A simple method for the determination of nitrate in potassium chloride extracts from forest soils

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Abstract

We developed a simple method to determine NO_3^--N in KCl extracts from forest soils. The measurement principle of the method is based on the difference of the spectral adsorption properties between dissolved organic carbon (DOC) and nitrate. In this method, NO_3^--N concentration was obtained by measuring the absorbance of KCl extracts at 220nm and 260nm wavelengths. We term this method as the UV method. We measured NO_3^--N concentration of fifty six KCl extracts from six forest soils in central and western Japan by using the UV method. The results were highly correlated to those by the automated Cd reduction method. It suggests that the UV method is a reliable method to determine NO_3^--N in KCl extracts. It is the most simple procedure to determine NO_3^--N in KCl extracts from forest soils.

Key Words

Nitrate, rapid determination, forest soils, available nitrogen, nitrogen mineralization.

Introduction

Nitrogen is a most important nutrient for higher plants, so available nitrogen and nitrogen mineralization potential of soils are indicators for soil fertility. This nitrogen is evaluated by the amounts of NH_4^+-N and NO_3^--N that are extracted with KCl solution from soils. For determining NO_3^--N, expensive equipments or time consuming methods such as flow injection (automated Cd reduction methods) and Kjeldahl determination method are needed. So, several rapid methods have been devised for NO_3^--N determination (Cataldo *et al.* 1975; Kanno *et al.*1968; Norman and Stucki 1981; Sakata 2000; Yang *et al.* 1998). Yang *et al.* (1998) developed a simple method by applying the salicylate method (Cataldo *et al.* 1975;Kanno *et al.*1968). This method is very simple but it needs some steps such as reagent adding and heating.

The aim of this study was to develop more simple method for determining NO_3^--N in KCl extracts. NO_3^--N could be determined by a UV absorption method in solution where the dissolved organic carbon (DOC) concentration is low. The UV absorption method, however, cannot be applied for the determination of NO_3^--N in KCl extracts from forest soils because these solutions usually contain considerable amount of DOC. DOC absorbs light at all wavelengths between 200 nm and 300nm. The absorbance of DOC gradually decreases from 200 nm to 300nm. On the other hand, NO_3^--N also absorbs UV light at less than 250 nm wavelength. Based on the difference of UV absorption properties between DOC and NO_3-$^-$N, we developed a NO_3^--N determination method.

Methods

Fourteen surface soil samples were collected from the forests in Mt. Tsukuba and Mt. Kaba, Ibaraki prefecture, Japan (Table 1). DOC was extracted from each soil by 100 ml of water added to 20g of Soil. NO_3^--N in the extracts was removed with ion exchange resins (the mixture of Amberlite IR120 and Amberlite IRA410). We measured absorption spectra of each extract within 200 nm to 300 nm by a spectrophotometer (Shimadzu UVmini-1240). We calculated the ratio of the absorbance at 220nm to that at 260nm in each extract and used the mean value of the ratio in the following nitrate estimations. NO_3^--N was extracted with 2M KCl solution from thirty-nine soil samples collected from a Japanese cedar forest and a Japanese cypress forest in Mt. Tsukuba. We measured the absorbance at 220nm and 260nm in each 2M KCl extracts. We also measured the absorbance at 220nm and 260nm of 2M KCl extracts from seventeen soil samples that were collected in forests in Kobe, Ehime and Kagoshima in western Japan. Then, the 220nm absorbance originated from NO_3^--N ($[UV220]_{NO3}$) was estimated by the following formula.

$$[UV220]_{NO3} = [UV220] - [UV260] * K_{220/260} \qquad (1)$$

Here, [UV220] is an absorbance value at 220nm in the extracts. [UV260] is an absorbance value at 260nm in the extracts. $K_{220/260}$ is the ratio of the absorbance at 220nm to that at 260nm originated from DOC.

Table 1. General information of soil samples and number of samples for water and KCl extraction.

Location	Latitude	Longitude	Vegetation[A]	Soil Type[B]	Parent Materials	Water extract Number	KCl extract Number
Tsukuba	36°10′N	140°10′E	Japanese cypress	BD	Volcanic Ash, Gneiss	2	20
Tsukuba	36°10′N	140°10′E	Japanese cedar	BD	Volcanic Ash, Gneiss	4	19
Kaba	36°18′N	140° 9′E	Deciduous Forests	BD(d)	Volcanic Ash, Granite	8	--
Kochi	33°28′N	133°00′E	Japanese cypress	BlD	Volcanic Ash	--	7
Kobe	34°43′N	135°10′E	Japanese cedar	BD	Tertiary sediment	--	6
Ehime	33°53′N	132°52′E	Mousoutiku Bamboo forests	BD(d)	Hornfels	--	4

[A] Japanese cypress: *Chamaecyparis obtusa*, Japanese cedar: *Cryptomeria Japonica*, Mousoutiku: *Phyllostachys pubescens.* [B] According to classification of forest soils in Japan (1975), BD: Moderately moist brown forest soils, BD(d) Moderately moist brown forest soils (drier subtype), BlD: Moderately moist black soils

NO_3^--N concentration at the KCl extracts was determined by comparing $[UV220]_{NO3}$ with the absorbancy of NO_3^--N standard solution. NO_3^--N concentration of each extract was also measured by the automated Cd reduction methods (Dia Instrument FI-N50). We call the former the UV method and the latter the automated Cd reduction method.

Results

The absorption spectra of DOC and NO_3^--N

Each water extract after deionization showed UV absorption, and its optical density was least at 300nm and gradually increased toward shorter wavelength (Figure 1). These extracts did not contain NO_3^--N, so the absorption curve observed was considered to be derived from DOC. On the contrary, the adsorption curve of NO_3^--N has no absorption in the range from 250nm to 300nm and abruptly increased at 240nm. The mean value of the ratio of absorbancy at 220nm to at 260nm was 1.543 in water extracts after deionization for fourteen soil/water extracts (Table 2), so we adopted 1.543 as the value of $K_{220/260}$ in the equation (1).

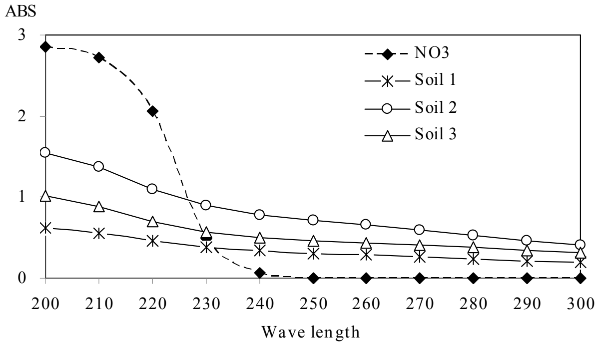

Figure 1. The absorption curves of three soil/water extracts after deionization and that of NO_3^--N solution.

The comparison of the UV method with the automated Cd reduction method

We compared the data by the UV method with those determined by the automated Cd reduction method for 2M KCl extracts from Mt. Tsukuba soils (Figure 2, left). There was a good relationship between the UV method and the automated Cd reduction method ($Y=1.040X-0.124$, $R^2=0.993$). If the absorbancy at 220nm of DOC was assumed to be 0, the slope of the regression line was 0.753. This indicated that NO_3^--N concentration by the UV method was overestimated compared with the automated Cd reduction method (Figure 2, right). We also compared both methods for NO_3^--N determination in KCl extracts from Kobe, Ehime and Kochi soils (Figure 3, left). The data for the UV method also were highly correlated with those by the automated Cd reduction method ($Y=1.099X-0.421$, $R^2=0.989$). On the other hand, in case no compensation was carried out for the absorbancy at 220nm, NO_3^--N concentration by the UV method was overestimated (Figure 3, right).

Discussion

Several simple methods have been proposed for the NO_3^--N determination (Cataldo *et al.* 1975; Kanno *et al.*1968; Norman and Stucki 1981; Sakata 2000; Yang *et al.* 1998). These methods are simple but they need some steps such as reagent adding and heating. The UV method, we developed, is more simple. It only needs only a spectrophotometer and the measurement of absorbancy at 220nm and 260nm. It is the most simple procedure to determine NO_3^--N in KCl extracts from soils.

Table 2. The ratio of absorbance at 220nm to at 260nm in soil/water extracts after deionization.

Location	Vegetation	Soil depth (cm)	UV220	UV260	K220/260
Tsukuba	Japanese Cypress	0-5	1.054	0.637	1.655
Tsukuba	Japanese Cypress	0-5	0.526	0.33	1.594
Tsukuba	Japanese Cedar	0-5	0.681	0.446	1.527
Tsukuba	Japanese Cedar	0-5	0.645	0.443	1.456
Tsukuba	Japanese Cedar	0-5	0.569	0.375	1.517
Tsukuba	Japanese Cedar	20-25	0.309	0.179	1.726
Tsukuba	deciduous Forests	0-5	2.382	1.551	1.536
Kaba	deciduous Forests	5-15	2.901	1.953	1.485
Kaba	deciduous Forests	15-30	0.457	0.287	1.592
Kaba	deciduous Forests	0-5	2.868	1.938	1.48
Kaba	deciduous Forests	5-15	0.31	0.204	1.52
Kaba	deciduous Forests	15-30	0.409	0.261	1.567
Kaba	deciduous Forests	0-10	1.059	0.717	1.477
Kaba	deciduous Forests	20-30	0.912	0.62	1.471
mean					1.543
Max.					1.726
Min.					1.456
S.D.					0.077

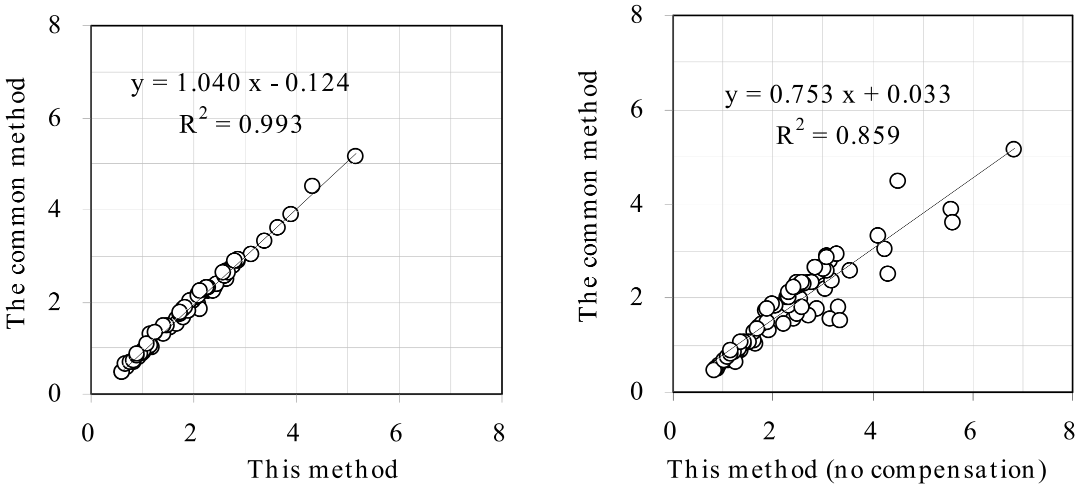

Figure 2. Comparison of NO_3^--N concentrations by the UV method (this method) with those by the automated Cd reduction method (the common method) for KCl extracts from Tsukuba soils (Unit is mg N/L). $K_{220/260}$ = 1.543 in the left figure. $K_{220/260}$=0 in the right figure.

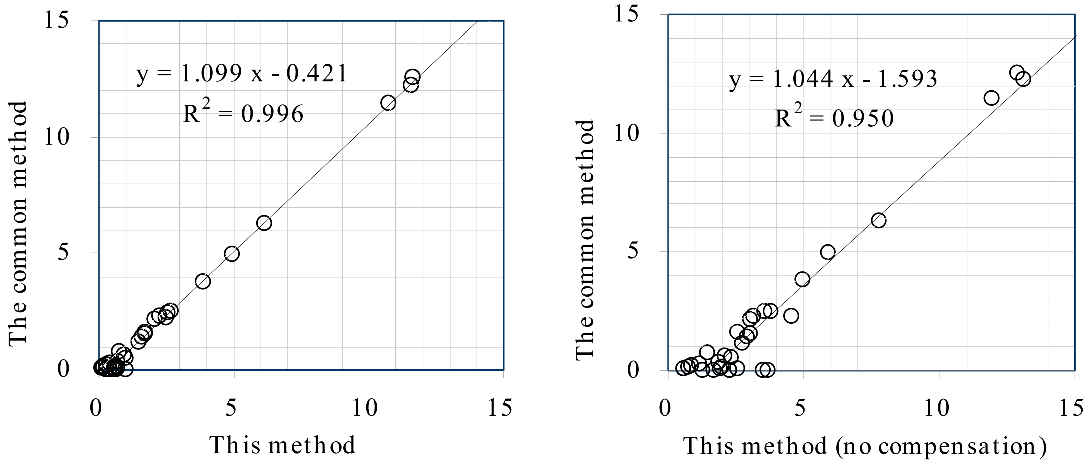

Figure 3. Comparison of NO_3^--N concentrations by the UV method (this method) with those by the automated Cd reduction method (the common method) for KCl extracts from Kobe, Ehime and Kagoshima soils (Unit is mg N/L). $K_{220/260}$ = 1.543 in the left figure. $K_{220/260}$=0 in the right figure.

The accuracy of the UV method depends on the value of $K_{220/260}$ in the equation(1). $K_{220/260}$ varied from 1.456 to 1.726 in the fourteen soil/water extracts. In the case of $K_{220/260}$ =1.456, the slope, the y-intercept and the R^2 of the regression line, are calculated to be as 1.064, 0.106 and 0.996 for the KCl extracts from Tsukuba soils, respectively. In the case of $K_{220/260}$ =1.726, the slope, the y-intercept and the R^2 are 1.027, 0.128 and 0.991, respectively. Each parameter is almost same in the case of $K_{220/260}$ =1.543 (Figure 2, left). This result suggests that the value of NO_3^--N estimation would be almost constant even if the value of $K_{220/260}$ varied to some extent. The NO_3^--N estimated by the UV method is highly correlated with values for the automated Cd reduction method for KCl extracts from forest soils in western Japan (Figure 3, left). However, as for the slope and the y-intercept of the regression line, the correspondence of two methods is somewhat low compared with the case of Tsukuba soils. The soil type of the Kochi soils is different from that of Tsukuba soils and vegetation for Ehime soils is different from that of Tsukuba soils (Table 1). We assumed that the $K_{220/260}$ of DOC is constant in KCl extracts from soils but the low correspondence suggests that the $K_{220/260}$ of DOC may vary with soil type and vegetation type. We need more information about $K_{220/260}$ of DOC extracted from soils under different environments.

Conclusion

The method that we developed to determine NO_3^--N in KCl extracts, only needs a spectrophotometer and the measurement of absorbance at 220nm and 260nm. We conclude that it is the simplest procedure to determine NO_3^--N in KCl extracts from forest soils.

References

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