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ORIGINAL ARTICLE

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. effeciency. nitrite extraction.. study we put forward some suggestions. improving NO-$_2$-N extraction based our resutls. NO-$_2$-N recovery measured by using ^{15}N-labelling method.-$_2$.-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 maintaken 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

Select5Of differentPHOf soil respectively for Jiangxi double Shenzhen Forest Field Red Soil(SZPHWater4.5), Of Jiangxi longhu mountain Paddy Soil(LHSPHWater 5.2), Jiangsu Yixing Paddy Soil(YxPHWater6.2), Inner Mongolia xilinhot chestnut soil(NmPHWater7.4)And Sichuan yanting PURPLE SOIL(Yt PHWater7.8). Sampling depth0 ~ 15 cm.2mmSieve after, Part in4℃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(SZPHWater4.5)Said take fresh soil(Equivalent to drying soilNatural 20g)Placed250 mlTriangle flask in. Is setPHAnd extraction time interaction processing test,3APHProcessing that is not regulationPH (pH 4.5About),PH 6.0,PH 8.0Processing eachPHProcessing again is set4A extraction time the Oscillation10,Natural 20,30,60 minIn12A processing. Each processing respectively is set

NO^-_2-NAnd$^{15}NH_4^+$-NTwo group the group join$Na^{15}NO_2$Solution(^{15}NAbundance2 atom %)In addition a group join$(^{15}NH_4)_2SO_4$Solution(^{15}NAbundance5.34 atom %)3Times repeat in72Bottle. Due to acid soil inNO^-_2-NBackground value low for can accurate to DetectionNO^-_2-NConcentration and degree of change this testNO^-_2-NThe amount3 mg/kgAndStevensAndLaughlin[14]Of study similar; andNH_4^+-NThe amount of reference the for try SoilNH_4^+-NBackground Value

(11.1 mg/kg)Is set10 mg/kg."KOHSolution regulation Soil/2 mol/L KClSuspensionPHTo Test Set Value(Soil/Water= 1/2. 5DeterminationPH)Quickly join Mark of after immediately in accordance with book

Extraction time oscillation extraction filter after determination filtrate inNH_4-N,NO^-_3-N,NO^-_2-NContent and$^{15}NH_4$-N,$^{15}NO^-_2$-NIn^{15}NAbundance at the same time determination ExtractPH.

1.2.2 PHBuffer Solution regulation ExtractPHEffect Test With

PHBuffer Solution: Will1/15 mol/LThe potassium dihydrogen phosphate solution and

Disodium hydrogen phosphate solution[19]According to the different proportion Mixed preparationPHFor7.5,8.0,8.4Of phosphate buffer solution. At the same time preparation concentration2,

2.5And3.33 mol/LOfKClSolution. ToSZ,LHS,YX,NM,YTFive soil for study object each soil said take some

A fresh soil(Equivalent to drying soil10g)To100 mlTriangle flask in is set3Of Processing: the control that don't and buffer direct and50 ml

Mol/L KClSolution after Shake static the determinationPHAfter placed Shaker,200 r/minRespectively Oscillation10,Natural 20,30,60 minAfter determinationPH; ②10 mlBuffer Solution Processing the extract40 ml 2.5 mol/L KClSolution and10 mlBuffer solution mixed solution whichSZAndLHSJoinPH 8.4Of Buffer Solution,YXJoinPH 8.0Buffer Solution,NMAndYTJoinPH 7.5Buffer solution the rest of the operation with the;

MLBuffer Solution Processing the extract30 ml 3.33 mol/L

KClSolution andNatural 20 mlBuffer solution mixed solution whichSZAndLHSJoinPH 8.4Of Buffer Solution,YXJoinPH 8.0Of Buffer Solution,NMAndYTJoinPH 7.5Of buffer solution the rest of the operation with. Study different buffer processingPHOf regulation effect.

1.2.3 SoilNO^-_2-NDynamic Test selectSZAndYTTwo kind of soil each soil said take fresh soil(Equivalent to drying soil10g)The

100 mlTriangle flask in45Bottle, adjust the water content

40% WHC, Pre-incubation3 DAfter adjusting the water content60% WHC,

Preservation film seal, hole to ensure ventilation, placed in25CIncubator cultures and every3 DWeigh water. In the cultivation0,1.,3.,5.,10 dConduct destructive sampling. Settings3.Different Extraction Methods: (1)

50 ml 2 mol/L KClSolutionCK)(2) join40 ml 2.5 mol/L KClSolution and10 mlBuffer Mixed Solution(T1)3. Join30 ml 3.33 mol/L KClSolution and20 mlBuffer Mixed Solution(T2),

In a shaker,200 r/min, Oscillation30 minAfter filtration, determination of the filtrate inNo^-_2.-NContent.

1.3 Analysis Method

NH_4^+-N,No^-_3.-N,No^-_2.-NDetermination of Content: Using Continuous Flow

Dynamic Analyzer(Skalar san^{++})Determination.^{15}NAbundance determination using isotope ratio mass spectrometer(Thermo Delta V plus)Determination, wherein

NH_4^+-NChina^{15}NAbundance samples were prepared by diffusion method[20],$^{15}No^-_2$.-NChina^{15}NAbundance sample usingN_2OProduction and legal preparation[21]. SoilPHTest

Set:PHOf(Mettler TOLEDO S220 Seven Compact)Determination.

1.4 Data Processing

Recovery rate calculation:

$$Q_2 \ (A_2 \ 0.3663 \text{ atom \%}) \ _{100\%}Q_1 \quad (A_1 \quad 0.3663 \text{ atom \%})$$

-In:RFor recovery rate,%;Q_1For join of nitrogen concentration,Mg/kg;Q_2For extraction Determination of nitrogen

concentration,Mg/kg;A_1For add^{15}NAbundance,Atom %;A_2For extraction after determination^{15}NAbundance,

Atom %.

Determination of get of dataExcel 2010The data processing "with Variance Analysis(SPSS16.0)Test the processing between the difference significantOrigin 8.0Drawing.

2. Results

2.1 Extraction Process in Soil ExtractPHOf change

1Data show that,2 mol/L KCl/Soil SuspensionPHTo

Was significantly lower than that of the water/Soil suspension. Test1For try Soil(Sz) 2 mol/L KCl/Soil SuspensionPHOnly3.8. Oscillation10 minWhen different processingKClExtractPHWere significantly decreased,PH 4.5,PH 6.0

PH 8.0ProcessingPHRespectively3.8,4.8And5.8 (Figure1). Of

After ExtractPHRelative stability.

2.2 DifferentPHAnd oscillation time of Acid SoilNO^-_2-N,NH_4^+-

N Recovery Rate of influence

Test results show that differentPHProcessingNO^-_2-NRecovery rate difference significantly(Figure2). WithPHThe increase,NO^-_2-NOf recovery rate

Also increased. Not regulationPHProcessing(PH 4.5)NO^-_2-NRecovery rate was only21% ~ 63%; Even though the Oscillation10 minAfter RegulationPHProcessingPHWere significantly decreasedNO^-_2-NRecovery Rate(> 90%)Were significantly higher than that of not regulationPHProcessing. Oscillation time also influenceNO^-_2-NOf recovery rate(Figure2). As the oscillation time increases,$No^-_{2.}$-NThe recovery rate decreased with the oscillation time being10,20,30 minCompared to oscillation time60 min $No^-_{2.}$-NThe recovery rate decreased more, and this phenomenon was unadjustedPHProcessing is particularly obvious, Its Oscillation60 minEmpress$No^-_{2.}$-NThe recovery rate is only Oscillation10 minTime30%. Except Oscillation60 minOutside,The pH 6.0AndThe pH 8.0The difference between treatments was not significant,$No^-_{2.}$-NAverage recovery rate> 92%. Oscillation60 minTime,The pH 6.0Processing$No^-_{2.}$-NRecovery rate was significantly lowerThe pH 8.0Processing. Two-factor variance analysis showed that,PHVersus oscillation time$No^-_{2.}$-NThere was a significant interaction effect on the recovery rate. Oscillation time30 minWithin,The pH 8.0Processing$No^-_{2.}$-NThe recovery rate is the highest, butThe pH 6.0The handling difference is not significant.

(Different lowercase letters represent differentPHDeal with the difference in recovery rate at the same oscillation time

P<0.05Significant horizontally; Different uppercase letters represent the samePHThe recovery rate at different extraction times was different.P<0.05Significant level)

2.DifferentPHDifferent oscillation times$No^-_{2.}$-NRecovery Rate

$NH_{4.}^+$-NRecovery Rate and$No^-_{2.}$-NDifferent.(Figure3). In addition to vibration 30 minOutside processing, adjustingPHOkay.$NH_{4.}^+$-NBut the oscillation time has little effect on the recovery rate.$NH_{4.}^+$-NThe recovery rate has a significant effect on the Oscillation30 min $NH_{4.}^+$-NThe recovery rate is the highest, significantly higher than other oscillation time, Oscillation10 minAnd20 min $NH_{4.}^+$-NThe recovery rate is the lowest. Two-factor variance analysis showed that,PHVersus oscillation time$NH_{4.}^+$-NThere was a significant interaction effect on the recovery rate. Oscillation time is30 min, UnadjustedPHAndThe pH 6.0The recovery rate was the highest, significantly higher than other treatments.

2.3 Buffer Solution on Soil ExtractPHInfluence

After adding buffer, with the increase of oscillation timePHEspecially the strong acidic soil.(Sz),PHIn Oscillation10 minWithin the rapid decline, Oscillation60 minAfter,

Join10 mlAnd20 mlBuffer treatment,PHFrom the beginning6.18Fall5.0And6.0. Remaining SoilPHThe amplitude of change with oscillation time is lessSZ, In the process of OscillationPHCan Keep in 6.0 ~ 7.0.

2.4 No^{-}_{2}-NImprovement Effect of Extraction Method

The results showed that with the increase of incubation timeNo^{-}_{2}-NContent gradually increased. Strong Acidic Soil(Sz)Add10 mlAnd

MLBuffer Solution for extraction and determinationNo^{-}_{2}-NThere is little difference in content,

Were significantly higherCKTreatment, cultivation10 dAfter the determination of SoilNo^{-}_{2}-NContent isCKOf2.5Times(Figure5A). Sichuan yanting alkaline purple soil(Yt)With the increase of incubation timeNo^{-}_{2}-NContent also gradually increased. But throughout the training process,3.Different TreatmentsNo^{-}_{2}-NNo significant difference in content(Figure5b).

3Discussion

The soil inNO^{-}_{2}-NOf extraction determination results and extractPHClose related. Due to soil of buffer ability andKClSolution of influence EXTRACTION PROCESS INPH 6.0AndPH 8.0Processing of Soil/KClSuspensionPHKeep in4.8And5.8The conditions underNO^{-}_{2}-NRecovery rate reached

90%More.Nelson, Bremner[22]The study also found,PHIn4.8 ~ 6.0 (Soil/Water= 1/2. 5)Between an arcaneNO^{-}_{2}-NThe recovery rate of69%But whenPHFor7.1 ~ 7.8When average recovery rate 95%. If taking into accountKClSolution will significantly reduce SuspensionPH NelsonAndBremner[22]Of results and this study of results should be basic consistent. Has some research show that in strong acid environment under,NO^{-}_{2}-N

Can easily be transformed into$HNO_2$$HNO_2$Then self-decomposition main produceNOAgain further oxidationNO_2[23]. WhenPHL high,NO^{-}_{2}-NOf decomposition by limit soNO^{-}_{2}-NLoss Reduce[17,24]. At the same time

NO^{-}_{2}-NThe recovery also and extraction time on with the extraction time of increase recovery rate decreased significantly.StevensAndLaughlin[14]Also observation to extraction time10 minAn arcaneNO^{-}_{2}-NAverage recovery rate(86%)Is Extraction70 minOf3Times. This study results show that,PHAnd oscillation timeNO^{-}_{2}-NOf recovery rate there significant of interaction influence oscillation time30 minInside, extractPHKeep in5.0About,No^{-}_{2}-NThe recovery rate can be> 92%. While remaining in the Process of ExtractionPHThe higher(SuchPH 7 ~ 8.Between)Can guarantee a higherNo^{-}_{2}-NRecovery Rate[22], But higherPHEnvironmental and long-term oscillations may contribute to the volatilization loss of ammonia, thus underestimating SoilNH_4^{+}-NContent. The results of this study showed thatPHKeep in5.0Around, the oscillation time is30 minTime,NH_4^{+}-NThe highest recovery rate. Considering this, the optimal extraction conditions of inorganic nitrogen from acidic soil are: ExtractPHKeep 5.0 ~ 6.0, Oscillation extraction time30 minCan also guarantee the soil

NH_4^{+}-NAndNo^{-}_{2}-NExtraction efficiency. 2 mol/L KClSolution Extraction of strong acidic soilNo^{-}_{2}-NWhen underestimatedNo^{-}_{2}-NThe actual contentPHFang

Law can effectively avoid this problem. The commonly used method in existing research is to useKohSolution-adjusted Soil/KClSuspensionPHHowever, this method is tedious./KClSuspensionPHNot easy to keep. In contrast, using a low concentration of phosphate buffer solutionPHThe method is more simple, easy to operate and in Oscillation60 minAfter all can maintain a higherPH. The results show that phosphate buffer solution can effectively maintainPHMeet the requirements of extraction conditions.

Not yet fully clear on2 mol/L KClThe accurate mechanism of nitrite changes during extraction.No^{-}_{2}-NThe rate of reaction depends onPHOrganic matter, reaction time and other factors that have not yet been identified[14]. Therefore, the impact of the Extraction ProcessNo^{-}_{2}-NThe 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 soilNo^{-}_{2}-NDuring extraction, soil/KClSuspensionPHShould be kept in5.0 ~ 6.0, Oscillation time30 minCan also guarantee the soilNH_4^{+}-NAndNo^{-}_{2}-NExtraction efficiency. For strongly acidic

soils(PH <6.0), RecommendedKClSolution andThe pH 8.4Of Buffer Solution(KClSolution/Buffer 4/1)As an extract(Soil/Liquid than1/5). ForPHIn7.5To

On the of soil samples recommended"KClSolution andPH 7.5Of Buffer Solution(KClSolution/Buffer than4/1)As an extract(Soil/Liquid than1/5). ForPHIn6.0 ~ 7.5Of soil samples can directly"2 mol/L KClSolution Extraction.

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