

Can Nonexchangeable Potassium be Differentiated from Structural Potassium in Soils?



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ABSTRACT

Nonexchangeable K (NEK) is the major portion of the reserve of available K in soil and a primary factor in determining soil K fertility. The questions of how much NEK is in soils and how to quantify total NEK in soils are so far still unclear due to the complicated effects of various minerals on K fixation. In this study, the NEK in 9 soils was extracted with sodium tetraphenylboron (NaBPh₄) for various time periods longer than 1 d. The results showed that the NEK extracted by NaBPh₄ gradually increased with time, but showed no more increase after the duration of extraction exceeded 10–20 d. As the temperature increased from 25 to 45 °C, the duration to obtain the maximum extraction of NEK was reduced from 20 to 10 d, and the maximum values of NEK released at both temperatures was almost the same for each soil. The maximum NEK (MNEK) of the 9 soils extracted by NaBPh₄ varied from 3074 to 10081 mg kg⁻¹, accounting for 21%–56% of the total soil K. There was no significant correlation between MNEK released by NaBPh₄ and other forms of K, such as NH₄OAc-extracted K, HNO₃-extracted K and total K in soils, which indicates that NEK is a special form of K that has no inevitable relationship to the other forms of K in soils. The MNEK extraction by NaBPh₄ in this study indicated that the total NEK in the soils could be differentiated from soil structural K and quantified with the modified NaBPh₄ method. The high MNEK in soils made NEK much more important in the role of the plant-available K pool. How to fractionate NEK into different fractions and establish the methods to quantify each NEK fraction according to their bioavailability is of great importance for future research.

Key Words: HNO₃-extracted K, K release dynamics, K reserve, NaBPh₄ method, NH₄OAc-extracted K, total K

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Soil potassium (K) fertility is important for plant growth and sustainable agriculture. In most soils, the total K reserve is generally large, but only a small portion is immediately, or slowly, available for plant uptake. The K in soils can be divided into water-soluble, exchangeable, nonexchangeable and structural forms. Nonexchangeable K (NEK) is the K held between adjacent tetrahedral layers of dioctahedral and trioctahedral micas, vermiculites and intergrade clay minerals (Martin and Sparks, 1985). The NEK is also sometimes referred to as “fixed K”, “interlayer K”, “slowly available K”, or “slowly exchangeable K”. The results of various previous studies demonstrated that the K absorbed by plants could be largely from NEK in the soils (Mengel and Uhlenbecker, 1993; Srinivasarao *et al.*, 2000; Syers, 2003; Hu and Wang, 2004; Øgaard and Krogstad, 2005; Wang *et al.*, 2010a).

As a reserve and the main part of soil available K, NEK is always a focus of research on soil K. Many methods such as dynamic (Meyer and Jungk, 1993; Cox

and Joern, 1997; Srinivasarao *et al.*, 2006), chemical (Cox *et al.*, 1999; Hosseinpour and Sinigani, 2007; Wang *et al.*, 2011), mineralogical (Hinsinger and Jaillard, 1993; Saha and Inoue, 1998; Andrist-Rangel *et al.*, 2010), electro-ultrafiltration (Wanisuria *et al.*, 1981; Mengel and Uhlenbecker, 1993; Lu *et al.*, 2007), cation exchange resin (Havlin *et al.*, 1985; Goulding and Loveland, 1986; Dhillon and Dhillon, 1990; Askegaard *et al.*, 2005), and plant-exhausting methods (Wang *et al.*, 2010a; Li *et al.*, 2015a) were used in various studies to investigate courses of K fixation, dynamics and mechanisms of NEK release, and NEK availability and utilization by plants. Much of the past research has also aimed at determining NEK with the above noted methods (Martin and Sparks, 1985). However, exactly how much NEK there is in soils is still unclear since there is currently no satisfactory method to measure or quantify total NEK in soils.

The amount of soil NEK is conventionally calculated as the difference between K extracted by 1 mol L⁻¹

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boiling HNO_3 and that by 1 mol L^{-1} neutral NH_4OAc (Sparks, 1980; Lu, 1999; Xie *et al.*, 2000). With this method, the estimated NEK content of soils varies from 2% to 10% of total K (Xie *et al.*, 2000, Römheld and Kirkby, 2011). The boiling HNO_3 method is so far not a satisfactory method for the extraction of NEK in soils. On one hand, it can destroy the structure of clay minerals and dissolve some small particles even made up of feldspar (Al-Kanani *et al.*, 1984; Xie *et al.*, 1989; Kobayashi *et al.*, 2001; Chardon *et al.*, 2006), which indicates that the boiling HNO_3 method extracts not only NEK but also some K not associated with the interlayers of 2:1 minerals. On the other hand, not all the NEK in the soil can be extracted with boiling HNO_3 . It has been shown that only a portion of fertilizer K in soil can be recovered with the boiling HNO_3 method even within 1 d (Wang *et al.*, 2010b). The soil K that can not be extracted by boiling HNO_3 could be available to plants, and sometimes acts as the main K resources to plant absorption (Richards and Bates, 1988; Yang *et al.*, 1998; Markewitz and Richter, 2000; Øgaard *et al.*, 2001). Some researchers recommended the successive extraction method with 1 mol L^{-1} boiling HNO_3 for determination of NEK. The sum of the constant amount of K released in several successive HNO_3 extractions is defined as “constant rate” K (CRK) (Haylock, 1956). Much more K released in the first few extractions than CRK is defined as step-K, which is an index of the most soluble fraction of the NEK (Haylock, 1956; Richards and Bates, 1988; Pal *et al.*, 2002). In addition, this method is relatively inconvenient and does not aim at measuring all NEK in soils. Andrist-Rangel *et al.* (2006) proved that both 2 mol L^{-1} boiling HCl - and aqua regia-extractable K are highly correlated with K in dioctahedral phyllosilicates and accounted for 1%–17% and 5%–45% of the total soil K, respectively. They also concluded that neither of the two methods extracted all K from the entire reserve of dioctahedral phyllosilicates in soils (Andrist-Rangel *et al.*, 2006).

The release of soil NEK depends on its position in clay minerals. So far, it seems that there is no way to clearly differentiate NEK from structural K in soils (Xie *et al.*, 2000). Thus, it is necessary to find an extractant that has strong extracting power and is preferential to NEK in soils. Sodium tetraphenylboron (NaBPh_4) seems to be an ideal extractant for this purpose. The NaBPh_4 method was first proposed for soil K extraction by Hanway (1956), and has since been developed by many researchers (Scott *et al.*, 1960; Reed and Scott, 1961; Cox *et al.*, 1996). Cox *et al.* (1996) used CuCl_2 instead of HgCl_2 in the recovery of K from

KBPh_4 , and developed a relatively convenient NaBPh_4 method for routine laboratory analysis. The NaBPh_4 method has proven to be a good method not only for evaluation of both soil K bioavailability and kinetic release of K from soils or K-bearing minerals (Cox and Joern, 1997; Cox *et al.*, 1999; Srinivasarao *et al.*, 2000; Wang *et al.*, 2010a), but also for the extraction of soil NEK (Cox *et al.*, 1996). However, whether the NaBPh_4 method could extract all the NEK in the soil remains unclear.

This study aimed to verify whether all soil NEK could be extracted with a modified NaBPh_4 method, and to investigate the relationship between NEK and other forms of K in soils, which might help to understand forms of soil K and to develop an appropriate method for the determination of total NEK in soils.

MATERIALS AND METHODS

Soils

Nine topsoils (0–20 cm) with a wide range of soil texture and K-buffering power were collected from different regions of China. Selected physicochemical properties of these soils are presented in Tables I and II. Each soil sample was air-dried and ground to pass through a 1-mm sieve. The mineralogy of soil samples (divided into < 2 and $> 2 \mu\text{m}$ parts) was directly evaluated by X-ray diffraction by using an Ultima IV X-ray diffractometer (Rigaku Corporation, Tokyo, Japan) with $\text{CuK}\alpha$ radiation (40 kV, 40 mA), from 3.0° to 60.0° with a scan speed of $2.0^\circ \text{ min}^{-1}$. Cation exchange capacity (CEC) was obtained by the NH_4OAc method, and CaCO_3 by the acid dissolution method. Soil organic matter was obtained by multiplying the organic carbon content determined by the Walkley-Black dichromate oxidation method (Lu, 1999) by a factor of 1.724. Soil pH was determined in a 1:2.5 soil to water suspension, and clay content was measured by a pipette method (Lu, 1999).

Extraction of K from soils and K determination

The readily available and slowly available K of the soils were extracted with the conventional methods of 1 mol L^{-1} neutral NH_4OAc and 1 mol L^{-1} boiling HNO_3 , respectively. Soil total K was extracted with NaOH (Lu, 1999).

The general procedure of the NaBPh_4 method used for extracting soil NEK was similar to that described by Cox *et al.* (1999). Modifications, such as 0.25 mol L^{-1} instead of 0.20 mol L^{-1} NaBPh_4 in the extracting solution and 0.14 mol L^{-1} instead of 0.11 mol L^{-1} CuCl_2 in the quenching solution, were adopted to in-

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