Nitrogen release from lake sediments

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BIOCHEMICAL and physico-chemical activity can release nitrogen from lake sediments in the form of ammonium that will accumulate under anaerobic conditions but will be oxidized to nitrate under aerobic conditions. Because nitrogen in these forms is readily used for algal growth, lake sediments represent an important potential source of nitrogen for the lake. A laboratory study was conducted to follow the release of nitrogenous compounds from sediments taken from two Wisconsin lakes. Ekman dredge samples of the sediment were taken from the shallow and deep areas of Lake Mendota and the deeper areas of Trout Lake in Wisconsin.

Lake Mendota is a hard water, eutrophic lake located in Dane County in southern Wisconsin, while Trout Lake is a soft water, oligotrophic-mesotrophic lake located in Vilas County in northern Wisconsin. The sediments were divided into two portions. One portion was used for the release studies, and the other was analyzed to determine the nitrogenous composition of the sediment.

The release studies were performed using a 500-g portion of wet sediment placed in a 21-l Pyrex bottle. Distilled water (20 l) was added to the bottle. Magnetic stirring bars were used to keep the sediment in constant motion. The stirring within the suspension was sufficient to have the release controlled by chemical-biochemical processes rather than rates of mixing. The suspensions were kept at room temperature 24 ± 2°C and shielded from the light by use of a heavy, black polyethylene cover. Samples of the suspension were withdrawn by means of a glass siphoning system and vacuum filtered through glass fiber filters. (Reeve Angel 934 AH). The filtrates were then analyzed for their nitrogenous composition using the procedures for ammonium nitrogen,[1,2] nitrite nitrogen,[3] nitrate nitrogen,[4] and soluble Kjeldahl nitrogen.[1,3] The sediments were analyzed using procedures for exchangeable ammonium and total Kjeldahl nitrogen,[5] total hydrolyzable nitrogen and total hydrolyzable ammonium nitrogen plus nitrite nitrogen,[3] and total solids.[1]

RESULTS

Sediments. The sediment sample taken from the deepest part (22 m) of Lake Mendota was dark brown or black and changed to a lighter color on exposure to air. It had a uniform consistency, and when mixed with distilled water under an atmosphere of nitrogen gas, it developed a strong hydrogen sulfide odor. When placed in an oxygenated suspension with distilled water, the sediment particles settled more rapidly than would the same sediment in an anaerobic suspension. The aerated suspensions filtered more quickly than the anaerobic suspensions, and there was no color or turbidity in the resulting filtrate.

A difference existed between the two systems because the sediments contained large amounts of ferrous iron. Under aeration, the iron was rapidly oxidized to ferric that precipitated as a flocculent ferric hydroxide. The sediment from the shallow-water area (5 m) of Lake Mendota was lighter brown in color and had a less homogeneous texture than the sediment from the deep water. It contained fragments of calcareous shells and other debris. It had a gritty, sand-like appearance and possessed no distinguishing odor.

The sediment taken from the deep-water area (27 to 32 m) of Trout Lake was medium brown in color and had a homoge-

TABLE I.—Sediment Chemical Composition

Site	Total Kjeldahl Nitrogen (g/kg)*	Total Hydrolyzable Nitrogen (g/kg)*	Total Hydrolyzable Ammonium Nitrogen (g/kg)*	Exchangeable Ammonium Nitrogen (g/kg)*	Nitrate Nitrogen (g/kg)*	Nitrite Nitrogen (g/kg)*	Total Solids (g/kg)*
Lake Mendota							
Deep water	8.7	8.0	2.0	0.4	—	0.0014	169
Shallow water	—	9.1	2.6	0.02	Not detectable	—	195
Trout Lake							
Deep water	19.6	25.6	7.1	0.2 0.4†	—	—	59

* All values calculated on a dry weight basis.
† This value was obtained from an analysis of the sediment from the 27-m Trout Lake composite sample. All remaining data is based on the analyses of the 32-m sample.

neous, jelly-like consistency. No odor could be detected. In comparison to the sediments from Lake Mendota, Trout Lake sediments had a low buffer capacity as a result of the absence of significant amounts of carbonates. In several release studies, particularly the aerobic studies, the pH of the simulated Trout Lake systems decreased steadily from the beginning of the incubation period. At the end of 100 days, the pH in one of the Trout Lake aerobic systems was 3.7, and in the duplicate aerobic study, it had decreased to 3.8. A separate system was set up, in addition to the two aerobic studies of Trout Lake sediment, in which the pH was maintained at approximately 7.7 throughout the incubation period. The addition of excess solid calcium carbonate controlled the higher pH.

The results of the sediment analyses are given in Table I. Previously collected data on sediments taken from the same areas of the lakes by Bortleson [6] are presented in Table II. A comparison between the deep-water sediment from Lake Mendota and that from Trout Lake revealed a threefold difference in total hydrolyzable nitrogen content, with the higher value (25.6 g/kg N) present in the Trout Lake sediment. There was also a threefold difference in the total hydrolyzable ammonium concentration, with the higher value again existing in the Trout Lake sediment (7.1 g/kg N). The total solids content of the Lake Mendota sediment was nearly three times greater than that of the Trout Lake sediment.

Release studies. Release studies (11) were performed on the sediments collected from Lake Mendota and Trout Lake. Four of these studies were duplicate runs, and their results were approximately the same as the original study. Only typical data have been presented in this paper; the complete data are available elsewhere.[7]

Sediment from the deep-water areas of Lake Mendota (25 m) and Trout Lake (32 m) were placed in Pyrex bottles, and both aerobic and anaerobic release studies were begun. The Lake Mendota studies were terminated after 200 days of incubation, whereas the Trout Lake studies lasted 100 days. An additional set of aerobic and anaerobic studies was performed on sedi-

TABLE II.—Chemical Composition of Surface Deposits from Lake Mendota and Trout Lake

Sample Characteristic	Lake Mendota	Trout Lake (South basin)
Date	10/26/66	8/6/66
Water depth (m)	23.3	32.6
Organic matter (%)	14.8	32.2
Carbonate (%)	36.8	1.9
Clastic material (%)	48.4	66.6
Mineral (%)	85.2	68.5
Phosphorous (mg/g)	1.79	7.88
Iron (mg/g)	18.9	66.3
Manganese (mg/g)	1.28	4.02
Calcium (mg/g)	125	3.5
Magnesium (mg/g)	13.4	2.5
Potassium (mg/g)	4.1	1.5

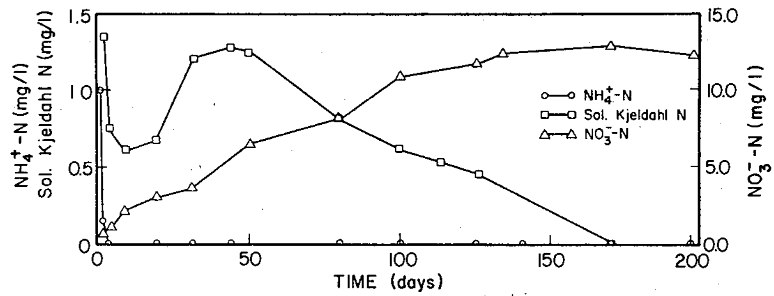

FIGURE 1.—Release of nitrogen compounds from Lake Mendota deep-water sediments under aerobic conditions.

ment taken from the shallow-water area of Lake Mendota (5 m). One of the aerobic studies of the Trout Lake sediment was repeated after it was noticed that with time the pH of the suspension became acidic under aerobic conditions. The repeated study was of much shorter duration (23 days).

Lake Mendota. In the aerobic deepwater release study of Lake Mendota (Figure 1), it can be seen that there was an immediate decrease in the ammonium concentration. Nitrate increased steadily for the first 170 days of the study but leveled off to a constant value of 12.3 mg/l NO_3^-–N by the end of the experiment. Soluble Kjeldahl nitrogen decreased initially but then increased in concentration after the 9th day. It reached a maximum of 1.3 mg/l N at the end of 44 days. It then decreased to the minimum detectable concentration over the remainder of the 200-day study. The soluble Kjeldahl nitrogen probably represented the summation of the dissolved organic nitrogen species and the particulate organic nitrogen that passed through the filters that were used to remove the coarse particulate matter. No nitrite was found throughout the study. The pH was 8.0 to 8.4 throughout the incubation period.

In the Lake Mendota anaerobic study (Figure 2), the concentration of soluble Kjeldahl nitrogen increased gradually over the first 80 days of the experiment. A sharp increase in concentration can be seen at the end of 149 days. The ammonium concentration also gradually increased over the first 80 days of the study and then de-

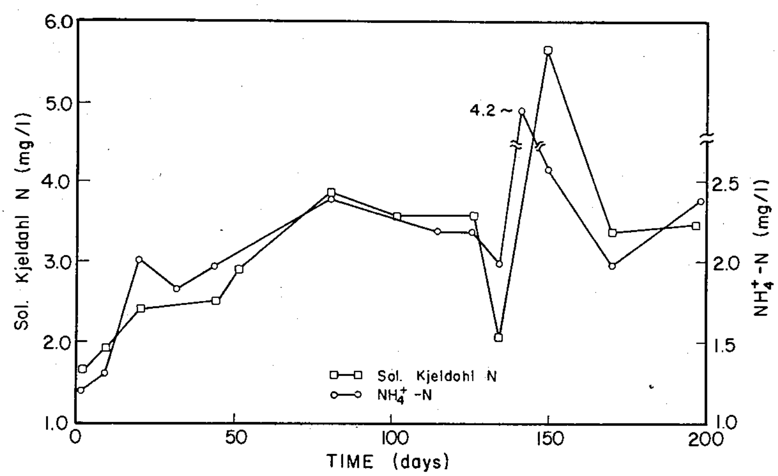

FIGURE 2.—Release of nitrogen compounds from Lake Mendota deep-water sediments under anaerobic conditions.

creased during the following 54 days. Between 134 and 142 days, there was an increase in ammonium concentration to 4.2 mg/l N. The concentration then decreased to 2.0 mg/l N at 170 days. No nitrate or nitrite was detected throughout the entire study. The pH ranged from 8.2 to 8.6 throughout the incubation period.

In the aerobic shallow-water sediment study (Figure 3), nitrate increased sharply after 5 days of incubation, and its concentration was up to 2.0 mg/l N by the end of the 25-day incubation period. Soluble Kjeldahl nitrogen did not change in concentration to any extent over the entire incubation period. Ammonium was detectable on both the 10th and 20th days of the study. A pH range of 7.4 to 7.9 was observed over the 25 days of the experiment. No nitrite was detected in this study.

In the anaerobic experiment using the same shallow-water sediment, no nitrate or nitrite was detected at any time over the 46-day incubation period. Both ammonium and soluble Kjeldahl nitrogen increased at similar rates during this experiment (Figure 4).

The main differences between the aerobic and anaerobic shallow-water sediment studies were (*a*) the presence of nitrate in increasing concentrations under aerobic conditions and its absence under anaerobic conditions; (*b*) the increasing concentration of ammonium in the anaerobic study and its intermittent but negligible appearance in the aerobic system; and (*c*) the increase in concentration of soluble Kjeldahl nitrogen in the anaerobic system, while its change in concentration in the aerobic study was negligible.

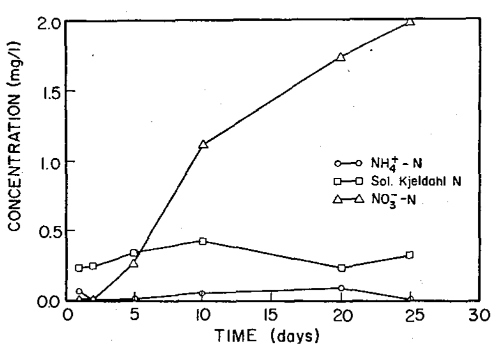

FIGURE 3.—Release of nitrogen compounds from Lake Mendota shallow-water sediments under aerobic conditions.

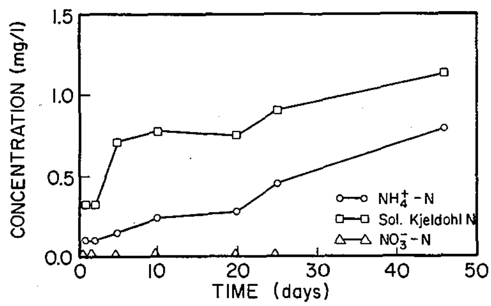

FIGURE 4.—Release of nitrogen compounds from Lake Mendota shallow-water sediments under anaerobic conditions.

Trout Lake. The release studies of deep-water sediment from Trout Lake (32 m) were conducted over a 100-day incubation period. In the aerobic experiment (Figure 5), the pH decreased from 6.6 to 3.7. It was expected that this change in pH would affect the release of nitrogenous compounds from the sediment, because it had an *in situ* pH of 7.0 at the sediment-water interface. Therefore, an additional experiment was performed in which solid calcium carbonate ($CaCO_3$) was added to the sediment suspension in order to maintain a neutral pH range.

In the aerobic, unbuffered system (Figure 5), both the soluble Kjeldahl nitrogen and the ammonium concentrations increased rapidly over the first 50 days of the experiment. They both then decreased, and their concentrations at the end of the study were 0.25 mg/l N (soluble Kjeldahl nitrogen) and 0.3 mg/l N (ammonium) (Run 1). The concentration of nitrate was negligible until after the 70th day of the study. By the 98th day, it had reached a concentration of 1.9 mg/l N (Run 1). No nitrite was detected throughout the study.

In the buffered ($CaCO_3$), aerobic release study of sediment taken from Trout Lake at a later date (27 m), the pH was maintained at 7.7 throughout the incubation period. The results can be seen in

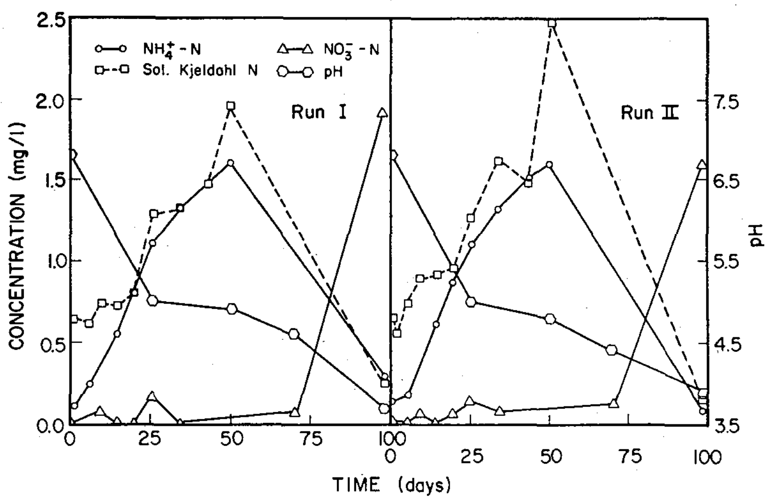

FIGURE 5.—Release of nitrogen compounds from Trout Lake sediments under aerobic, duplicate conditions with no pH control.

Figure 6. Ammonium decreased to an undetectable amount during the first 11 days of incubation. The oxidation of ammonium became evident with a sharp increase in nitrate on the 14th day of the study, from less than 0.1 mg/l N on the 11th day to 1.2 mg/l N on the 23rd day. It showed no sign of leveling off at the end of the experiment. Nitrite was negligible until the 9th day of the study when it suddenly increased to a concentration of 0.19 mg/l N on the 10th day. It subsequently showed a rapid decrease to an undetectable amount after 14 days. The soluble Kjeldahl nitrogen concentration showed an initial increase but generally remained constant at about 0.5 mg/l N for the entire study.

In the comparison of the results of the unbuffered and buffered aerobic experiments, distinct differences were observed. (Note the differences in the time and concentration scales in Figures 5 and 6.) The buffered study was 23 days long, and therefore, the comparison of the two studies takes only that amount of time into consideration. The ammonium concentration increased in the unbuffered (acidic) studies but decreased in the buffered system. There was not a rapid increase in nitrate in the unbuffered suspensions in the first 23 days, but a rapid increase did occur in the buffered system on the 14th day of the study. The soluble Kjeldahl nitrogen concentration increased in the unbuffered systems over the first 24 days but remained fairly constant in the buffered system. Ammonium concentrations were much higher in the unbuffered studies than in the buffered system. Nitrite was detected only in the buffered system.

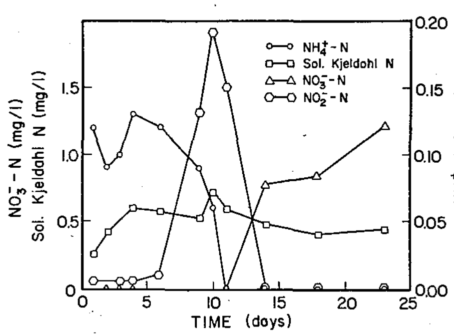

FIGURE 6.—Release of nitrogen compounds from Trout Lake sediments under aerobic conditions and pH of 7.7, controlled by the addition of excess solid calcium carbonate.

In the anaerobic release studies using Trout Lake sediment, both ammonium and soluble Kjeldahl nitrogen increased at similar rates throughout the 100-day incubation

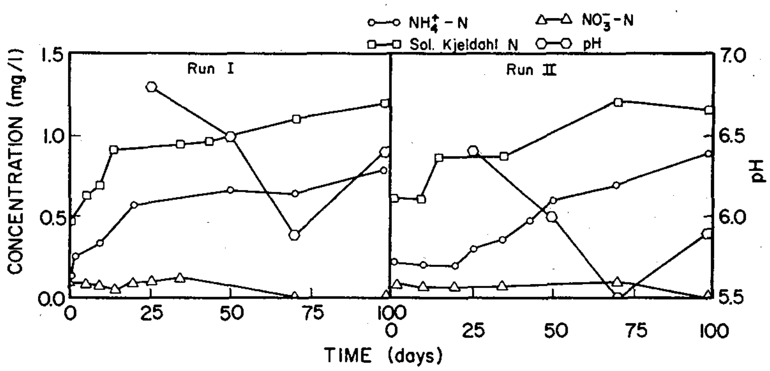

FIGURE 7.—Release of nitrogen compounds from Trout Lake sediments under anaerobic, duplicate conditions with no pH control.

period. The rate of increase in concentration was higher over the first 15 to 20 days of the study than it was in the remainder of the experiment (Figure 7) (Run 1).

The results of the anaerobic study differed greatly from those obtained in the buffered aerobic experiment (Figures 6 and 7). Ammonium increased in the anaerobic study while it decreased in the buffered aerobic experiment. The concentration of soluble Kjeldahl nitrogen increased in the anaerobic studies but remained fairly constant at lower concentrations in the aerobic experiment. Nitrate was measured in both studies but increased to much higher concentrations in the aerobic system. The nitrate in the anaerobic experiment was likely an analytical error as a result of the failure of the blank to completely compensate for the color in the water. Nitrite was not detected in the anaerobic studies, whereas it was measured at a maximum concentration of 0.19 mg/l N in the buffered aerobic study.

DISCUSSION

The individual nitrate, nitrite, and ammonium nitrogen values for each sample taken from the release studies were added together (as nitrogen) and plotted against time. The slope of each "summation curve" was then estimated and listed in Table III. The time interval over which each slope was estimated is also given in Table III.

An additional table was compiled to account for the differences in nitrogen content as well as total solids content of the individual sediment samples used in each of the release studies. In order to make these calculations, the individual values for total dissolved inorganic nitrogen, that is nitrite plus nitrate plus ammonium, were recorded from the summation curves every 20 days for the first 100 days of each release experiment. In the long-term studies of 200 days, additional values were taken on the 150th and 200th day of each study. Each of these summation values was divided by the total hydrolyzable nitrogen concentration of the original sediment (dry weight basis). The calculations were then converted to percent nitrogen released from the sediment and are presented in this way in Table IV.

Lake Mendota sediment from both deep- and shallow-water areas released large amounts of dissolved inorganic nitrogen, mainly in the form of nitrate, under aerobic conditions. In the first 20 days of each aerobic study (duplicates), the deep-water sediment from Lake Mendota released more than twice the amount of dissolved inorganic nitrogen as did the shallow-water sediment (Table IV). Compared to the aerobic Lake Mendota studies, there was less dissolved inorganic nitrogen released from either the deep- or shallow-water sediment of Lake Mendota under anaerobic

TABLE III.—Rate of Nitrogen Release from Lake Sediments

Location	Sediment	
	Aerobic	Anaerobic
Inorganic nitrogen		
Lake Mendota		
Deep water (mg/day/l)	0.09	<0.01
Days*	25–125	0–198
Shallow water (mg/day/l)	0.09	0.02
Days	1–20	2–25
Trout Lake		
Deep-water (mg/day/l)	0.04† <0.01† 0.07‡	<0.01
Days	5–34§ 45–95‖ 10–23#	15–75
Kjeldahl nitrogen		
Lake Mendota		
Deep water (mg/day/l)	<0.01	<0.01
Days	0–198	0–198
Shallow water (mg/day/l)	<0.01	<0.01
Days	0–25	5–25
Trout Lake		
Deep water (mg/day/l)	0.04 −0.02	<0.01
Days	0–34§ 35–95**	15–75

* Days of incubation period over which the slope of the curve was measured.
† No calcium carbonate added.
‡ Calcium carbonate added.
§ Period of 0.04 mg/day/l release.
‖ Period of <0.01 mg/day/l release.
Period of 0.07 mg/day/l release.
** Period of negative release.

conditions in the first 20 days of the incubation period. The release of dissolved inorganic nitrogen from the deep-water sediment was nearly 10 times greater than that of the shallow-water sediment after the first 20 days of the anaerobic study. At the end of 200 days of incubation under aerobic conditions, the Lake Mendota deep-water sediment released 44 percent of its original hydrolyzable nitrogen in the form of dissolved inorganic nitrogen. The anaerobic sediment (deep-water), in contrast, showed only a release of 6.2 percent of its original hydrolyzable nitrogen. Trout Lake released less dissolved inorganic nitrogen in both its aerobic and anaerobic experiments than did Lake Mendota.

After 20 days of incubation, under both aerobic and anaerobic conditions, Lake Mendota deep-water sediment released 4.5 times as much dissolved inorganic nitrogen as Trout Lake sediment. After 100 days of incubation, Lake Mendota sediment released six times more dissolved inorganic nitrogen than that of Trout Lake under aerobic conditions and four times more dissolved inorganic nitrogen under anaerobic conditions. In the additional aerobic study in which $CaCO_3$ was added to the Trout Lake sediment suspension to control the pH, Lake Mendota still released four times more dissolved inorganic nitrogen as Trout Lake sediment over the first 20 days of the aerobic experiments.

It is interesting to note that Trout Lake sediments (with the higher concentrations of available nitrogen) under similar aerobic and anaerobic conditions released considerably less inorganic nitrogen than did the Lake Mendota deep-water sediments. The reasons for this difference are not readily apparent but are probably related to the fact that the Lake Mendota sediments provided a more optimum environment for the bacteria that cause the solu-

TABLE IV.—Percent Nitrogen Released from Sediment

Days	Lake Mendota (deep-water sediment)		Trout Lake (deep-water sediment)	
	Aerobic	Anaerobic	Aerobic	Anaerobic
20	10.4	5.9	2.3	1.3
40	16.6	5.9	3.8	1.4
60	20.7	6.2	4.3	1.8
80	23.4	6.5	4.7	2.0
100	30.5	8.3	5.1	2.2
150	39.4	6.2	—	—
200	44.1	6.2	—	—
	(shallow-water sediment)		(deep-water sediment, $CaCO_3$ added)	
20	4.1	0.6	2.6	—
40	—	1.1	—	—

bilization and nitrification reactions. Additional chemical characteristics of the Lake Mendota and Trout Lake sediments are presented in Table II. Examination of this data does not show any obvious reasons for the differences in nitrogen release rates. It is also possible that the forms of the organic nitrogen in the two lake sediments are markedly different possibly because of the difference in photosynthetic productivity that takes place in both lakes. Further studies would be necessary on the forms of organic nitrogen present in the sediments and the bacterial population that develops in these experimental systems in order that the mechanisms controlling the rates of mineralization of nitrogen compounds in various types of lake sediments can be determined.

It is expected that the high iron concentration of the Trout Lake sediment had an effect on pH through protolysis reaction under aerobic conditions. The concentration of iron in Trout Lake sediment (about 2 cm depth) was found to be approximately 66 g/kg.[6] In the study in which solid $CaCO_3$ was used to adjust and maintain the pH of the aerobic sediment suspension, the release of nitrogen followed a completely different pattern from that of the unbuffered aerobic experiment. After the system came to equilibrium with the $CaCO_3$ (about 10 days), there was a release of nitrate as in the Lake Mendota studies but not until the disappearance of nitrite and ammonium from solution. The formation of nitrite and its subsequent rapid disappearance was not seen in any of the other release studies on Trout Lake or Lake Mendota sediment. The appearance of nitrite was also experienced in studies of the regeneration of nutrients from dead plankton material of marine origin.[8] After the formation of nitrate in the carbonate-adjusted study, the release pattern proceeded in a manner similar to that of the aerobic Lake Mendota studies.

It should be emphasized that the temperature of the release experiments (24 ± 2°C) was considerably higher than that of the hypolimnetic waters at either of the sediment-water boundaries of Lake Mendota or Trout Lake. The quantity of nitrogen released from the sediment was no doubt increased greatly by the higher temperature of the laboratory suspension.

The concentration of dissolved oxygen was probably the most important chemical parameter affecting the amount and species of nitrogen released from a sediment. Over a 200-day period, the amount of dissolved inorganic nitrogen released from Lake Mendota sediment under aerobic conditions was seven times greater than that released from the same sediment under anaerobic conditions. In the Trout Lake studies, the concentration of dissolved inorganic nitrogen after 100 days of incubation was 2.3 times higher in the aerobic than in the anaerobic studies.

The chemical composition of a lake sediment could affect the amount of nitrogen that would be released from it. The buffering ability of Lake Mendota sediment can be partially attributed to its natural carbonate content. An average of 36.8 percent carbonate content in the surface deposits of Lake Mendota was determined.[6] In contrast, the analyses of Trout Lake sediment showed a carbonate content of 1.9 percent (Table II).[6]

Sawyer *et al.*[9] investigated the release of nutrients from lake sediments using stationary sediment-water systems in the laboratory. Lake Mendota sediment release was studied, and it was found that over a period of 100 days, there was a release of 0.6 percent of the total nitrogen from the sediment to the overlying water. The experiments were performed in the dark at room temperature.[9]

In the current study, the sediments were completely mixed in order to eliminate the effect of mixing processes in the sediments and water on the rate of release of nitrogen compounds.

Under somewhat similar conditions except for mixing, the Lake Mendota sediment showed a release of 30.5 percent of its total hydrolyzable nitrogen content in the form of dissolved inorganic nitrogen in the same amount of time, 100 days, as in the previous study.[9] This was more than 50 times the nitrogen release found on sediments from the same area of Lake Mendota but studied under aerobic, quiescent conditions. In the release studies of this investigation, the same conditions of temperature and light were used, but the sediment:water ratio was much higher in earlier experiments;[9] 1 l of mud in 3 to 4 l of water as compared with 0.5 l of mud in 20 l of water used in this study.

Although the degree to which mixing processes in the water and sediments influence the rate of release of nitrogen compounds is poorly understood, it is reasonable to propose that the mixing that takes place in quiescent laboratory conditions is less than that which actually takes place in the lake. It is certain that with the exception of sediments from areas of shallow water that are subjected to intense wave action and/or organism stirring, the completely mixed laboratory conditions used in this study exceeded the normal mixing that takes place in natural water sediments. Therefore, as a first approximation taking into account the difficulty of attempting to translate laboratory experiments to field conditions, the rates of release of nitrogenous compounds from lake sediments in Lake Mendota and for several other lakes would be expected to be less than those measured in this study but somewhat greater than those reported by earlier researchers.[9] Because lake sediments, in general, tend to accumulate large concentrations of nitrogen compounds, especially in deep-water areas, and this study has shown that a substantial fraction of these nitrogen compounds can be released under completely mixed aerobic conditions, it is reasonable to propose that one of the primary reasons why lake sediments act as a nitrogen sink is because of the relatively poor mixing that takes place within the sediments and between sediments and the overlying waters.

One reviewer reported on the relative importance of hydrodynamic factors in influencing sediment water exchange.[9, 10] This review should be examined for further discussion of the amount of mixing that takes place in lake sediments and the factors that influence this mixing.

Another factor that tends to inhibit the release of nitrogen compounds from lake sediments in eutrophic lakes is the anaerobic conditions that are present in the sediments throughout the year and hypolimnion during periods of thermal stratification.

The low organic nitrogen content and slow rates of nitrogen release from the shallow-water sediment of Lake Mendota probably reflected the much higher energy (geological sense) of the nearshore environment. This energy promotes organic nitrogen solubilization and mineralization resulting in a relatively refractory low concentration of organic nitrogen in the sample.

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REFERENCES

1. "Standard Methods for the Examination of Water and Wastewater." 12th Ed., Amer. Pub. Health Assn., New York, N. Y. (1965).
2. Bremner, J. M., and Keeney, D. R., "Steam Distillation Methods for Determination of Ammonium, Nitrate and Nitrite." *Anal. Chim. Acta*, **32**, 485 (1965).
3. Bremner, J. M., "Organic Forms of Nitrogen." In "Methods of Soil Analysis," C. A. Black [Ed.]. Amer. Soc. Agronomy, Madison, Wis. (1965).
4. Jenkins, D., and Medsker, L. L., "Brucine Method for Determination of Nitrate in

Oceans, Estuarine and Fresh Waters." *Anal. Chem.*, **36**, 610 (1964).

5. Jackson, M. L., "Soil Chemical Analysis." Prentice-Hall, Inc., Englewood Cliffs, N. J. (1958).
6. Bortleson, G. C., "Chemistry of Lake Sediment Cores." M.S. thesis, Water Chem. Program, Univ. of Wisconsin, Madison (1968).
7. Austin, E. R., "The Release of Nitrogenous Compounds from Lake Sediments." M.S. thesis, Water Chem. Program, Univ. of Wisconsin, Madison (1970).
8. Von Brand, T., *et al.*, "The Experimental Decomposition and Regeneration of Nitrogenous Organic Matter in Sea Water." Biol. Bull., Marine Biol. Lab., Woods Hole, Mass. (1937).
9. Sawyer, C. N., *et al.*, "An Investigation of the Odor Nuisances Occurring in the Madison Lakes, Particularly Monona, Waubesa, and Kegonsa from July 1942–44." Rept. of Governor's Comm., Madison, Wis. (1945).
10. Lee, G. F., "Factors Affecting the Transfer of Materials Between Water and Sediments." Lit. Rev., Eutrophication Info. Program, Univ. of Wisconsin, Madison (1970).