Improving Extraction Method. Determining NO$_2$-N. Acidic Soil

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Abstract: Nitrite (No$_2$-N) A key intermediate. both nitrification, denitrification which. also closely related. Emission. kg gas from soil. since its Rapid Reaction. soil, instability. acidic condition a large number. decomposition will occur. extraction process using 2 mol/L KCl. order. enhance. accuracy. study. NO$_2$-N changes. acidic soil a more appropriate extraction method should be investigated. improve. efficiency. nitrite extraction.. study we put forward some suggestions. improving NO$_2$-N extraction based our results. NO$_2$-N recovery measured by using $^{15}$N-labelling method.-$N$ and NH$_4$-$N$ in Alibaba socks. Within 30 min breaking, the recovery of no$_2$-$N$ was highest in the adjusted pH 6.0 and pH 8.0 treatments, while the recovery of ammo was highest in the unadjusted and adjusted pH 6.0 treatments. in addition, even after breaking, the pH of extraction solution in the adjusted pH 6.0 and pH 8.0 treatments were maintained at about 4.8 and 5.8, which were significant higher than that of control (3.8 ). in all, the pH of extraction solution should be adjusted to 5.0-6.0 and aging time should be 30 min to get a more efficient extraction of both no$_2$-$N$ and NH$_4$-$N$. For strong acidic soils (pH < 6.0), a mixture of KCl and pH 8.4 buffer (KCl : Buffer = 4 : 1) should be used as extraction solution (the ratio. soil. extraction solution. 1 : 5). alkaline soils (pH ≥ 7.5) A mixture. KCL, pH 7.5 buffer (KCl : Buffer = 4 : 1) should be used as extraction solution (the ratio. soil. extraction solution. 1 : 5). soil samples Ph ~ 6.0. 7.5 It can be extracted directly by 2 mol/L KCL solution.

Keywords: Nitrite; recovery; pH buffer; acidic soil

1. Material and Methods

1.1 For try Soil

Select 5 of different PH of soil respectively for Jiangxi double Shenzhen Forest Field Red Soil (SZPH Water 4.5), Of Jiangxi longhu mountain Paddy Soil (LHSHPH Water 5.2), Jiangsu Yixing Paddy Soil (YxPH Water 6.2), Inner Mongolia xilinhot chestnut soil (NmPH Water 7.4) And Sichuan yanting PURPLE SOIL (Yt PH Water 7.8). Sampling depth 0 ~ 15 cm, 2mm Sieve after, Part in 4℃ Save spare other part in at room temperature under air-dry determination soil physical and chemical properties. For try soil of Main Physical and Chemical Properties see

1.2 Test Design

1.2.1 PHAnd extraction time of soil inNO$_2$-N,NH$_4^+$-NRecovery Rate of influence test select a kind of strong acid soil (SZPH Water 4.5) Said take fresh soil (Equivalent to drying soil Natural 20g) Placed 250 ml Triangle flask in. Is set PH and extraction time interaction processing test, 3APH Processing that is not regulation PH (pH 4.5 About), PH 6.0, PH 8.0 Processing each PH Processing again is set 4A extraction time the Oscillation 10, Natural 20, 30, 60 min In 12A processing. Each processing respectively is set

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NO$_2^-$-N and $^{15}$NH$_4^+$-N Two group the group join Na$^{15}$NO$_2$ Solution ($^{15}$N Abundance 2 atom %) In addition a group join ($^{15}$NH$_4^+$) SO$_4$ Solution ($^{15}$N Abundance 5.34 atom %) 3 Times repeat in 72 Bottle. Due to acid soil in NO$_2^-$-N Background value low for can accurate to Detection NO$_2^-$-N Concentration and degree of change this test NO$_2^-$-N The amount 3 mg/kg and Stevens And Laughlin [14] Of study similar; and NH$_4^+$-N The amount of reference the for try Soil NH$_4^+$-N Background Value 

$$(11.1 \text{ mg/kg}) \times 10 \text{ mg/kg}.$$ KOH Solution regulation Soil/2 mol/L KCl Suspension PH Test Set Value (Soil/Water = 1/2). 5 Determination PH) Quickly join Mark of after immediately in accordance with book

Extraction time oscillation extraction filter after determination filtrate in NH$_4^+$-N, NO$_3^-$-N, NO$_2^-$-N Content and $^{15}$NH$_4^+$-N, $^{15}$NO$_2^-$-N $^{15}$N Abundance at the same time determination Extract PH.

### 1.2.2 PH Buffer Solution regulation Extract PH Effect Test With

PH Buffer Solution: Will 1/15 mol/L The potassium dihydrogen phosphate solution and Disodium hydrogen phosphate solution According to the different proportion Mixed preparation PH For 7.5, 8.0, 8.4 Of phosphate buffer solution. At the same time preparation concentration 2, 2.5 And 3.33 mol/L Of KCl Solution. To SZ, LHS, YX, NM, YT Five soil for study object each soil said take some A fresh soil (Equivalent to drying soil 10 g) To 100 ml Triangle flask in is set 3 Of Processing: the control that don't and buffer direct and 50 ml Mol/L KCl Solution after Shake static the determination PH After placed Shaker, 200 r/min Respectively Oscillation 10, 20, 30, 60 min After determination PH; $\text{②}$ 10 ml Buffer Solution Processing the extract 40 ml 2.5 mol/L KCl Solution and 10 ml Buffer solution mixed solution which SZ And LHS Join PH 8.4 Of Buffer Solution, YX Join PH 8.0 Buffer Solution, NM And YT Join PH 7.5 Buffer solution the rest of the operation with the;

ML Buffer Solution Processing the extract 30 ml 3.33 mol/L KCl Solution and Natural 20 ml Buffer solution mixed solution which SZ And LHS Join PH 8.4 Of Buffer Solution, YX Join PH 8.0 Of Buffer Solution, NM And YT Join PH 7.5 Of buffer solution the rest of the operation with. Study different buffer processing PH Of regulation effect.

### 1.2.3 Soil NO$_2^-$-N Dynamic Test select SZ And YT Two kind of soil each soil said take fresh soil (Equivalent to drying soil 10 g) The

100 ml Triangle flask in 45 Bottle, adjust the water content 40% WHC, Pre-incubation 3 DA After adjusting the water content 60% WHC; Preservation film seal, hole to ensure ventilation, placed in 25 C Incubator cultures and every 3 D Weigh water. In the cultivation 0, 1, 3, 5, 10 d Conduct destructive sampling. Settings 3. Different Extraction Methods: (1) 50 ml 2 mol/L KCl Solution CK (2) Join 40 ml 2.5 mol/L KCl Solution and 10 ml Buffer Mixed Solution (T1) 3. Join 30 ml 3.33 mol/L KCl Solution and 20 ml Buffer Mixed Solution (T2) In a shaker, 200 r/min, Oscillation 30 min After filtration, determination of the filtrate in NO$_2^-$-N Content.

### 1.3 Analysis Method

NH$_4^+$-N, NO$_3^-$-N, NO$_2^-$-N Determination of Content: Using Continuous Flow Dynamic Analyzer (Skalar san++) Determination. $^{15}$N Abundance determination using isotope ratio mass spectrometer (Thermo Delta V plus) Determination, wherein

NH$_4^+$-N China $^{15}$N Abundance samples were prepared by diffusion method [20], $^{15}$NO$_2^-$-N China $^{15}$N Abundance sample using $N_2$ OP Production and legal preparation [21]. Soil PH Test Set: PH Of (Mettler TOLEDO S220 Seven Compact) Determination.

### 1.4 Data Processing

Recovery rate calculation:

$$Q_2 \left(\frac{1}{20} \times 0.3663 \text{ atom %} \right) = 100\% Q_1 \left(\frac{1}{20} \times 0.3663 \text{ atom %} \right)$$

- In: R For recovery rate, %; Q$_1$ For join of nitrogen concentration, Mg/kg; Q$_2$ For extraction Determination of nitrogen
concentration,Mg/kg; A1 For add15N Abundance, Atom %; A2 For extraction after determination15N Abundance, Atom %.

Determination of the data Excel 2010 The data processing "with Variance Analysis (SPSS 16.0) Test the processing between the difference significant Origin 8.0 Drawing.

2. Results

2.1 Extraction Process in Soil Extract PH Of change

1 Data show that, 2 mol/L KCl/Soil Suspension PH To was significantly lower than that of the water/Soil suspension. Test 1 For try Soil (Sz) 2 mol/L KCl/Soil Suspension PH Only 3.8. Oscillation 10 min When different processing KCl Extract PH Were significantly decreased, PH 4.5, PH 6.0 PH 8.0 Processing PH Respectively 3.8, 4.8 And 5.8 (Figure 1). Of After Extract PH Relative stability.

2.2 Different PH And oscillation time of Acid Soil NO2- N, NH4+-

N Recovery Rate of influence Test results show that different PH Processing NO2- N Recovery rate difference significantly (Figure 2). With PH The increase, NO2- NO of recovery rate Also increased. Not regulation PH Processing (PH 4.5) NO2- N Recovery rate was only 21% ~ 63%; Even though the Oscillation 10 min After Regulation PH Processing PH Were significantly decreased NO2- N Recovery Rate (> 90%) Were significantly higher than that of not regulation PH Processing. Oscillation time also influence NO2- NO of recovery rate (Figure 2). As the oscillation time increases, NO2- N The recovery rate decreased with the oscillation time being 10, 20, 30 min Compared to oscillation time 60 min NO2- N The recovery rate decreased more, and this phenomenon was unadjusted PH Processing is particularly obvious. Its Oscillation 60 min Empress NO2- N The recovery rate is only Oscillation 10 min Time 30%. Except Oscillation 60 min Outside, The pH 6.0 And The pH 8.0 The difference between treatments was not significant, NO2- N Average recovery rate > 92%. Oscillation 60 min Time, The pH 6.0 Processing NO2- N Recovery rate was significantly lower The pH 8.0 Processing. Two-factor variance analysis showed that, PH Versus oscillation time NO2- N There was a significant interaction effect on the recovery rate. Oscillation time 30 min Within, The pH 8.0 Processing NO2- N The recovery rate is the highest, but The pH 6.0 The handling difference is not significant.

(Different lowercase letters represent different PH Deal with the difference in recovery rate at the same oscillation time P < 0.05 Significant horizontally; Different uppercase letters represent the same PH The recovery rate at different extraction times was different. P < 0.05 Significant level)

2. Different PH Different oscillation times NO2- N Recovery Rate

NH4+ - N Recovery Rate and NO2- N Different (Figure 3). In addition to vibration 30 min Outside processing, adjusting PH Okay NH4+ - N But the oscillation time has little effect on the recovery rate. NH4+ - N The recovery rate has a significant effect on the Oscillation 30 min NH4+ - N The recovery rate is the highest, significantly higher than other oscillation time, Oscillation 10 min And 20 min NH4+ - N The recovery rate is the lowest. Two-factor variance analysis showed that, PH Versus oscillation time NH4+ - N There was a significant interaction effect on the recovery rate. Oscillation time is 30 min, Unadjusted PH And The pH 6.0 The recovery rate was the highest, significantly higher than other treatments.

2.3 Buffer Solution on Soil Extract PH Influence

After adding buffer, with the increase of oscillation time PH Especially the strong acidic soil (Sz), PH In Oscillation 10 min Within the rapid decline, Oscillation 60 min After,
Join 10 ml And 20 ml Buffer treatment, PH from the beginning 6.18 Fall 5.0 and 6.0. Remaining Soil PH the amplitude of change with oscillation time is less S (small), In the process of Oscillation PH can keep in 6.0 ~ 7.0.

2.4 $\text{NO}_2^-$ Improvement Effect of Extraction Method

The results showed that with the increase of incubation time $\text{NO}_2^-$ content gradually increased. Strong Acidic Soil (Sz) Add 10 ml And ML Buffer Solution for extraction and determination $\text{NO}_2^-$ there is little difference in content.

Were significantly higher CK Treatment, cultivation 10 d After the determination of Soil $\text{NO}_2^-$ content is CK Of 2.5 Times (Figure 5A). Sichuan yanting alkaline purple soil (Yt) With the increase of incubation time $\text{NO}_2^-$ content also gradually increased. But throughout the training process, 3. Different Treatments $\text{NO}_2^-$ No significant difference in content (Figure 5b).

3. Discussion

The soil in $\text{NO}_2^-$ Of extraction determination results and extract PH close related. Due to soil of buffer ability and KCISolution of influence EXTRACTION PROCESS IN PH 6.0 And PH 8.0 Processing of Soil/KCl Suspension PH keep in 4.8 And 5.8 The conditions under $\text{NO}_2^-$ Recovery rate reached 90% More. Nelson, Bremner[22] The study also found, PH In 4.8 ~ 6.0 (Soil/Water = 1/2. 5) Between an arcane $\text{NO}_2^-$ The recovery rate of 69% But when PH For 7.1 ~ 7.8 When average recovery rate 95%. If taking into account KCISolution will significantly reduce Suspension PH Nelson And Bremner[22] Of results and this study of results should be basic consistent. Has some research show that in strong acid environment under, $\text{NO}_2^-$

Can easily be transformed into $\text{HNO}_2$, $\text{HNO}_3$ Then self-decomposition main produce $\text{NO}_2^-$ Again further oxidation $\text{NO}_2^-$[23]. When PH high, $\text{NO}_2^-$-NOf decomposition by limit so $\text{NO}_2^-$-N PH loss Reduce[17, 24]. At the same time

$\text{NO}_2^-$ The recovery also and extraction time on with the extraction time of increase recovery rate decreased significantly. Stevens And Laughlin[14] Also observation to extraction time 10 min An arcane $\text{NO}_2^-$-N Average recovery rate (86%) Is Extraction 70 min Of 3 Times. This study results show that, PH And oscillation time $\text{NO}_2^-$-NOf recovery rate there significant of interaction influence oscillation time 30 min Inside, extract PH Keep in 5.0 About, $\text{NO}_2^-$-N The recovery rate can be > 92%. While remaining in the Process of Extraction PH The higher (Such PH 7 ~ 8. Between) Can guarantee a higher $\text{NO}_2^-$-N Recovery Rate[22], But higher PH Environmental and long-term oscillations may contribute to the volatilization loss of ammonia, thus underestimating Soil $\text{NH}_4^+$-N Content. The results of this study showed that PH Keep in 5.0 Around, the oscillation time is 30 min Time, $\text{NH}_4^+$-N The highest recovery rate. Considering this, the optimal extraction conditions of inorganic nitrogen from acidic soil are: Extract PH Keep 5.0 ~ 6.0, Oscillation extraction time 30 min Can also guarantee the soil

$\text{NH}_4^+$-N And $\text{NO}_2^-$-N Extraction efficiency. 2 mol/L KCISolution Extraction of strong acidic soil $\text{NO}_2^-$-N When underestimated $\text{NO}_2^-$-N The actual content PH Fang

Law can effectively avoid this problem. The commonly used method in existing research is to use Koh Solution-adjusted Soil/KCISuspension PH However, this method is tedious. /KCISuspension PH Not easy to keep. In contrast, using a low concentration of phosphate buffer solution PH The method is more simple, easy to operate and in Oscillation 60 min After all can maintain a higher PH. The results show that phosphate buffer solution can effectively maintain PH Meet the requirements of extraction conditions.

Not yet fully clear on 2 mol/L KCIThe accurate mechanism of nitrite changes during extraction $\text{NO}_2^-$-N The rate of reaction depends on PH Organic matter, reaction time and other factors that have not yet been identified[14]. Therefore, the impact of the Extraction Process $\text{NO}_2^-$-N The dynamic mechanism needs to be further studied, which is important for the further optimization of the extraction method.

4. Conclusion

The results show that the strong acidic soil $\text{NO}_2^-$-N During extraction, soil/KCISuspension PH Should be kept in 5.0 ~ 6.0, Oscillation time 30 min Can also guarantee the soil $\text{NH}_4^+$-N And $\text{NO}_2^-$-N Extraction efficiency. For strongly acidic
soils (PH < 6.0), Recommended KCl Solution and The pH 8.40f Buffer Solution (KCl Solution/Buffer 4/1) As an extract (Soil/Liquid than 1/5). For PH in 7.5 To

On the of soil samples recommended “KCl Solution and PH 7.50f Buffer Solution (KCl Solution/Buffer than 4/1) As an extract (Soil/Liquid than 1/5). For PH in 6.0 ~ 7.50f soil samples can directly” 2 mol/L KCl Solution Extraction.

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