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Sulphur mineralization kinetics as influenced by soil properties

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Abstract A 10-week laboratory study, using an open incubation technique, was carried out to determine net sulphur (S) mineralization potentials of soil samples obtained from some representative soils in Tuscany, Italy. The time-course of organic S mineralization in the soils was analyzed by fitting the experimental values to three kinetic models (first-order, first-order E, zero-order). The first-order model was found to be the most suitable because it provided the best fit to the experimental data and for its simplicity. Potentially mineralized S (S_0) values ranged from a minimum of 13.6 to a maximum of 50.7 mg kg^{-1} soil and the mineralization rate k varied from 0.111 to 0.615 week^{-1}. It was also positively related to organic C, N, and S, protease, arylsulphatase, and dehydrogenase activities. The mineralization rate did not show any significant relationship with soil properties.

Keywords Soil properties · Sulphur mineralization · Kinetic models · Arylsulphatase · Protease · Dehydrogenase

Introduction

Sulphur has long been recognized as essential for plant and animal growth, having an important role in many reactions in every living cell.

In the past, fertilizers and atmospheric inputs supplied the soils with adequate amounts of this element, and only in recent years have deficiencies of sulphur become common. The significant reductions in SO_2 emissions as a result of national policies to reduce concentrations of primary air pollutants, as well as the use of high-analysis low-S fertilizers and the declining use of S-containing fungicides have resulted in a negative sulphur balance becoming widespread throughout the world (Scherer 2001). Coupled with the reductions in the supply of S to soils and plants, the decline in soil fertility is largely a result of the decline of soil organic matter. Inorganic S is generally much less abundant than organically bound S in most agricultural soils (Bohn et al. 1986). Because organic S compounds are unavailable to plants, they must be converted by biochemical (hydrolyzation of sulphate esters by different sulphatases) or microbiological activity (mineralization of C-bonded S) to inorganic SO_4^{2-} before plant uptake (Castellano and Dick 1991). In many instances, the mineralization of S from soil organic matter may potentially be the major supply of S for plant nutrition. In assessing potential soil productivity, it is, therefore, of value to know the rate of mineralization of organic S and equilibrium levels that can be maintained in different soils.

Mineralization and immobilization of S occur concurrently (Ghani et al. 1993), and the release or incorporation of inorganic sulphate is, thus, a net result of several processes. The factors affecting the balance between these two reactions are not clearly defined.

Variables affecting microbial and enzymatic activity, such as temperature, moisture, pH, substrate availability, as well as C/S ratio of soil organic matter, provide a rough guide to the potential to release SO_4^{2-}. Their influence is reported by Scherer (2001) in a wide review on S in crop production. However, very limited and controversial information is available concerning the influence of soil properties on the extent of S mineralization.

Williams (1967) indicated that neither the amounts nor the patterns of S mineralized in 17 Australian soils were closely related to soil types, total amount of C, N, S, the ratios of C/S, N/S, C/N, or to soil pH. Similarly, Freney (1986) reported that S release from soils is not directly related to soil type, while Tabatabai and Chae (1991) reported that soil types have marked effects on the mineralizable S pool (S_0) and first-order rate constants (k). In incubation experiments on five Danish soils, the rate

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of SO_4^{2-}–S release was not significantly correlated with total organic C, N, and S, but was with the microbial activity, as measured by ATP content (Eriksen et al. 1995). Tabatabai and Al-Khafaji (1980) reported that soil organic S is depleted faster than organic N, although the two processes showed a linear relationship. They also found that the rates of S mineralization and the ratio of N/S mineralized in incubated soils were respectively correlated, negatively and positively, with soil pH. In incubation experiments, a poor relationship was reported between arylsulphatase and S mineralization owing to enzyme activity repression by SO_4^{2-} and/or the inhibiting effect of soluble orthophosphate in the soil (Knauff and Scherer 1999).

The mathematical description of the dynamics of S mineralization is of great interest in the prediction of the ability of soils to supply potentially mineralizable organic S and, more generally, for organic matter balance. Among the researchers that used different kinetic models for describing net S mineralization, Ellert and Bettany (1988, 1992), in two studies on Canadian soils, used a first-order, a zero-order, and a consecutive first-order model with identical rate coefficients, while Pirela and Tabatabai (1988), on Iowa and Chilean soils, and Ghani et al. (1991), on New Zealand soils, used both an exponential equation and a reciprocal-plot technique. Zhou et al. (1999) found a first model and a Gompertz model as best describing S mineralization in upland soils of North China. Consequently, it is important to select a model that will simulate the behavior of S mineralization for a wide range of soils.

The objectives of the present study were: 1) to determine, by an open incubation technique, the S net mineralization of some representative agricultural soils in Tuscany (Italy) with widely differing properties; 2) to compare the effectiveness of some commonly used models for describing rates and amounts of soil S mineralization; and 3) to evaluate the relationship between soil properties and S mineralization parameters derived from the best fit kinetic model.

Materials and methods

Six agricultural soils (0–15 cm) from various areas of Tuscany were examined. Samples of field-moist soil, consisting of 20 cores measuring 2.5 cm diameter×10 cm, were air-dried, crushed to pass a 2-mm sieve to remove large residue fragments, and analyzed for the major physico-chemical properties (Table 1). Texture was determined by the pipette method and pH was determined in water (soil-to-water ratio of 1:2.5) potentiometrically. Nitrogen determination was made by the Kjeldahl procedure after acid digestion (Bremner and Mulvaney 1982). Organic carbon and sulphur were determined, after removing carbonate C (Nelson and Sommers 1982) and sulphate S (Tabatabai 1982), by dry combustion with the automatic carbon and sulphur determinator (induction furnace) 900 CS, Eltra.

On the field-moist soil, arylsulphatase, dehydrogenase and protease were determined, as reported by Tabatabai and Bremner (1970), Casida et al. (1964), and Ladd and Butler (1972), respectively.

The Tabatabai and Al-Khafaji (1980) procedure for S mineralization study was followed. Fifty grams (on an oven-dry basis) of field-moist soil and an equal weight of coarse glass beads were mixed and transferred into a leaching tube. The soil–glass beads mixture was retained by means of a glass wool pad and leached with 250 ml of 0.01 M KCl to remove the initial mineral SO_4^{2-}–S. The tubes (5 cm in diameter and 20 cm in length) were incubated at 20±1°C and moistened to 60% of the maximum water-holding capacity of soils. The leaching procedure was repeated every 14 days for 10 weeks. The moisture content was adjusted by weighing the tubes every 4 days. Leaching the soils periodically during incubation may be similar to the removal of S by plants and may represent the leaching process of this element in soils under moist conditions (Tabatabai and Al-Khafaji 1980).

The leachate was filtered on a Whatman No. 42 paper disc and then analyzed for SO_4^{2-}–S by ion chromatography (DIONEX DX 100), after filtration through a 0.2-µm glass fiber. The IC column was an Ionpac AS4A (4×250 mm). The composition of the eluent was 1.8 mM sodium carbonate/1.7 mM sodium bicarbonate. All incubations were carried out in duplicate.

A non-linear least-square regression analysis was used to calculate parameters from cumulative data of S mineralization. The mean coefficient of variation was always lower than 3%.

Table 1 Some properties of the tested soils

Soil no.	Soil classification	Sand (%)	Silt (%)	Clay (%)	pH	Org. C (%)	Org. N (%)	Org. S (%)	C/N	C/S	N/S	Arylsulphatase activity (µg *p*-nitrophenol $g^{-1}h^{-1}$)	Protease activity (µg tyrosine $g^{-1}2\ h^{-1}$)	Dehydrogenase activity (µg triphenylformazan $g^{-1}h^{-1}$)
1	Xerorthent	20.4	53.3	26.3	6.9	0.79	0.21	0.04	3.8	19.8	5.2	34.3	12.4	2.37
2	Xerorthent	61.8	21.5	16.7	7.6	1.04	0.07	0.02	14.8	52.0	3.5	18.4	11.9	2.03
3	Fluvaquent	43.8	40.2	16.0	6.2	11.10	0.80	0.42	13.9	26.4	1.9	82.9	142.6	9.79
4	Xerochrept	72.0	19.6	8.4	4.7	1.83	0.18	0.04	10.2	45.8	4.5	0.1	50.6	2.07
5	Xeropsamment	93.9	2.6	3.5	6.0	0.57	0.07	0.02	8.1	28.5	3.5	1.6	41.4	0.19
6	Xeropsamment	88.4	2.6	9.0	7.9	0.16	0.03	0.02	5.3	8.0	1.5	2.3	15.1	0.06

Mineralization patterns

The following kinetic models were used to describe soil S mineralization:

First-order (F0): $S_t = S_0\,(1-e^{-kt})$ (Stanford and Smith 1972)
First-order E (F0E): $S_t = S_0(1-e^{-kt}) + Se$ (Jones 1984)
Zero-order (Z0): S_t=kt+intercept (Ellert and Bettany 1988)

Where, S_t is the cumulative amount of S mineralized after time t and it is expressed as mg kg^{-1} (dependent variable); t is the time from start of incubation when t=0, and it is expressed in weeks (independent variable); S_0 is the potentially mineralizable organic S (amount present at t=0), and it is expressed as mg kg^{-1}; k is the proportionality constant specific to the model, and it is expressed in week^{-1}; S_e is the easily mineralizable fraction of S, and it is expressed as mg kg^{-1}of soil.

Results and discussion

As shown in Table 2, the SO_4^{2-}–S that evolved during the incubation period varied considerably among the soils, ranging from 10.3 for soil 2, to 41.1 mg kg^{-1} for soil 3 (mean, 21.1 mg kg^{-1}). Expressed as percentages of total S in the soils studied, the cumulative amounts of S mineralized ranged from 1.0 for soil 3, to 8.9% for soil 6 (mean 5.5%).

The net amounts of S mineralized and percentages of organic S mineralized during the 10-week incubation period were slightly higher than the values reported previously for Iowa (S mineralized: average 9.7 mg kg^{-1}; percentage of organic S mineralized: average 4.1%) and Chilean soils (average 13.2 mg kg^{-1} and 3.1%), incubated at the same temperature of 20°C for 14 weeks (Pirela and Tabatabai 1988). The results were more similar to those reported previously by Zhou et al. (1999) for 12 upland soils of north China, but incubated for 28 weeks at 30°C (mean 25.9 mg kg^{-1}, percentage of organic S mineralized, mean 5%). This suggests a higher microbiological activity in our soils.

Table 2 Amount and percentage of organic S mineralized in soils incubated at 20°C for 10 weeks and quality-of-fit statistics (R^2) for comparing kinetic models used to represent soil S mineralization (F0: first-order model; F0E: first-order)

Soil no.	S_t (mg kg^{-1}soil)	(%)-S	F0	F0E	Z0
1	22.2	5.1	0.999	0.999	0.932
2	10.3	5.8	0.998	0.998	0.951
3	41.1	1.0	0.998	0.998	0.949
4	14.1	3.5	1.000	1.000	0.975
5	20.5	8.8	0.999	0.999	0.736
6	18.6	8.9	0.999	0.999	0.656
Avg	21.1	5.5			

St is the SO_4^{2-}–S mineralized after 10 weeks incubation
(%)-S is the percentage of organic S mineralized after 10 weeks incubation

As shown in Fig. 1, cumulative mineralized S showed a curvilinear relationship with time. Soils 5 and 6, which have a high content of sand and low dehydrogenase activity, showed a rapid release of SO_4^{2-}–S during the first 2 weeks followed by a slower, almost linear rate of release for the remainder of the incubation. This trend, less evident for soils 2 and 4, which showed a more constant curvilinear trend, was similar to that described by Zhou et al. (1999) for the soils of north China and to that found by Pirela and Tabatabai (1988) for some of the Iowa and Chilean soils. From some of the soils examined by these authors, the relationship between cumulative S and incubation time was linear. In our study, although the release of S was not perfectly linear, soils 1 and 3, which had particularly provided high clay content and enzyme activities (Table 1), showed a less reduction in the rate of S-mineralization with time.

The time-course of organic S mineralization in the soil was analyzed by fitting the experimental values to some kinetics model used by Ellert and Bettany (1988) for describing net sulphur mineralization. From a visual inspection of the figures, three models of S mineralization were chosen. The equations were tested for each of the six soils examined in the incubation trials, and the coefficients of determination (R^2) used to compare the different kinetic models are reported in Table 2.

The zero-order (Z0) model described the patterns less well than the F0 and F0E models, which even showed the same R^2 value for each soil sample. The first-order E model, originally proposed by Jones (1984) for N mineralization and applied by Riffaldi et al. (1996) to C mineralization in 14 agricultural soils, offered as good a description of the cumulative SO_4^{2-}–S release as the simple first-order model (Stanford and Smith 1972). Jones (1984) used the first-order model and added a parameter able to define a separate

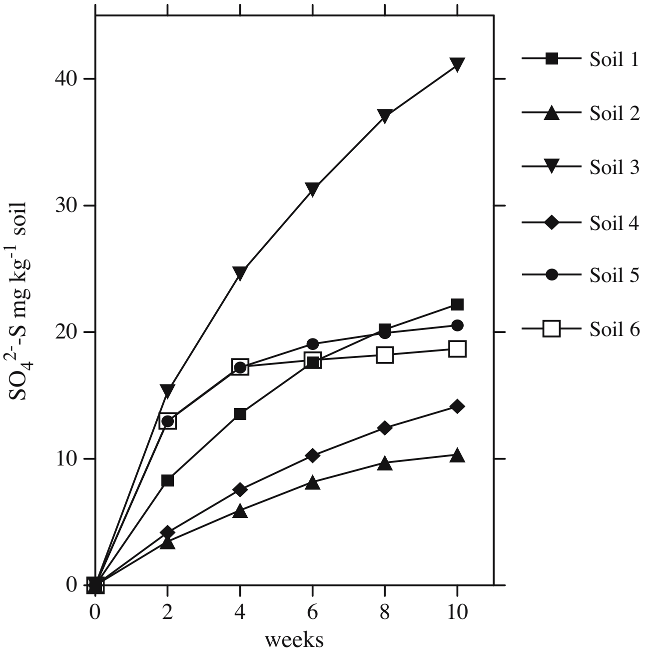

Fig. 1 Cumulative SO_4^{2-}–S production by the soils

Table 3 Parameter estimates according to the first-order model for S soil net mineralization

Soil no.	S_0 (mg kg^{-1} soil)	k (week^{-1})	S_0k (mg kg^{-1} soil week^{-1})	$1/k$ (week)
1	26.1 (±0.3)[a]	0.190 (±0.005)	4.9	5.3
2	13.6 (±0.6)	0.150 (±0.013)	2.0	6.7
3	50.7 (±2.1)	0.167 (±0.013)	8.5	6.0
4	21.2 (±0.1)	0.111 (±0.001)	2.3	9.0
5	20.4 (±0.2)	0.487 (±0.019)	9.9	2.0
6	18.5 (±0.2)	0.615 (±0.027)	11.4	1.6

[a] Standard error

S_0 is the potentially mineralizable organic S

k is the proportionality constant specific to the model

S_0k is the initial potential rate of S mineralization

$1/k$ is the turnover times for S pools

pool of easily decomposable substrate producing a mineralization flush during the first incubation interval. However, since the contribution from the rapidly mineralizing pool did not extend beyond the first incubation period, the additional complexity of this model does not justify it being chosen for the tested materials. For this reason Table 3 shows the parameters calculated only according to the first-order model.

Potentially mineralizable S (S_0) values ranged from a minimum of 13.6 for soil 2 to a maximum of 50.7 mg kg^{-1} for soil 3. These values were within the ranges reported previously by Pirela and Tabatabai (1988) for the potentially mineralizable S pool (S_0) calculated by using an exponential equation ranging from 5 to 44 mg kg^{-1} for the Iowa soils and from 10 to 25 mg kg^{-1} for the Chilean soils, respectively. Subsequently, Tabatabai and Chae (1991) found that only three out of five investigated unamended soils fitted the first-order model and reported values of S_0 equal to 49, 61, and 96 mg kg^{-1} soil, which were generally higher than the values reported in the present study.

Rate constants for S mineralization differed among the soils, with values falling within a relatively broad range (Table 3), from 0.111 for soil 4 to 0.615 week^{-1} for soil 6. The relatively broad range of k values among the soils suggests that microorganisms metabolized organic S compounds which were different or had a different degree of availability. Furthermore, soil microflora may have differences in the use of S compounds, with the autotrophic chemolithotrophic bacteria, belonging to the genus *Thiobacillus*, being the most efficient group (Scherer 2001). About the same order of magnitude of k values, from 0.25 to 1.27 and from 0.48 to 1.00 mg kg^{-1} week^{-1} for Iowa soils and Chilean soils, respectively, was reported by Pirela and Tabatabai (1988). In a later study, however, Tabatabai and Chae (1991) reported for three soils that fitted the first-order model, values ranging from 0.02 to 0.045 wk^{-1}, which were about ten times lower than those observed in our study (Table 3).

Turnover times for S pools, calculated from the rate constants (1/k), were relatively rapid and ranged from about 2 to 9 weeks among soil types. Table 3 also shows the data relative to the initial potential rate of S mineralization (S_0k) which, as reported by Stanford and Smith (1972) for C mineralization, can be a more precise estimate than the two parameters (S_0 and k) when evaluated separately, as the potentially mineralizable substrate and the mineralization rate are interdependent. The S_0k values varied considerably, from 2.0 in soil 2, with a very low k value, to 11.4 in soil 6, showing the highest k value.

Table 4 shows the significant linear correlations between S mineralization parameters and soil characteristics. No relationship was observed between S mineralization parameters and

Table 4 Significant correlation coefficient between S-mineralization parameters of the first-order model (Table 3) and soil characteristics (*P<0.05, **P<0.01)

Parameter	Soil characteristics					
	Org. C	Org. N	Org. S	Protease activity	Dehydrogenase activity	Arylsulphatase activity
S_t	0.889*	0.907*	0.919**	0.863*	0.839*	0.863*
% S	–	–	–	–	−0.837*	–
S_0	0.945**	0.978**	0.963**	0.917**	0.931**	0.909*
k	–	–	–	–	–	–
S_0k	–	–	–	–	–	–

For symbols, see Tables 2 and 3

texture or pH, whereas, Tabatabai and Al-Khafaji (1980) found a significant negative correlation between S mineralized, expressed as a percentage of total S, and pH.

Similarly, ratios of initial C, N, and S analyses (C:N, C:S, and N:S) were not helpful in predicting relative amounts of S mineralized after 10 weeks incubation. Also Williams (1967) did not find such relationships in Australian soils. Nevertheless, results reported for other incubation studies seem to suggest that large amounts of S are generally mineralized from soils with low C/N/S ratios (Bettany et al. 1974, Kowalenko and Lowe 1975).

Both the cumulative amounts of S mineralized and the potentially mineralizable S were strongly related to each other (r=0.969, P<0.01), and both were significantly correlated, at P<0.01, with organic C, N, and S contents. This contradicts the findings of Tabatabai and Al-Khafaji (1980) and Pirela and Tabatabai (1988), who could not find similar correlations. Eriksen et al. (1995), in a study on Danish soils, also found no correlation of net mineralization of S with total organic S of soils. The positive correlations found between the cumulative amount of SO_4^{2-}–S mineralized or the potentially mineralizable S (S_0), and the content of organic C, N, and S in soils suggest that the content of nutritive elements is an important controlling factor in the S mineralization process in soil.

The effect of nutrients on S mineralization was further emphasized by the significant positive correlations between either S_0 (P<0.01) and cumulative SO_4^{2-}–S (P<0.05) with arylsulphatase and protease activities, involved in soil S and N metabolism, respectively. A positive correlation was also obtained between the S mineralization parameters and dehydrogenase activity which, as it only occurs within living cells, is a valuable indicator of the overall microbial activity in soils (Nannipieri et al. 1990). This agrees with the findings of Eriksen et al. (1995), who reported that, although net mineralization of S was not correlated with total organic S, there was a positive correlation between S mineralization and microbial activity, as measured by ATP content in the soils. When the differences in total organic S were taken into account (S_0 divided by organic S content, Table 2), an inverse relationship between the normalized S_0 and dehydrogenase activity was found. A possible explanation is that the source of the released S may have differed among the studied soils, that is, the lowest S_0/organic S values occurred in soils with the highest levels of relatively recalcitrant organic S. Indeed, cumulative SO_4^{2-}–S mineralized and percentage of mineralized organic S were negatively related (r=−0.45), although not significantly.

According to Eriksen et al. (1998), the positive correlations between S mineralization and arylsulphatase activity and dehydrogenase activity—the latter being an indicator of microbiological activity—could testify that organic S in soil is converted to inorganic SO_4^{2-} by biochemical and microbiological mineralization.

There were no significant correlations between k and potentially mineralizable S_0, indicating that differences in k values among soils cannot be attributed to differences in the relative size of the S pool (S_0).

The parameter S_0k also showed a lack of significant correlation with soil properties. Consequently, it cannot be considered a useful index for delineating the effects of soil properties on S mineralization in soil organic matter.

On the basis of the results obtained under the present study conditions, it can be concluded that the net S-mineralization parameters, calculated according to the first-order model, varied among soils. In particular, potentially mineralizable S_0 was significantly related to organic C, N, and S, to hydrolytic enzymes, protease, and arylsulphatase, as well as to dehydrogenase.

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