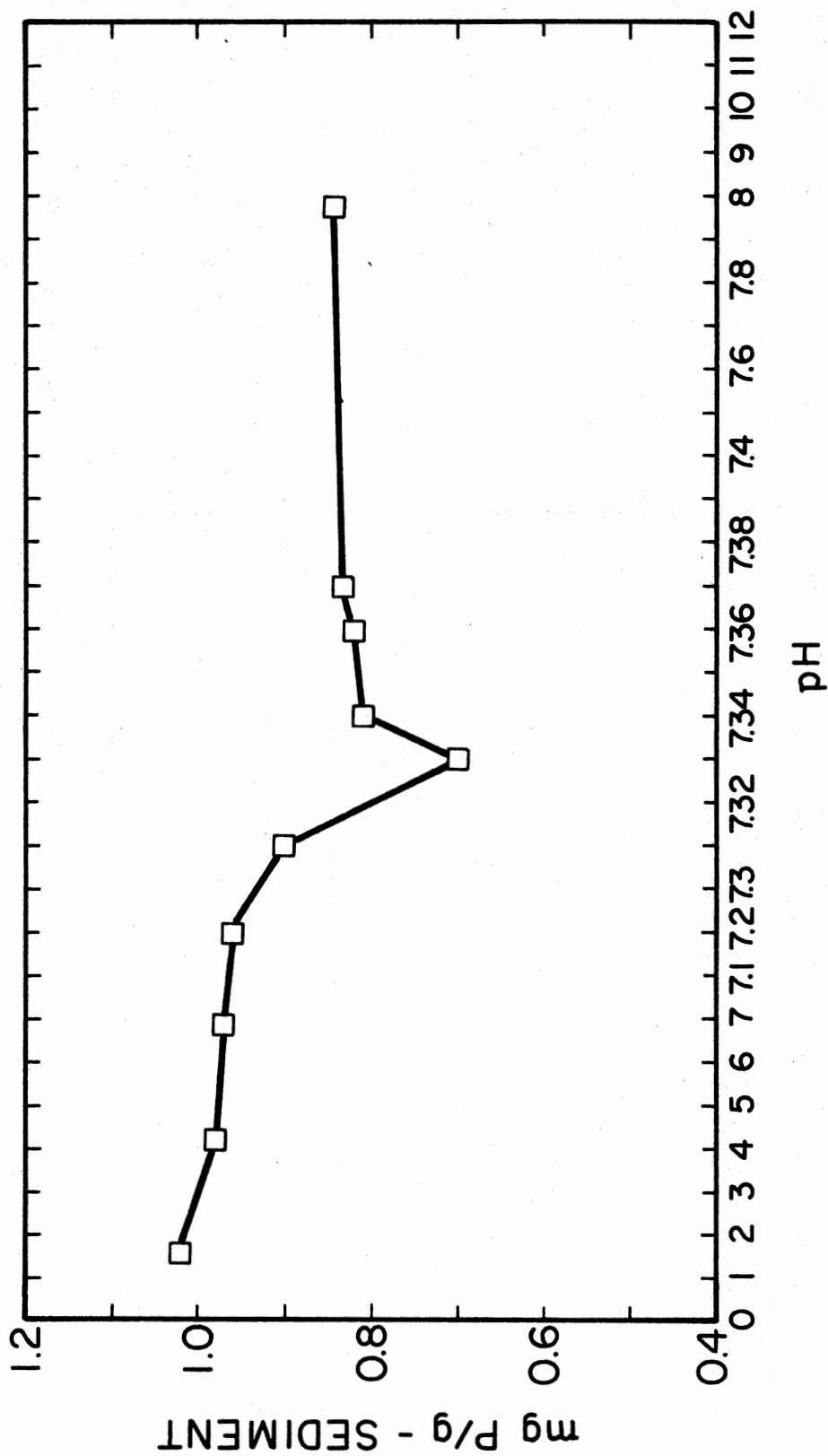Versus Sediment pH

Figure 7. Total Phosphorus in Saloid, Aluminum, Iron, Calcium
Phosphate Fractions Versus Sediment pH

CHAPTER V

DISCUSSION

With respect to the pH at which minimum sediment phosphorus fixation occurs, the results of this experiment agree with those of previous researchers. However, since no significant work could be found in the literature in which the affects of Ph on the various phosphate fractions were ascertained, little comparison can be made.

Saloid bound phosphates are easily soluble and loosely bound, and as can be seen from Figure 2, one gram of sediment sample contained only an average of 0.03 mg of phosphate. This finding is agreeable with the findings of Chang and Jackson who, after extraction of their sediment sample with 1 N NH_4Cl to determine the saloid bound fraction, found that phosphorus in the solution was generally very low. Figure 2 also indicates that in acidic environments the amount of saloid bound phosphate was at a maximum and the quantity decreased as the sediment became more alkaline.

Figure 3 concerning aluminum phosphate and Figure 4 concerning iron phosphate indicate that both the aluminum and iron phosphate fractions predominate in acidic environments, and as can be seen, the sediment contained more iron bound phosphate than aluminum bound phosphate at the lower pH values. Both the aluminum and iron phosphate fraction reached their point of minimum fixation at pH 7.33. These results agree with findings of other investigators which claim that

maximum solubilization or minimum fixation of a sediment's phosphate occur at a neutral pH.

Calcium phosphate, however, reached a point of minimum fixation at acidic pH values and its maximum fixation occurred at pH 7.31 as can be seen in Figure 5. According to the literature calcium phosphate should predominate or reach its point of maximum fixation in alkaline environments. However in this experimental work the concentration of calcium phosphate was near minimum in the pH ranges investigated. This occurrence might be because $Ca_5(OH)(Po4)_3$, calcium hydroxyapatite, which is one of the major precipitated forms of calcium in an alkaline environment is essentially soluble at pH values less than 9, and due to the buffering capacity of sediment, the highest pH experimentally achieved was 8.95. Therefore, phosphorus bound as calcium hydroxyapatite would not be found in sediment and this could probably account for the lack of increase in calcium bound phosphate that was expected.

Iron and aluminum phosphate are the major phosphorus fractions found in the Lake Carl Blackwell sediments analyzed. Figure 7, which represents total phosphorus fixed versus pH, indicates that the point of minimum fixation occurred at a pH of 7.33. At this point the quantity of iron bound phosphorus decreased to a greater extent than any other fraction.

The pH at which fixation is at minimum is well within the range of natural water pH, since most lakes' sediment pH ranges from 6 to 8, and with slight shifts in pH, release could occur. However this does not necessarily mean that all the phosphate released from sediment is directly received by overlying water since microbial populations and aquatic plants in the bottom sediments are known to take up these

nutrients for growth and reproduction purposes. The released phosphate may also be resorbed by sediments or form other compounds by chemical reactions with other components present in the bottom of the lake. The fraction of phosphorus released from the bottom sediments directly to the overlying water is available for uptake by suspended algae and microorganisms. Upon their death and decomposition, phosphorus is released and is again potentially available for fixation by the sediments, consequently maintaining the continuous phosphorus cycle.

The results of this experimental work are valuable in that they provide specific information relative to the effects of pH on phosphorus fixation by iron, aluminum and calcium. Past investigators have measured total phosphate release from sediment under different pH values or measured total phosphate uptake by sediment from overlying water by methods of radioactive P^{32}. Another group of authors have utilized different fractionation methods to determine the amount of aluminum, iron and calcium phosphate present in certain lake sediment but the work has been done at a natural sediment pH which is around 6 to 8. However, this investigation has utilized various pH values to determine to what extent aluminum, iron and calcium phosphate are potentially available for release from sediments to the overlying waters.

CHAPTER VI

CONCLUSIONS

The following conclusions can be drawn from the results of this experimental work:

1. There is more iron and aluminum phosphate than calcium phosphate in the Lake Carl Blackwell sediments analyzed.

2. The point of minimum fixation occurs at pH 7.33.

3. There is more phosphorus bound in the acidic pH range than in the alkaline.

4. The quantity of saloid bound phosphorus is minimal.

5. There is more phosphate released from iron than from any other fraction analyzed at pH 7.33.

CHAPTER VII

FUTURE WORK

The following suggestion for future work is made:

1. Determine what percent of the phosphorus released at the point of minimum fixation is available for algal and microbial utilization.

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