Received: 15 August 2022 | Accepted: 27 March 2023

DOI: 10.1111/2041-210X.14107

Methods in Ecology and Evolution

RESEARCH ARTICLE

Uncorrected soil water isotopes through cryogenic vacuum distillation may lead to a false estimation on plant water sources

Bin Yang[1] | **Gbadamassi G. O. Dossa**[1] | **Yue-Hua Hu**[1] | **Lu-Lu Liu**[1,2] | **Xian-Jing Meng**[3] | **Yi-Yuan Du**[1,2] | **Jia-Yuan Li**[1,2] | **Xi-Ai Zhu**[1] | **Yong-Jiang Zhang**[4] | **Ashutosh K. Singh**[1] | **Xia Yuan**[1] | **Jun-En Wu**[5] | **Sissou Zakari**[6] | **Wen-Jie Liu**[1] | **Liang Song**[1]

[1]CAS Key Laboratory of Tropical Forest Ecology, Xishuangbanna Tropical Botanical Garden, Chinese Academy of Sciences, Menglun, Yunnan, China; [2]University of Chinese Academy of Sciences, Beijing, China; [3]Thermo Fisher Scientific, Shanghai, China; [4]School of Biology and Ecology, University of Maine, Orono, Maine, USA; [5]Faculty of Geography, Yunnan Normal University, Yunnan, China and [6]Laboratory of Hydraulics and Environmental Modeling, Faculté d'Agronomie, Université de Parakou, Parakou, Benin

Correspondence
Wen-Jie Liu
Email: lwj@xtbg.org.cn

Liang Song
Email: songliang@xtbg.ac.cn

Funding information
Candidates of the Young and Middle Aged Academic Leaders of Yunnan Province, Grant/Award Number: 2019HB040; National Natural Science Foundation of China, Grant/Award Number: 32171557 and 32171529; Youth Innovation Promotion Association of CAS, Grant/Award Number: 2021397; Yunnan Fundamental Research Projects, Grant/Award Number: 202201AT070221 and 202101AS070010; Yunnan High Level Talents Special Support Plan, Grant/Award Number: YNWR-QNBJ-2020-066

Handling Editor: Clive Trueman

Abstract

1. Successful use of stable isotopes (δ^2H and $\delta^{18}O$) in ecohydrological studies relies on the accurate extraction of unfractionated water from different types of soil samples. Cryogenic vacuum distillation (CVD) is a common laboratory-based technique used for soil water extraction; however, the reliability of this technique in reflecting soil water δ^2H and $\delta^{18}O$ is still of concern.
2. This study examines the reliability of a newly developed automatic cryogenic vacuum distillation (ACVD) system. We further assessed the impacts of extraction parameters (i.e. extraction time, temperature and vacuum) and soil properties on the recovery of soil water δ^2H and $\delta^{18}O$ for the ACVD and traditional cryogenic vacuum distillation (TCVD) systems. Finally, we investigated the potential influence of CVD (ACVD and TCVD) technique on the prediction of plant water uptake through a sensitivity analysis.
3. Both ACVD and TCVD similarly extracted water from the rewetted soils, but none of the CVD systems successfully recovered the isotopic signatures of doped water from soil materials. Mean δ^2H offsets of extracted soil water were -2.6 ± 1.3‰ and -2.4 ± 1.7‰ for ACVD and TCVD, respectively, while mean $\delta^{18}O$ offsets were -0.16 ± 0.14‰ and -0.39 ± 0.37‰. The isotopic offsets of CVD systems were positively correlated with soil clay content, and negatively correlated with soil water content. Using corrected soil data (with CVD offsets) could improve the prediction of plant water uptake based on its high correlation with the environmental factors.

This is an open access article under the terms of the Creative Commons Attribution License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited.
© 2023 The Authors. *Methods in Ecology and Evolution* published by John Wiley & Sons Ltd on behalf of British Ecological Society.

4. This study identifies the isotopic offsets of CVD systems (i.e. ACVD and TCVD) and provides possible solutions for better predicting plant water sources. Even though, the wide use of CVD techniques probably induce noticeable uncertainties in the prediction of plants water uptake depths. The dataset of soil water extraction in this study will have implications for the technological development of CVD techniques.

KEYWORDS
eco-hydrology, extraction technique, inter-laboratory comparison, isotopic offsets, stable isotopes, water uptake

1 | INTRODUCTION

Soil water hydrogen and oxygen stable isotopes (δ^2H and δ^{18}O, respectively) have broad applications in ecological studies (Coplen, 2011; Ding et al., 2018; Gat, 1996). Such studies have significantly improved our understanding of the eco-hydrological processes, particularly the prediction of pant water sources (Ehleringer & Dawson, 1992; Grossiord et al., 2017), the interpretation of intra-interspecific water competition for water resources (Nie et al., 2019; Schwendenmann et al., 2010) and the identification of hydrological niche segregation among plant communities (Moreno-Gutiérrez et al., 2012; Rodríguez-Robles et al., 2020). Soil water δ^2H and δ^{18}O have also been applied to assume the existence of two water worlds (TWW), whereby plant-available water in the soil is distinct from stream water (Brooks et al., 2010; Chen et al., 2020; Good et al., 2015). Given the technical advancements in isotope ratio mass and infrared spectrometers (IRMS/IRIS), a bottleneck in soil stable isotope studies remains the ability to obtain measurable amounts of liquid water from samples rather than the analysis process itself (Gehre et al., 2004; Griffis, 2013; Wassenaar et al., 2012). Hence, the outcomes of the above-mentioned ecological studies highly rely on the accurate extraction of unfractionated water from different types of soils (Barbeta et al., 2019, 2020; Jiang et al., 2022; Orlowski, Breuer, et al., 2016, 2018; Orlowski, Pratt, et al., 2016; Orlowski, Winkler, et al., 2018; von Freyberg et al., 2020).

Laboratory-based techniques for soil water extraction have made formidable progress over the past three decades (Fischer et al., 2019; Orlowski et al., 2013; Revesz & Woods, 1990; West et al., 2006). These techniques can be roughly categorized into the following categories, namely, micro-distillation (Turner & Gailitis, 1988), azeotropic distillation (Ingraham & Shadel, 1992), cryogenic vacuum distillation (Araguás-Araguás et al., 1995), mechanical squeezing (Böttcher et al., 1997), centrifugation (Peters & Yakir, 2008), Helium (He) purging distillation (Ignatev et al., 2013), microwave extraction (Munksgaard et al., 2014), accelerated solvent extraction (Zhu et al., 2014) and headspace vapour equilibration (Millar et al., 2018; Wassenaar et al., 2008). Among these techniques, cryogenic vacuum distillation (referred to hereafter as CVD) is the most widely used in eco-hydrological studies (Koeniger et al., 2011; Oerter et al., 2014; Tsuruta et al., 2019). The main advantage of CVD relies on its effective extraction of liquid water from a small amount (~10 g) of soil samples. Moreover, the CVD technique has been unremittingly improved to increase its extraction efficiency and reduce the required labour-intensive manual manipulation (Fischer et al., 2019; Ignatev et al., 2013; Koeniger et al., 2011; Orlowski et al., 2013). For example, a new type of automatic cryogenic vacuum distillation (ACVD) system (LI-2100; Lica United Technology Limited Inc.) has been currently applied for soil water extraction (Liang et al., 2018; Qiu et al., 2019; Song et al., 2022). Though ACVD is automated and controlled by computer, the effectiveness of its extraction process is yet to be established.

The use of stable isotopes as tracers of plant water uptake is typically based on three prerequisites. First (P1), there are vertical gradients in water δ^2H and δ^{18}O within the soil profiles. The isotopic gradients in soil water are mainly caused by two independent processes, which are soil water evaporation and mixing between new and old water (e.g. rain, dew and fog) inputs (Dawson & Simonin, 2011; Gat, 1996). Second (P2), the dual-water isotopes of δ^2H and δ^{18}O would not fractionate during root water uptake and/or xylem redistribution of the source water (Ehleringer & Dawson, 1992; White et al., 1985). This assumption has been widely proved by previous studies apart from the halobiotic and xerophytic species (Ellsworth & Williams, 2007; Lin et al., 1993). Third (P3), the CVD technique is a robust method toward the 'truthful' isotope recovery of plant xylem and soil water (Newberry, Nelson, et al., 2017; West et al., 2006). Several studies have challenged the last prerequisite and showed isotopic biases for plant and soil materials (Barbeta et al., 2019; Chen et al., 2020; Meißner et al., 2014). The isotopic effects of CVD techniques will further complicate the quantification of source water contributions to plants (Orlowski, Breuer, et al., 2016; Yang et al., 2015). For instance, Chen et al. (2020) suspected that the TWW hypothesis (i.e. mature riparian trees do not utilize stream water) might result from the artefacts associated with xylem water cryogenic extraction. Therefore, the concept of soil water-excess (SW-excess) has been suggested to correct the isotopic biases of plant xylem water (Barbeta et al., 2019; Landwehr & Coplen, 2006; Li et al., 2021). However, the potential impacts of CVD on plant water source prediction have not been elucidated yet for soil water extraction. In addition, an accurate reflection of soil water δ^2H and δ^{18}O is also the basis of the SW-excess correction (Barbeta et al., 2020).

To fill the above-mentioned knowledge gaps, an inter-laboratory comparison of different CVD systems is required to test their performances on soil water extraction. We examined an automatic

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

cryogenic vacuum distillation (ACVD) and two traditional extraction (TCVD) systems. This study aims to identify the reliability of soil water CVD techniques and evaluate the isotopic effects of soil water extraction during the determination of plant water sources. We also introduced the SW-excess correction of Barbeta et al. (2019) and re-analysed the dual-water isotopes in plant xylem and soils across seven sites, including one grassland, two croplands, three plantations and one evergreen broadleaf forest in China. We hypothesized that (i) the ACVD and TCVD have different performances on recovering the soil water δ^2H and δ^{18}O, and (ii) the partitioning results of plant water sources will be affected by local environmental factors at the research sites.

2 | MATERIALS AND METHODS

2.1 | Overview

This study assessed the CVD systems for stable isotope analysis and plant water source prediction through the following experiments. Experiment (I): 225 soil samples containing various rates of clay particles were oven-dried, doped with deionized water, and cryogenically extracted using ACVD and TCVD systems. Experiment (II): a sensitivity analysis of plant water uptake under four types of input data was performed among seven sites. The National Natural Science Foundation of China granted us research permission (permit numbers: 32171557, 32171529).

2.2 | Description of two types of CVD systems

The automatic cryogenic vacuum distillation (ACVD) system (LI-2100; Lica United Technology Limited Inc.) consists of an electronic unit, a heating-cold trap unit and a vacuum manifold with 14 extraction lines (Figure S1). The vacuum pump (N84.3 AN-DC; KNF Neuberger Inc.) of ACVD can pump to a pressure of approximately 30 Pa (5 L min^{-1}). The heating unit can provide a maximum heating temperature of 130°C. During the xylem/soil water extraction process, the liquid water can be frozen at −95°C by an electronic cold trap (HG-95; TDJ Limited Inc.). Longer extraction times (~180 min) are recommended, because the solenoid valves (EV-2M-12-H-M5-V; Clippard Instrument Laboratory Inc.) will reduce the vacuum threshold (100–400 Pa) of the extraction lines. The ACVD system is installed in Institutional Center for Shared Technologies and Facilities of Xishuangbanna Tropical Botanical Garden (XTBG), Chinese Academy of Sciences.

The traditional mode of cryogenic vacuum distillation (TCVD) system consists of five independent extraction lines (West et al., 2006). The collection tubes are cooled by manually adding liquid nitrogen. The extraction time was visually determined for the traditional extraction system when no additional water vapour appeared in the collection tubes. The TCVD system generally performs better in terms of vacuum threshold (0.3–2 Pa) than ACVD (Orlowski et al., 2013; West et al., 2006). However, the performance of TCVD system can highly depend on the operator's experience and skills. Two TCVD systems are installed in the Technical Laboratory of LICA United Technology Limited (LICA) and the Key Laboratory of Northwest A & F University (NAFU).

Before formal water extraction, the extraction system should be evaluated to check its manifold tightness. The ACVD system can automatically check leakage according to a predetermined program. Within 10 min, the vacuum degrees of ACVD and TCVD systems must be lower than 600 and 8 Pa (i.e. the threshold for the leak-check). Once the xylem/soil samples are frozen in the extraction tubes, the extraction lines will be pumped into vacuum again. A certain amount of glass wool or fleece can be packed above the extracted samples to prevent the spread of soil particles (Orlowski et al., 2013; West et al., 2006). If the vacuum degree is better than the leak-check at this step, the extraction tubes will be heated until water vapour emanating from the samples is completely trapped in the collection tubes.

2.3 | Functionality test of CVD systems on soil water extraction

In Experiment (I), we selected three soil textures for rehydration treatments: sandy loam soil from Loess Plateau, loam soil from Northeast Plain and clay soil from Yunnan-Guizhou Plateau (Figure 1; Table 1). Soil textures were classified based on United States Department of Agriculture (USDA) classification procedures (Baillie, 2001). Each soil texture (~3 kg each) was homogenized, sieved (2 mm) and divided into 75 parallel sets (75 sets/samples × 3 sites/textures). Step by step, 15 soil samples were randomly picked from each of the three textures. Thus, we obtained five subsets of soil materials (5 subsets × 3 textures × 15 replications), which were pre-dried (105°C, 72 h) and shipped to the following laboratories: three sets to the Institutional Center for Shared Technologies and Facilities of XTBG, one set to the Technical Laboratory of LICA and one set to the Key Laboratory of NAFU. Simultaneously, 2.5 L of deionized (DI) water (δ^2H = −144.9 ± 0.5‰; δ^{18}O = −19.42 ± 0.12‰) was divided into quadrants, filled into plastic bottles (100 mL) and also delivered to these laboratories. The remaining bottle of DI water was kept as a standby reference. In each laboratory, the sub-samples of soils were packed into 10-mL screw-cap glass vials, oven-dried (105°C, 12 h) and doped with DI water in a decreasing order from 3.0 ± 0.6 to 1.2 ± 0.2 mL (SWC = ~10%–30%). Finally, the remoistened sub-samples were extracted using:

(i) ACVD system: soil water extraction was first conducted at 180 min, 85°C and ≤400 Pa (ACVD^{3H}). Then, the extraction time was extended to 240 min (ACVD^{4H}). Finally, the extraction time (240 min) and heating temperature (105°C) were both improved (ACVD^{4HT}).
(ii) TCVD system: soil water extractions were conducted at 105°C, and 0.3–8 Pa. Extraction times of 120–180 min were required

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

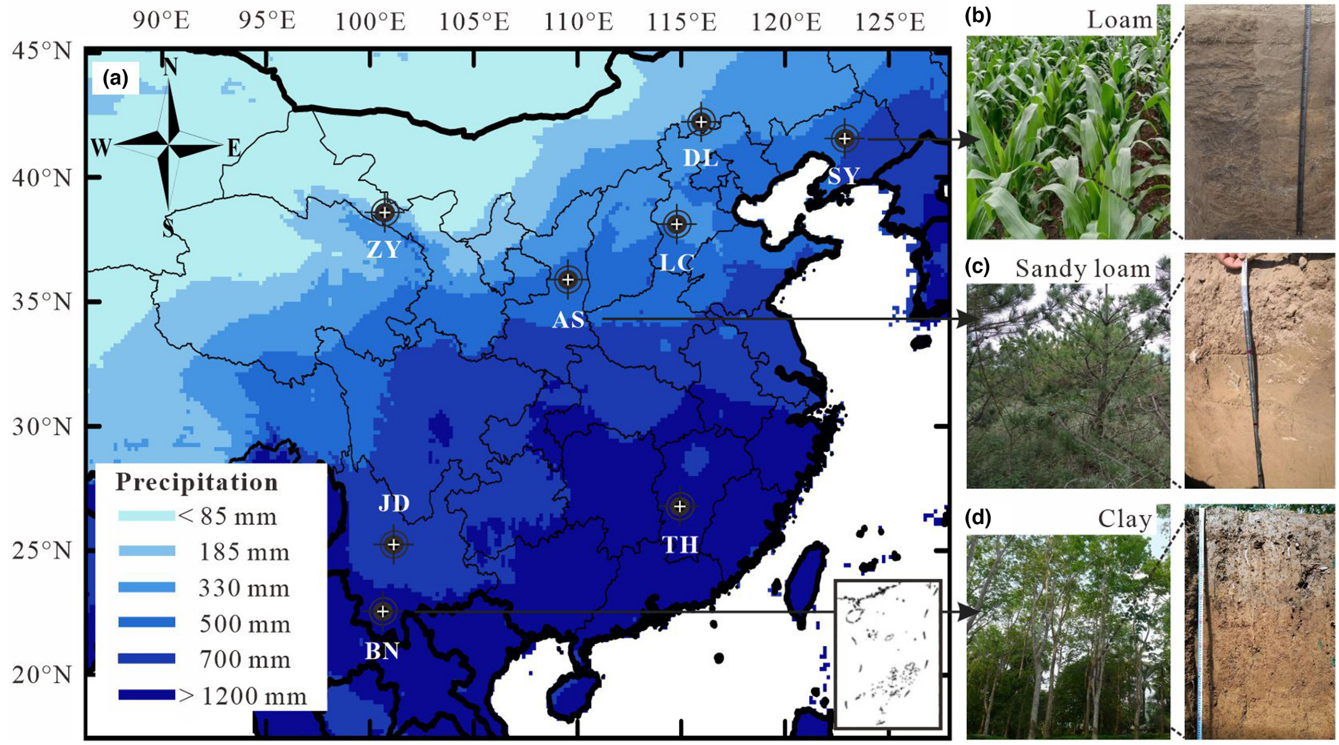

FIGURE 1 Map showing locations of eight experimental sites (a) and three soils for rehydration treatments (b–d) in this study. Data of precipitation is downloaded from the Global Climate Data (http://www.worldclim.org/).

TABLE 1 Location and physicochemical properties of three soil materials (AS: soil from Loess Plateau, SY: soil from Northeast Plain and BN: soil from Yunnan-Guizhou Plateau) used for rehydration experiment.

		Texture (%)			Bulk density		Total chemical element ($mg\,g^{-1}$)			
Site	Soil type	Sand	Silt	Clay	($g\,cm^{-3}$)	pH	Ca	Mg	Fe	Na
AS (36.51°N, 109.19°E)	Sandy loam	75.2	14.3	10.5	1.27–1.42	7.8	52.9 ± 0.7	11.3 ± 0.2	22.6 ± 0.2	13.8 ± 0.1
SY (41.98°N, 123.47°E)	Loam	41.9	35.1	23.0	1.18–1.27	6.2	1.5 ± 0.1	2.8 ± 0.1	14.5 ± 0.3	2.1 ± 0.1
BN (21.93°N, 101.27°E)	Clay	22.5	29.7	47.8	1.16–1.29	4.5	11.6 ± 0.3	9.2 ± 0.2	32.1 ± 0.7	17. 8 ± 0.6

in the Technical Laboratory of LICA (TCVD$^{1\#}$) and the Key Laboratory of NAFU (TCVD$^{2\#}$).

The water recovery rate (WRR) of the rehydration experiment was carefully determined (precision 0.0001 g) to check whether the DI water was completely removed from the soil materials (ME204; Mettler-Toledo Inc.):

$$\mathrm{WRR} = \frac{W_{\mathrm{wet}} - W_{\mathrm{extracted}}}{W_{\mathrm{DI\ water}}} \times 100\,\%, \tag{1}$$

where W_{wet} is the weight of remoistened soil (g), $W_{\mathrm{extracted}}$ is the weight of soil after extraction (g) and $W_{\mathrm{DI\ water}}$ is the initial weight of DI water (g). All samples were weighed after equalizing to room temperatures.

2.4 | Sensitivity analysis of CVD on plant water source prediction

In Experiment (II), we re-analysed the dual-water isotopes of seven sites across diverse vegetation types by considering the extraction-caused isotopic biases into plant water source prediction (Figure 1). The field sampling campaigns were conducted in a cropland (*Triticum aestivum* L.) in Luancheng (LC) County (Xiao et al., 2012), a grassland (*Stipa kryroii*) in Duolun (DL) County (Hu et al., 2014), a plantation (*Pinus massoniana* Lamb.) in Taihe (TH) County (Yang et al., 2015), a cropland (*Zea mays* L.) in Zhangye (ZY) City (Wen et al., 2016), a plantation (*Pinus tabuliformis*) in Ansai area (Tang et al., 2019), a plantation (*Hevea brasiliensi*) in Xishuangbanna (BN) Autonomous Prefecture (Yang et al., 2020) and an evergreen broadleaf forest (choosing one of the co-dominated canopy tree

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

species of *Castanopsis wattii* as a representative) in Jingdong (JD) County (Song et al., 2022). The soil cores of those sites were split into top soil (0–10 cm) and deep soil (10–200 cm), depending on the depth of the sampling campaigns. The nonphotosynthetic plant tissues (i.e. twigs for trees and root crowns for herbaceous species) were sampled simultaneously during the soil samplings. The field samples were extracted using the TCVD system for LC, DL, TH, ZY and AS (90–180 min, 105°C, and ≤8 Pa), and ACVD system for BN and JD (240 min, 105°C and ≤400 Pa).

Proportion contributions of the top and deep soil water to plants were estimated using a Bayesian mixing model of the MixSIAR package (Stock & Semmens, 2013). We entered four different types of input data into the MixSIAR model as follows: (i) raw dual-water isotopes of xylem and soil water, (ii) only soil water δ^2H and δ^{18}O corrected by isotopic effects of CVD (details in Appendix B), (iii) only xylem data corrected by SW-excess based on the raw soil water δ^2H and δ^{18}O and (iv) both xylem data corrected by SW-excess based on the corrected soil water δ^2H and δ^{18}O and soil data corrected by isotopic effects of CVD. The SW-excess correction was derived from the concept of line-conditioned excess (Landwehr & Coplen, 2006), which was used to quantify the isotopic biases of given xylem water with respect to the soil water line (Barbeta et al., 2019; Li et al., 2021):

$$\text{SW}-\text{excess}=\delta^{2}\text{H}_{\text{xylem}}-a_{\text{soil}}\times\delta^{18}\text{O}_{\text{xylem}}-b_{\text{soil}}, \tag{2}$$

where $\delta^2\text{H}_{\text{xylem}}$ and $\delta^{18}\text{O}_{\text{xylem}}$ are the dual-water isotopes of a xylem sample collected for a given site and date, a_{soil} and b_{soil} are the slope and intercept of the soil water line (SWL) at the same site, respectively.

We calculated the slope and intercept of SWL using linear model fitting of all the soil water δ^2H and δ^{18}O at the top and deep soil layers. A positive SW-excess indicates that ^{2}H of xylem water is more enriched than soil water, whereas a negative SW-excess suggests that xylem water is depleted than soil water (Barbeta et al., 2019). The greater the absolute value of SW-excess, the more significant the difference of isotopic offset between xylem water and its source water. This study corrected the raw xylem data by subtracting the corresponding SW-excess on each sampling date. Outputs of the MixSIAR model were finally assessed by the environmental factors, such as vapour pressure deficit (VPD) and soil water content (SWC), which were monitored by a suite of sensors for providing net radiation (CNR-1; Kipp and Zonen Inc.), soil water content (CS615-L/CS616-L; Campbell Scientific Inc.) and rainfall amount (52203, RM Young Inc.; TE525, Campbell Scientific Inc.).

2.5 | Measurement of stable isotopes of hydrogen and oxygen

In experiments (I), the δ^2H and δ^{18}O of water samples were measured using a Los Gatos Research Liquid Water Isotope Analyzer (DLT-100; Los Gatos Research Inc.). Because the isotopic ratio infrared spectroscopy (IRIS) can only measure the absolute ratios for isotopologues, it is necessary to address the unknown water samples to Vienna Standard Mean Ocean Water (VSMOW):

$$\delta_{\text{sample}}=\left(R_{\text{sample}}/R_{\text{VSMOW}}-1\right)\times1000, \tag{3}$$

where R_{sample} is the isotope ratio of a water sample and R_{VSMOW} is the isotope ratio of the VSMOW standard.

It is critical for the inter-laboratory comparison to have normalized water standards and measurement procedures (Coleman & Meier-Augenstein, 2014). Therefore, we pre-calibrated the laboratory water standards against the IAEA (International Atomic Energy Agency) water standards to the VSMOW. This procedure used two calibration standards ($\delta^2\text{H}=-165.7\pm0.5$‰, $\delta^{18}\text{O}=-21.28\pm0.15$‰; $\delta^2\text{H}=-9.9\pm0.5$‰, $\delta^{18}\text{O}=-2.99\pm0.15$‰) and a control ($\delta^2\text{H}=-49.2\pm0.5$‰, $\delta^{18}\text{O}=-7.81\pm0.15$‰). Measurements of the known δ-value standards and unknown water samples were interpolated employing a linear regression model. The control standard was used as an indicator of the analytical accuracy. In response to the spectral interference of methanol (NB)/ethanol (BB), the post-processing software (Spectral Contamination Identifier; Los Gatos Research Inc.) was used to identify the spectral contamination following the procedure of Schultz et al. (2011) and Xiao et al. (2012). The recision of the off-axis integrated cavity output spectroscopy (OA-ICOS) was ±0.6‰ for δ^2H and ±0.20‰ for δ^{18}O.

In experiment (II), the δ^2H and δ^{18}O of field water samples were measured using the isotopic ratio infrared spectroscopy (DLT-100; Los Gatos Research) for LC, DL, TH, ZY and AS (Hu et al., 2014; Tang et al., 2019; Wen et al., 2016; Xiao et al., 2012; Yang et al., 2015). The measuring procedures of IRIS were similar to those described above in experiments (I). For the BN and JD sites, measurements of water samples were conducted using a DELTA-V-Advantage isotope ratio mass spectrometer (Thermo Fisher Scientific) (Song et al., 2022; Yang et al., 2020). The isotope ratio mass spectrometer was coupled with a high-temperature conversion elemental analyser (HTC/EA) and an AS1310 auto-sampler (Thermo Fisher Scientific). According to our test results, the precision of the IRMS was ±0.4‰ for δ^2H and ±0.14‰ for δ^{18}O.

2.6 | Statistical analysis

The statistical analyses were performed with R software (Version 3.6.3; R Core Team, 2021), and the probability levels were $p\leq0.05$ and $p\leq0.001$. Shapiro–Wilk's normality test and Bartlett's homoscedasticity test were performed to check the normality and homogeneity of data. Welch's *t*-test and nonparametric Wilcoxon rank-sum test were then used for testing the significant differences in water δ^2H and δ^{18}O. A linear regression model was performed to compute the relationship between isotopic offsets on clay contents and CVD techniques, and we subsequently partitioned the variance contribution of the latter. To assess the relationship between source water contribution to plants and environmental factors, they were fitted using a generalized linear mixed model for each input data. The site and date were modelled as nested random effects. Meanwhile, 0 s and 1 s in source water contributions were removed before

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

logarithmic transformations. The model estimates were computed using 'NLME' package (Pinheiro et al., 2021; Stoffel et al., 2017).

3 | RESULTS

3.1 | Performance of cryogenic vacuum distillation (CVD) techniques

Both ACVD and TCVD techniques showed significant isotopic offsets between the extracted soil water and the spiked reference DI water (Figure 2). Mean δ^2H offsets of the ACVD system gradually changed from −5.8 ± 1.7‰ (ACVD^{3H}) to −3.8 ± 1.4‰ (ACVD^{4H}), and −2.6 ± 1.3‰ (ACVD^{4HT}) for the soil water extraction ($p < 0.001$). Similarly, mean δ^{18}O offsets of ACVD system fluctuated from −0.75 ± 0.23‰ (ACVD^{3H}) to −0.46 ± 0.29‰ (ACVD^{4H}) and −0.16 ± 0.14‰ (ACVD^{4HT}) ($p < 0.001$). However, the δ^{18}O offsets of ACVD extraction were negligible for the loam soils (Figure 2e; $p > 0.05$). The δ^2H offsets of the TCVD systems were −1.5 ± 1.8‰ (TCVD$^{1\#}$) and −3.4 ± 2.6‰ (TCVD$^{2\#}$) ($p \leq 0.05$), and their δ^{18}O offsets were −0.45 ± 0.57‰ (TCVD$^{1\#}$) and −0.34 ± 0.49‰ (TCVD$^{2\#}$) ($p \leq 0.05$). ACVD and TCVD systems could 'completely' extract soil water (Figure S2). Mean water recovery rate (WRR) was 98.68 ± 0.80% (ACVD^{3H}), 99.84 ± 0.34% (ACVD^{4H}) and 99.86 ± 0.30% (ACVD^{4HT}) for the ACVD system, and 99.06 ± 1.00% (TCVD$^{1\#}$) and 99.24 ± 0.92% (TCVD$^{2\#}$) for the TCVD system, respectively.

3.2 | Effects of soil properties on soil water isotopes

The isotopic offsets of CVD techniques were influenced by soil textures and soil moisture conditions (Figure 3). Here, only the

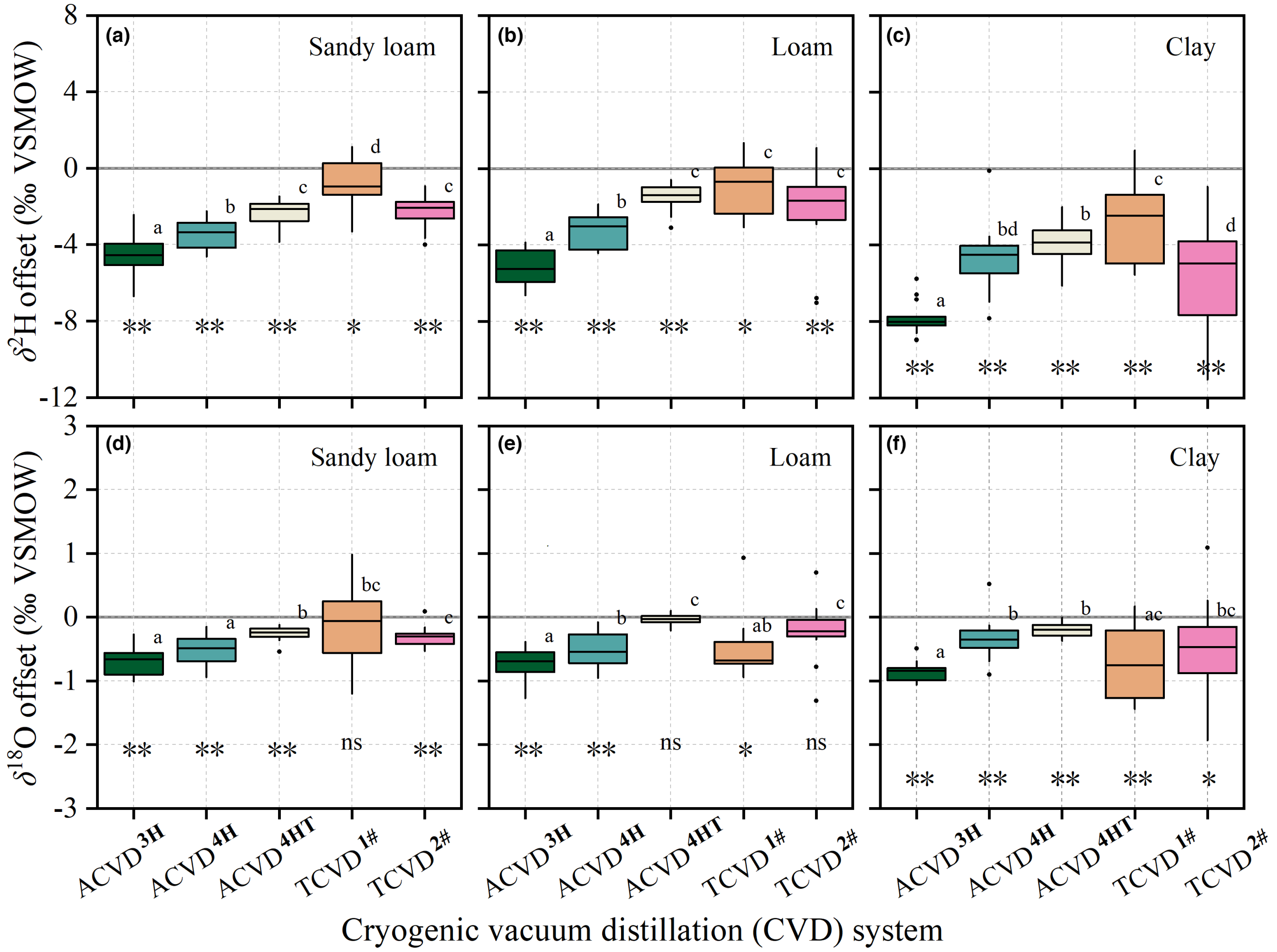

FIGURE 2 Hydrogen (a–c) and oxygen (d–f) isotope offsets between deionized (DI) water and the same water cryogenically extracted from rehydrated soils (clay loam, loam, and clay, $n = 15$). Isotopic offsets are differences between the extracted soil water and the spiked DI water ($\delta^2H/^{18}O_{\text{Soil water}} - \delta^2H/^{18}O_{\text{DI water}}$). ACVD: automatic cryogenic vacuum distillation (3H: 85°C and 180 min; 4H: 85°C and 240 min; 4HT: 105°C and 240 min). TCVD: traditional cryogenic vacuum distillation (1# and 2#: 105°C and 120 min in two laboratories). Box size represents the interquartile range, whiskers indicate variability outside the upper and lower quartiles, black lines are the medians, and individual points are outliers. Box values not sharing the same letters are significantly different ($p < 0.05$). The presence of an asterisk below a box indicates that the value is significantly different from zero (ns: $p > 0.05$, *$p < 0.05$, **$p < 0.001$).

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

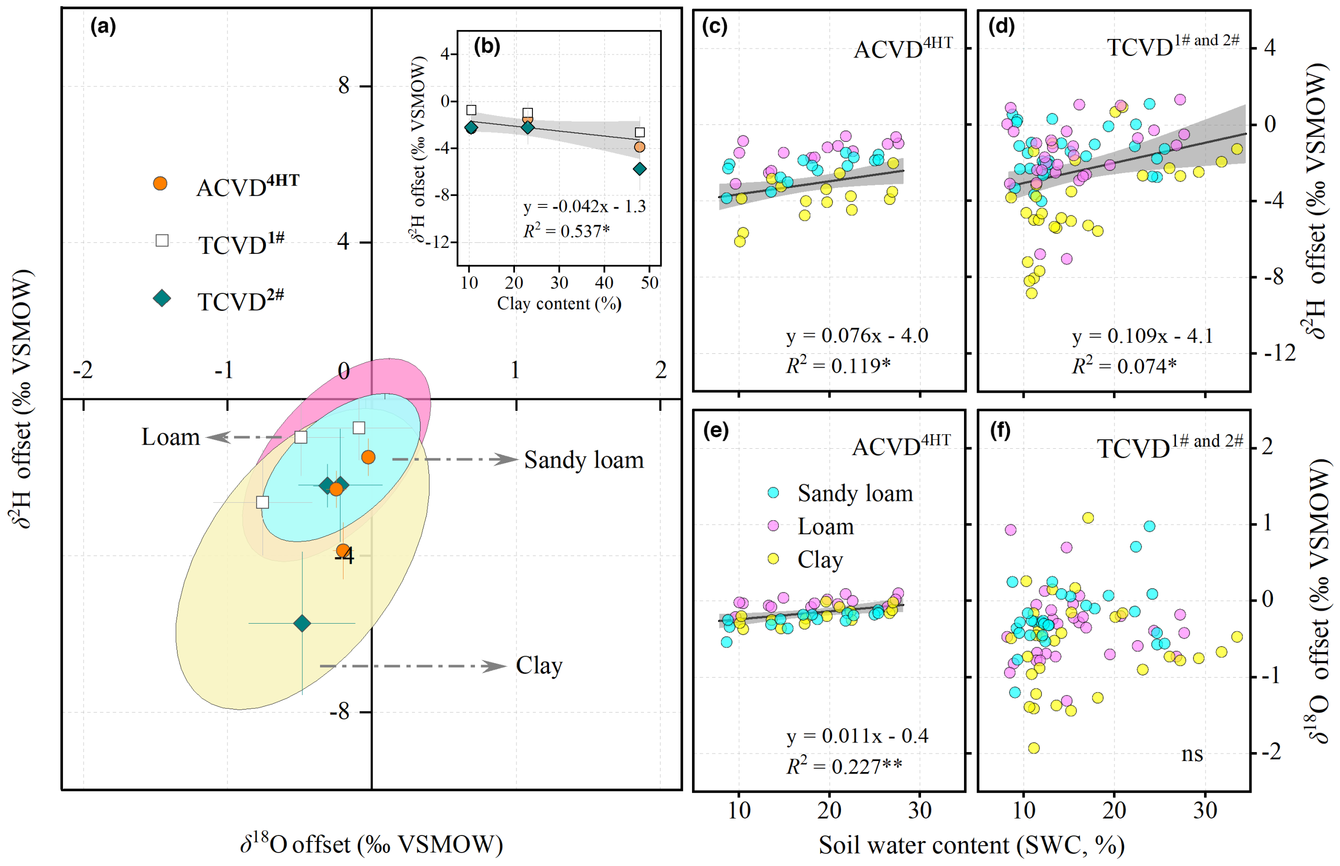

FIGURE 3 Soil water δ^{2}H offsets as a function of soil water δ^{18}O offsets (a) for two types of cryogenic vacuum distillation systems (n = 15). Confidence ellipses and light grey shaded areas indicate the 95% prediction areas and 95% confidence intervals of linear regressions, respectively. The insert panel depicts the relationship between soil water δ^{2}H offsets and soil clay content (b) for automatic cryogenic vacuum distillation (ACVD: 105°C and 240 min) and traditional cryogenic vacuum distillation (TCVD$^{1\#}$ and TCVD$^{2\#}$: 105°C and 120 min in two laboratories). Relationships of soil water δ^{2}H (c, d) and δ^{18}O (e, f) offsets to soil water content (SWC) are also presented in (c)–(f) (ns: $p > 0.05$, *$p < 0.05$, **$p < 0.001$).

results of ACVD^{4HT} were used for further comparisons with TCVD$^{1\#}$ and TCVD$^{2\#}$. The isotopic offsets of ACVD^{3H} and ACVD^{4H} were higher than those of the other treatments under lower extraction temperatures. All the CVD systems tended to produce more isotopic offsets for soils with higher clay contents (Figure 3a,b). The isotopic offsets were positively correlated with soil clay content for δ^{2}H ($R^2 = 0.537$, $p < 0.05$) and δ^{18}O ($R^2 = 0.055$, $p < 0.05$, data not shown). Furthermore, soil clay content explained more variation (29.4%) in δ^{2}H offsets than in CVD techniques (13.9%). The ^{2}H of ACVD extracted soil water (i.e. ACVD^{4HT}) was depleted by −2.3 ± 1.0‰ for sandy loam, −1.5 ± 0.7‰ for loam soil and −3.9 ± 1.0‰ for clay soil, and those of TCVD extracted soil water (i.e. the average value of TCVD$^{1\#}$ and TCVD$^{2\#}$) were depleted by −1.5 ± 0.8‰, −1.6 ± 1.3‰, and −4.2 ± 1.4‰ for sandy loam, loam and clay soil, respectively. No significant relationship occurred between ^{18}O offsets of extracted soil water and soil clay contents, which were depleted by −0.02 ± 0.08‰ to −0.25 ± 0.11‰ (ACVD) and −0.20 ± 0.28‰ to −0.62 ± 0.28‰ (TCVD) for different soil textures.

The isotopic offsets of CVD techniques were positively correlated with soil moisture condition ($p < 0.05$). The extraction stability of ACVD was superior remarkably to TCVD systems (Figure 3c–f), though the two types of CVD systems have approximately equal offsets for soil water δ^{2}H and δ^{18}O. Mean δ^{2}H offsets of extracted soil water were −2.6 ± 1.3‰ for ACVD and −2.4 ± 1.7‰ for TCVD, while those of δ^{18}O offsets were −0.16 ± 0.14‰ and −0.39 ± 0.37‰ for ACVD and TCVD. Positive correlations occurred between δ^{2}H offsets and soil water content (SWC) for both two types of CVD systems ($p < 0.05$). Moreover, a positive relationship existed between δ^{18}O offsets of ACVD and SWC ($p < 0.001$), while no significant correlation was found between δ^{18}O offsets of TCVD and SWC (Figure 3f). Based on these results, the isotopic offsets of ACVD and TCVD techniques were further used to correct the cryogenic extracted soil water δ^{2}H and δ^{18}O of different experimental sites.

3.3 | Impacts of CVD techniques on plant water source prediction

Soil water δ^{2}H and δ^{18}O of different sites occupied the dual-isotope plots from the LWMLs to their right sides (Figure 4). The slopes of the

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

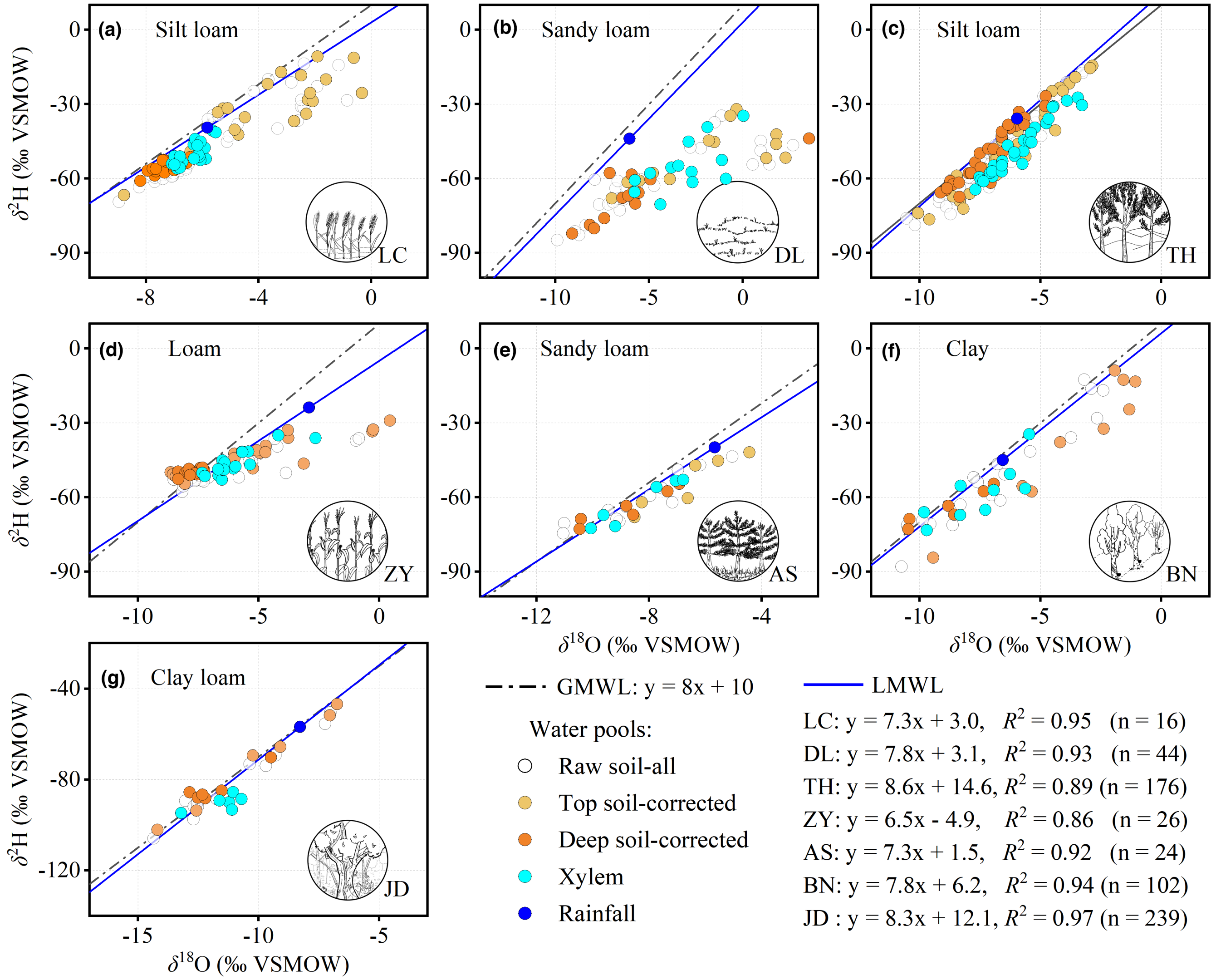

FIGURE 4 Dual-isotope (δ^2H and δ^{18}O) plots of xylem water at seven experimental sites and their potential sources of soil water corrected based on isotopic offsets of cryogenic vacuum distillation (CVD) techniques. The raw data of soil water δ^2H and δ^{18}O and mean rainwater δ^2H and δ^{18}O (n = 16–239) are also plotted on each panel for reference. The dashed black line indicates the global meteoric water line (GMWL), whereas the blue line indicates the local meteoric water line (LWML) at each site.

LMWLs increased gradually from 6.5 to 8.6, reflecting the climate characteristics of the study sites from inland oasis (ZY) to coastal hills (TH). Most of xylem water fell within the δ^2H–δ^{18}O spaces of soil water. However, some part of xylem water δ^2H and δ^{18}O distributed besides the ranges of soil water. Thus, raw soil water data were corrected based on the isotopic correction equations (details in Appendix B):

(i) ACVD system:

$$\delta^2\mathrm{H}_{\mathrm{offset}} = -2.7 - 1.2\times\mathrm{SCC} - 1.5\times\mathrm{SWC}, \tag{4}$$

$$\delta^{18}\mathrm{O}_{\mathrm{offset}} = -0.78 + 3.94\times\mathrm{SCC} - 11.58\times\mathrm{SCC}^2 + 1.07\times\mathrm{SWC}, \tag{5}$$

(ii) TCVD system:

$$\delta^2\mathrm{H}_{\mathrm{offset}} = -0.2 - 15.0\times\mathrm{SCC} + 13.6\times\mathrm{SCC}\times\mathrm{SWC}, \tag{6}$$

$$\delta^{18}\mathrm{O}_{\mathrm{offset}} = -0.29 - 4.08\times\mathrm{SCC} + 15.49\times\mathrm{SCC}\times\mathrm{SWC}, \tag{7}$$

where $\delta^2\mathrm{H}_{\mathrm{offset}}$ and $\delta^{18}\mathrm{O}_{\mathrm{offset}}$ are the potential isotopic offsets of soil water extraction caused by TCVD and ACVD systems, SCC and SWC are the soil clay contents (%) and soil water content (%), respectively.

Mean corrections of soil water δ^2H and δ^{18}O were −2.7±0.1‰ and −0.21±0.07‰ for LC, −2.6±0.1‰ and −0.79±0.07‰ for DL, −1.9±0.1‰ and −0.23±0.06‰ for TH, −3.1±0.3‰ and −0.10±0.23‰ for ZY, −1.7±0.1‰ and −0.60±0.03‰ for AS, −3.6±0.1‰ and −1.32±0.03‰ for BN, and −3.7±0.1‰ and −0.27±0.06‰ for JD.

The isotopic biases between xylem and soil water were assessed by calculating the SW-excess of raw soil data and corrected soil data (Figure 5). Based on the raw soil data, the SW-excess of xylem water samples were 0.5±3.1‰ for LC, 1.5±7.8‰ for DL, −6.6±2.3‰ for TH, 2.1±3.0‰ for ZY, −0.8±3.1‰ for AS, −11.7±7.8‰ for BN and

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

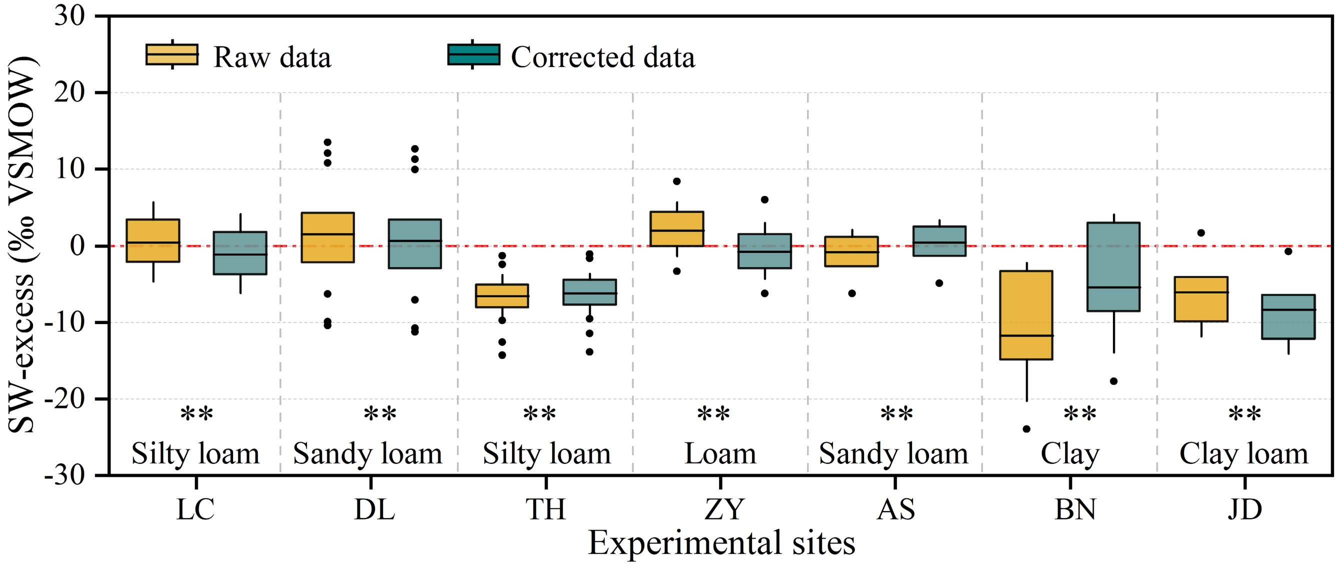

FIGURE 5 Variations of xylem water SW-excess at seven experimental sites. Box size represents the interquartile range, whiskers indicate variability outside the upper and lower quartiles, black lines is the median, and individual points are outliers. The presence of an asterisk below a box indicates that the difference is significant between pairs of data (ns: $p > 0.05$, *$p < 0.05$, **$p < 0.001$).

TABLE 2 Parameter estimates of fixed effects and *R* squares of the generalized linear mixed models on source water contributions for each input data. VPD is vapour pressure deficit and SWC is soil water content. Bold numbers are the largest estimates of fixed effects (i.e. VPD and SWC at two soil depths) and marginal *R* squares (reflecting variances in source water contributions explained by independent fixed variables) for each model.

Input data	Source water	VPD (kPa)	SWC of top soil (%)	SWC of deep soil (%)	Marginal R^2
Raw data	Top soil	**0.565**	0.117	0.122	0.008
	Deep soil	−2.835	−1.261	0.603	**0.078**
Only soil data corrected	Top soil	−0.276	−2.251	1.353	0.016
	Deep soil	0.963	**6.467**	−2.621	**0.097**
Only xylem data corrected	Top soil	**1.371**	−1.038	0.459	**0.081**
	Deep soil	−0.215	−0.626	0.423	0.013
Both xylem and soil corrected	Top soil	−0.089	−0.817	−1.038	**0.183**
	Deep soil	0.791	**2.195**	−1.737	0.039

−6.1 ± 4.8‰ for JD (Table S1). Generally, the SW-excess decreased (more negative) with the increasing soil clay content (ranging from 10.5% to 47%) of the experimental sites. However, the SW-excess values, using the soil data corrected by CVD offsets, were −1.1 ± 3.1‰ for LC, 0.7 ± 7.8‰ for DL, −6.2 ± 2.4‰ for TH, −0.7 ± 3.2‰ for ZY, 0.5 ± 3.1‰ for AS, −5.4 ± 7.8‰ for BN and −8.3 ± 4.8‰ for JD. The effectiveness of CVD offset correction was reflected by the relationships between the MixSIAR outputs and environmental factors (Table 2). Models using xylem data corrected by SW-excess and soil data corrected by CVD offsets exhibited strong correlation with environmental factors (marginal $R^2 = 0.183$), indicating a plausible correction of CVD offsets on soil water δ^2H and δ^{18}O. Surprisingly, the use of corrected soil data only (marginal $R^2 = 0.097$) performed better results than using corrected xylem data only (marginal $R^2 = 0.081$). However, all the models using corrected data (i.e. SW-excess and/or CVD offset correction) reasonably predicted plant water uptake depths than the input of raw data (marginal $R^2 = 0.078$). The potential isotopic biases in plant xylem and soil water could alter the definition of principal environmental factors that related to plant water uptake.

4 | DISCUSSION

This study investigates the reliability of two laboratory-based CVD techniques for soil water extraction. To avoid incomplete extraction caused by artificial manipulations, the amounts of the input water into soil materials were strictly tracked before and after extraction. Theoretically, an incomplete extraction leads to more negative δ-values in the collected water (Orlowski et al., 2013). Based on the Rayleigh distillation mechanisms, the δ^2H and δ^{18}O of the removed water will be 'lighter' than the remaining fraction (Gat, 1996; Lee et al., 2005). A small leakage in the vacuum system may also cause the condensation of laboratory ambient air moisture in the extraction lines, which could commonly conduct to more negative δ^2H and δ^{18}O of extracted water. Here, outputs of the spectral-contamination-identifier were 0.0614 ± 0.0089 for methanol (NB) and 1.0001 ± 0.0002 for ethanol (BB) interferences. This spectral contamination probably conducts to more positive soil water δ^2H (0.14 ± 0.15‰) and δ^{18}O (0.07 ± 0.09‰), which was not considered because these isotopic errors were smaller than the measurement

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

precisions of the OA-ICOS. The WRR of our extractions generally exceeded 98% (Figure S2), which was sufficient to obtain unfractionated water samples (Araguás-Araguás et al., 1995; West et al., 2006). Moreover, the WRR of ACVD and TCVD systems occasionally exceeds 100% (100.02%–100.67%), as shown the results of Meißner et al. (2014) and Orlowski, Breuer, et al. (2018), where WRR higher (>100%) are produced during soil water extraction. A time gap (1–2 h) occurred between the CVD extractions and weighing procedures, suggesting that the soil samples would inevitably absorb small amount of ambient water vapour. The slight difference in weight (0.0092 ± 0.0058 g) reflected that CVD systems had better drying performance than ovens under similar working temperatures (~105°C). Such weighing errors will not affect the δ^2H and δ^{18}O of extracted soil water (Araguás-Araguás et al., 1995; Meißner et al., 2014).

The CVD techniques are usually recognized as a standard for soil water δ^2H and δ^{18}O analyses (Koeniger et al., 2011; Munksgaard et al., 2014; Newberry, Nelson, et al., 2017; West et al., 2006). However, the use of CVD techniques can be associated with potential isotopic biases, since a controversy pertains regarding CVD recovering the isotopic signatures of the added water. As previous findings, our results also showed that unfractionated pure water was obtained using the ACVD system (Figure S3). The extraction errors of δ^2H (−0.1 ± 0.3‰) and δ^{18}O (0.04 ± 0.09‰) were similar to the IRMS/IRIS measuring precision. However, the CVD systems depleted the soil water ^{2}H and ^{18}O (Figure 2). This finding is in agreement with other studies (Araguás-Araguás et al., 1995; Meißner et al., 2014; Orlowski et al., 2013; Orlowski, Breuer, et al., 2016; Orlowski, Pratt, et al., 2016; Thielemann et al., 2019; Tsuruta et al., 2019; Turner & Gailitis, 1988). For example, the inter-laboratory comparisons exhibit unacceptable uncertainties (up to −108.4‰ for δ^2H, and −14.9‰ for δ^{18}O) during soil water extraction (Orlowski, Breuer, et al., 2018; Walker et al., 1994).

Under a longer extraction time (240 min), the ACVD system performed equally or even better than TCVD systems (Figure 3). In fact, the constant δ^2H and δ^{18}O of extracts can still be inconsistent with their 'actual values' (Orlowski et al., 2013; Orlowski, Winkler, et al., 2018), although a minimum time of 30–40 min is sufficient for field sample extraction (West et al., 2006). The results also showed that the isotopic offsets of ACVD decreased with the increasing extraction temperature (85–105°C), highlighting the importance of extraction temperature for a low vacuum extraction system. The dependence of isotopic bias on the extraction temperature has also previously been reported by Walker et al. (1994), Araguás-Araguás et al. (1995) and Palacio et al. (2014). Higher extraction temperature (100–150°C) will speed up the rate at which extraction is completed; however, it might also decompose the soil organic matter and release crystallized water (Walker et al., 1994). Orlowski, Winkler, et al. (2018) still could not recover the isotopic values of added water with a high (200°C) extraction temperature.

The stagnant bound water in soil aggregates is one of the most predominant problems causing extraction failures (Gaj et al., 2017; Newberry, Prechsl, et al., 2017; Orlowski, Breuer, et al., 2016). In this study, SWC and isotopic deviations exhibited a weak relationship (Figure 3), due to the influence of a relatively high proportion of heavily bound water to the dry soil (Araguás-Araguás et al., 1995; Orlowski, Pratt, et al., 2016). The isotopic effects of this heat-labile water were more important when the tightly bound water composed large fraction of the total added water (Ingraham & Shadel, 1992). Our results also supported this conclusion since the ^{2}H and ^{18}O of extracted water were more depleted for clay soils (Figure 3a,b), which commonly have a high amount of bound water (Araguás-Araguás et al., 1995; Koeniger et al., 2011; Orlowski, Breuer, et al., 2018). In our rehydration experiment, the soil materials were collected from the Loess Plateau (AS), Northeast Plain (SY) and Yunnan-Guizhou Plateau (BN). While the exchange effect between soil-bound water and spiked water is an essential process for the rehydration experiment, we cannot exclude the influence that may derive from soil materials with very different isotopic compositions. According to our measurements and the results from previous studies (Tang et al., 2019; Yang et al., 2020), the δ^2H and δ^{18}O in precipitation were −41.2 ± 18.7‰ and −5.67 ± 2.49‰ for AS (n = 24), −57.9 ± 22.5‰ and −8.65 ± 2.84‰ for SY (n = 16), and −44.9 ± 33.0‰ and −6.56 ± 4.23‰ for BN (n = 102). Mean soil water δ^2H and δ^{18}O in the 0–60 cm depths were −64.9 ± 11.0‰ and −9.22 ± 2.13‰ for AS (n = 49), −61.7 ± 5.7‰ and −7.89 ± 1.12‰ for SY (n = 36), and −60.0 ± 18.7‰ and −8.09 ± 2.36‰ for BN (n = 36). These findings could primary infer that isotopic background values were not the key driving factors behind the depleted water for clay soils, because the soil water (including precipitation) δ^2H and δ^{18}O of BN generally varied within the ranges of AS and SY sites. However, the soil materials should be rewetted with repeated addition of the spiked water to diminish the possible background influences (Newberry, Prechsl, et al., 2017; Orlowski, Pratt, et al., 2016; Thielemann et al., 2019). If more spiking waters with two strongly different isotopic compositions are used in the rehydration experiments, it will also increase the confidence in assessing the soil-bound water effects. Therefore, a better understanding of the isotopic exchange effect between soil-bound water and spiked water is urgently needed in future research.

Nowadays, several studies highlighted the substantial 'unexplained' uncertainties concerning isotope-based plant water source partitioning (Barbeta et al., 2019; Li et al., 2007; Meißner et al., 2014; Orlowski et al., 2013; Yang et al., 2015). Therefore, one should pay attention to the isotopic offsets of CVD techniques. Our results showed that the ACVD and TCVD systems resulted in noticeable biases in soil water δ^2H and δ^{18}O (Figure 3). The isotopic offsets of CVD might vary significantly across study locations and laboratories (Orlowski, Breuer, et al., 2018; Orlowski, Pratt, et al., 2016). Here, among the seven experimental sites in China, the isotopic offsets of CVD systems ranged from −3.7‰ to −1.7‰ for soil water δ^2H, and from −0.27‰ to −0.10‰ for soil water δ^{18}O (Figure 4). Using soil data corrected by CVD offsets improved the prediction of plant water sources according to its correction with environmental factors (Table 2). The SW-excess can also be used to reduce the possible impacts of different isotopic biases in plant water source partitioning

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

(Barbeta et al., 2019; Li et al., 2021). Therefore, the effective use of SW-excess would highly depend on the accurate extraction of unfractionated water from different types of soils (Figure 5). The sensitivity analysis of this study suggested that the isotopic biases of CVD systems would lead to a high risk of miscalculating the water uptake fractions (e.g. at BN and JD sites). These findings suggest that one should pay more attention for examining and optimizing CVD techniques for ecohydrological studies.

5 | CONCLUSIONS

The present study assessed the reliability of CVD techniques on soil water extraction, and evaluated the potential influences of cryogenic extraction on plant water source partitioning. Our results showed that the newly designed ACVD technique could have similar or better performance compared with TCVD system. However, the two CVD techniques did not achieve unfractionated soil water δ^2H and δ^{18}O in laboratory. Meanwhile, the isotopic offsets of CVD techniques were correlated with soil clay contents and soil water contents of the extracted soil materials. The wide use of CVD techniques probably induced noticeable uncertainties in predicting plant water uptake depths. Sensitivity analysis showed that the use of soil data corrected by CVD offsets improved the prediction performance on plant water uptake. The isotopic offsets of CVD techniques may introduce contradictory findings of plant water uptake, which is originating from the artefacts in laboratory soil water extraction. These findings will have important implications for further studies, which can provide a new solution for promoting the prediction of plant water sources. More research on the isotopic exchange effect of soil-bound water is recommended in the future.

AUTHOR CONTRIBUTIONS

Bin Yang and Liang Song conceived the research ideas, designed the methodology and led the draft writing with inputs from other co-authors. Wen-Jie Liu, Gbadamassi G.O. Dossa, Yue-Hua Hu, and Yong-Jiang Zhang were involved in data interpretation. Sissou Zakari helped to polish the language of the manuscript. All the co-authors contributed critically to the present work and gave final approval for publication.

ACKNOWLEDGEMENTS

We are thankful to Xue-Fa Wen, Ya-Kun Tang, Bin Chen, Jie Zhao and Corlett Richard for providing scientific data and suggestions. We further acknowledge the supports from the Institutional Center for Shared Technologies and Facilities of Xishuangbanna Tropical Botanical Garden, CAS. This work was funded by the National Natural Science Foundation of China (32171557, 32171529), the Yunnan Fundamental Research Projects (202201AT070221, 202101AS070010), the Youth Innovation Promotion Association of CAS (2021397), the Candidates of the Young and Middle Aged Academic Leaders of Yunnan Province (2019HB040) and the Yunnan High Level Talents Special Support Plan (YNWR-QNBJ-2020-066).

CONFLICT OF INTEREST STATEMENT

The authors declare that there are no known competing financial interests or personal relationships that will influence the current work reported in this paper.

PEER REVIEW

The peer review history for this article is available at https://www.webofscience.com/api/gateway/wos/peer-review/10.1111/2041-210X.14107.

DATA AVAILABILITY STATEMENT

Data for this study consist of water isotopes and environmental variables that are available at Dryad Digital Repository https://doi.org/10.5061/dryad.fxpnvx0w1 (Yang et al., 2023).

ORCID

Bin Yang https://orcid.org/0000-0003-1893-2562
Gbadamassi G. O. Dossa https://orcid.org/0000-0001-9767-7306
Yue-Hua Hu https://orcid.org/0000-0003-3027-1410
Lu-Lu Liu https://orcid.org/0000-0003-2194-4556
Xian-Jing Meng https://orcid.org/0000-0001-7261-7403
Yi-Yuan Du https://orcid.org/0000-0003-0759-6460
Jia-Yuan Li https://orcid.org/0000-0003-0945-5688
Xi-Ai Zhu https://orcid.org/0000-0003-1501-2322
Yong-Jiang Zhang https://orcid.org/0000-0001-5637-3015
Ashutosh K. Singh https://orcid.org/0000-0003-2616-0697
Xia Yuan https://orcid.org/0000-0002-5950-7856
Jun-En Wu https://orcid.org/0000-0002-5240-2314
Sissou Zakari https://orcid.org/0000-0002-0062-5997
Wen-Jie Liu https://orcid.org/0000-0002-9918-3462
Liang Song https://orcid.org/0000-0002-1452-9939

REFERENCES

Araguás-Araguás, L., Rozanski, K., Gonfiantini, R., & Louvat, D. (1995). Isotope effects accompanying vacuum extraction of soil water for stable isotope analyses. *Journal of Hydrology*, *168*(1–4), 159–171. https://doi.org/10.1016/0022-1694(94)02636-P

Baillie, I. C. (2001). Book review 'soil survey staff 1999, soil taxonomy—A basic system of soil classification for making and interpreting soil surveys; 2nd edition. Agriculture handbook 436; Natural Resources Conservation Service, USDA, Washington, DC, USA, pp. 869'. *Soil Use and Management*, *17*, 57–60. https://doi.org/10.1111/j.1475-2743.2001.tb00008.x

Barbeta, A., Gimeno, T. E., Clavé, L., Fréjaville, B., Jones, S. P., Delvigne, C., Wingate, L., & Ogée, J. (2020). An explanation for the isotopic offset between soil and stemwater in a temperate tree species. *New Phytologist*, *227*(3), 766–779. https://doi.org/10.1111/nph.16564

Barbeta, A., Jones, S. P., Clavé, L., Wingate, L., Gimeno, T. E., Fréjaville, B., Wohl, S., & Ogée, J. (2019). Unexplained hydrogen isotope offsets complicate the identification and quantification of tree water sources in a riparian forest. *Hydrology Earth System Scinces*, *23*(4), 2129–2146. https://doi.org/10.5194/hess-23-2129-2019

Böttcher, G., Brumsack, H. J., Heinrichs, H., & Pohlmann, M. (1997). A new high pressure squeezing technique for pore fluid extraction from terrestrial soils. *Water, Air, & Soil Pollution*, *94*(3–4), 289–296. https://doi.org/10.1023/A:1026499319006

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

Brooks, J. R., Barnard, H. R., Coulombe, R., & McDonnell, J. J. (2010). Ecohydrologic separation of water between trees and streams in a Mediterranean climate. *Nature Geoscince*, *3*, 100–104. https://doi.org/10.1038/ngeo722

Chen, Y. L., Helliker, B. R., Tang, X. H., Li, F., Zhou, Y. P., & Song, X. (2020). Stem water cryogenic extraction biases estimation in deuterium isotope composition of plant source water. *Proceedings of the National Academy of Sciences of the United States of America*, *118*(17), 33345–33350. https://doi.org/10.1073/pnas.2014422117

Coleman, M., & Meier-Augenstein, W. (2014). Ignoring IUPAC guidelines for measurement and reporting of stable isotope abundance values affects us all. *Rapid Communications in Mass Spectrometry*, *28*(17), 1953–1955. https://doi.org/10.1002/rcm.6971

Coplen, T. B. (2011). Guidelines and recommended terms for expression of stable-isotope-ratio and gas-ratio measurement results. *Rapid Communications in Mass Spectrometry*, *25*(17), 2538–2560. https://doi.org/10.1002/rcm.5129

Dawson, T. E., & Simonin, K. A. (2011). The roles of stable isotopes in forest hydrology and biogeochemistry. In D. F. Levia, D. Carlyle-Moses, & T. Tanaka (Eds.), *Forest hydrology and biogeochemistry: Synthesis of past research and future directions* (pp. 137–161). Springer. https://doi.org/10.1007/978-94-007-1363-5_7

Ding, Y. L., Nie, Y. P., Schwinning, S., Chen, H. S., Yang, J., Zhang, W., & Wang, K. L. (2018). A novel approach for estimating groundwater use by plants in rock-dominated habitats. *Journal of Hydrology*, *565*, 760–769. https://doi.org/10.1016/j.jhydrol.2018.08.033

Ehleringer, J. R., & Dawson, T. E. (1992). Water uptake by plants: Perspectives from stable isotope composition. *Plant, Cell & Environment*, *15*(9), 1073–1082. https://doi.org/10.1111/j.1365-3040.1992.tb01657.x

Ellsworth, P. Z., & Williams, D. G. (2007). Hydrogen isotope fractionation during water uptake by woody xerophytes. *Plant and Soil*, *291*, 93–107. https://doi.org/10.1007/s11104-006-9177-1

Fischer, B. M. C., Frentress, J., Manzoni, S., Cousins, S. A. O., Hugelius, G., Greger, M., Smittenberg, R. H., & Lyon, S. W. (2019). Mojito, anyone? An exploration of low-tech plant water extraction methods for isotopic analysis using locally-sourced materials. *Frontiers in Earth Science*, *7*. https://doi.org/10.3389/feart.2019.00150

Gaj, M., Kaufhold, S., Koeniger, P., Beyer, M., Weiler, M., & Himmelsbach, T. (2017). Mineral mediated isotope fractionation of soil water. *Rapid Communications in Mass Spectrometry*, *31*(3), 269–280. https://doi.org/10.1002/rcm.7787

Gat, J. R. (1996). Oxygen and hydrogen isotopes in the hydrologic cycle. *Annual Review of Earth and Planetary Sciences*, *24*, 225–262. https://doi.org/10.1146/annurev.earth.24.1.225

Gehre, M., Geilmann, H., Richter, J., Werner, R. A., & Brand, W. A. (2004). Continuous flow ^{2}H/^{1}H and ^{18}O/^{16}O analysis of water samples with dual inlet precision. *Rapid Communications in Mass Spectrometry*, *18*(22), 2650–2660. https://doi.org/10.1002/rcm.1672

Good, S. P., Noone, D., & Bowen, G. (2015). Hydrologic connectivity constrains partitioning of global terrestrial water fluxes. *Science*, *349*(6244), 175–177. https://doi.org/10.1126/science.aaa5931

Griffis, T. J. (2013). Tracing the flow of carbon dioxide and water vapor between the biosphere and atmosphere: A review of optical isotope techniques and their application. *Agricultural and Forest Meteorology*, *174–175*, 85–109. https://doi.org/10.1016/j.agrformet.2013.02.009

Grossiord, C., Sevanto, S., Dawson, T. E., Adams, H. D., Collins, A. D., Dickman, L. T., Newman, B. D., Stockton, E. A., & McDowell, N. G. (2017). Warming combined with more extreme precipitation regimes modifies the water sources used by trees. *New Phytologist*, *213*(2), 584–596. https://doi.org/10.1111/nph.14192

Hu, Z. M., Wen, X. F., Sun, X. M., Li, S. G., Li, L. H., Yu, G. R., & Lee, X. H. (2014). Partitioning of evapotranspiration through oxygen isotopic measurements of water pools and fluxes in a temperate grassland. *Journal of Geophysical Resesearch: Biogeosciences*, *119*(3), 358–371. https://doi.org/10.1002/2013JG002367

Ignatev, A., Velivetckaia, T., Sugimoto, A., & Ueta, A. (2013). A soil water distillation technique using He-purging for stable isotope analysis. *Journal of Hydrology*, *498*, 265–273. https://doi.org/10.1016/j.jhydrol.2013.06.032

Ingraham, N. L., & Shadel, C. (1992). A comparison of the toluene distillation and vacuum/heat methods for extracting soil water for stable isotopic analysis. *Journal of Hydrology*, *140*(1–4), 371–387. https://doi.org/10.1016/0022-1694(92)90249-U

Jiang, N. N., Wang, Z. C., Ma, X. Y., Liu, W. N., Chen, H. S., & Nie, Y. P. (2022). Isotopic deviations of water extracted from carbonate soil by cryogenic vacuum extraction: Implication for root water uptake analysis. *Plant and Soil*, *475*, 79–89. https://doi.org/10.1007/s11104-022-05455-9

Koeniger, P., Marshall, J. D., Link, T., & Mulch, A. (2011). An inexpensive, fast, and reliable method for vacuum extraction of soil and plant water for stable isotope analyses by mass spectrometry. *Rapid Communications in Mass Spectrometry*, *25*(20), 3041–3048. https://doi.org/10.1002/rcm.5198

Landwehr, J. M., & Coplen, T. B. (2006). Line-conditioned excess: A new method for characterizing stable hydrogen and oxygen isotope ratios in hydrologic systems. In *Isotopes in environmental studies isotopes in environmental studies* (pp. 132–135). International Atomic Energy Agency.

Lee, X. H., Sargent, S., Smith, R., & Tanner, B. (2005). In situ measurement of the water vapor ^{18}O/^{16}O isotope ratio for atmospheric and ecological applications. *Journal of Atmospheric and Oceanic Technology*, *22*(5), 555–565. https://doi.org/10.1175/JTECH1719.1

Li, S. G., Romero-Saltos, H., Tsujimura, M., Sugimoto, A., Sasaki, L., Davaa, G., & Oyunbaatar, D. (2007). Plant water sources in the cold semiarid ecosystem of the upper Kherlen River catchment in Mongolia: A stable isotope approach. *Journal of Hydrology*, *333*(1), 109–117. https://doi.org/10.1016/j.jhydrol.2006.07.020

Li, Y., Ma, Y., Song, X. F., Wang, L. X., & Han, D. M. (2021). A δ^{2}H offset correction method for quantifying root water uptake of riparian trees. *Journal of Hydrology*, *593*, 125811. https://doi.org/10.1016/j.jhydrol.2020.125811

Liang, J., Wright, J. S., Cui, X. W., Sternberg, L., Gan, W. X., & Lin, G. H. (2018). Leaf anatomical traits determine the ^{18}O enrichment of leaf water in coastal halophytes. *Plant, Cell & Environment*, *41*(12), 2744–2757. https://doi.org/10.1111/pce.13398

Lin, G. H., Sternberg, L., Ehleringer, J. R., Hall, A. E., & Farquhar, G. D. (1993). Hydrogen isotopic fractionation by plant roots during water uptake in coastal wetland plant. In J. R. Ehleringer, A. E. Hall, & G. D. Farquhar (Eds.), *Stable isotopes and plant carbon-water relations* (pp. 497–510). Academic Press Inc. https://doi.org/10.1016/B978-0-08-091801-3.50041-6

Meißner, M., Köhler, M., Schwendenmann, L., Hölscher, D., & Dyckmanns, J. (2014). Soil water uptake by trees using water stable isotopes (δ^{2}H and δ^{18}O)—A method test regarding soil moisture, texture and carbonate. *Plant and Soil*, *376*, 327–335. https://doi.org/10.1007/s11104-013-1970-z

Millar, C., Pratt, D., Schneider, D. J., & McDonnell, J. J. (2018). A comparison of extraction systems for plant water stable isotope analysis. *Rapid Communications in Mass Spectrometry*, *32*(13), 1031–1044. https://doi.org/10.1002/rcm.8136

Moreno-Gutiérrez, C., Dawson, T., Nicolás, E., & Querejeta, J. I. (2012). Isotopes reveal contrasting water use strategies among coexisting plant species in a Mediterranean ecosystem. *New Phytologist*, *196*(2), 489–496. https://doi.org/10.1111/j.1469-8137.2012.04276.x

Munksgaard, N. C., Cheesman, A. W., Wurster, C. M., Cernusak, L. A., & Bird, M. I. (2014). Microwave extraction-isotope ratio infrared spectroscopy (ME-IRIS): A novel technique for rapid extraction and

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

in-line analysis of $\delta^{18}O$ and $\delta^{2}H$ values of water in plants, soils and insects. *Rapid Communications in Mass Spectrometry*, *28*(20), 2151–2161. https://doi.org/10.1002/rcm.7005

Newberry, S. L., Nelson, D. B., & Kahmen, A. (2017). Cryogenic vacuum artifacts do not affect plant water-uptake studies using stable isotope analysis. *Ecohydrology*, *10*, e1892. https://doi.org/10.1002/eco.1892

Newberry, S. L., Prechsl, U. E., Pace, M., & Kahmen, A. (2017). Tightly bound soil water introduces isotopic memory effects on mobile and extractable soil water pools. *Isotopes in Environmental and Health Studies*, *53*(4), 368–381. https://doi.org/10.1080/10256016.2017.1302446

Nie, Y. P., Chen, H. S., Ding, Y. L., Zou, Q. Y., & Ma, X. Y. (2019). Qualitative identification of hydrologically different water sources used by plants in rock-dominated environments. *Journal of Hydrology*, *573*, 386–394. https://doi.org/10.1016/j.jhydrol.2019.03.097

Oerter, E., Finstad, K., Schaefer, J., Goldsmith, G. R., Dawson, T. E., & Amundson, R. (2014). Oxygen isotope fractionation effects in soil water via interaction with cations (Mg, Ca, K, Na) adsorbed to phyllosilicate clay minerals. *Journal of Hydrology*, *515*, 1–9. https://doi.org/10.1016/j.jhydrol.2014.04.029

Orlowski, N., Breuer, L., Angeli, N., Boeckx, P., Brumbt, C., Cook, C. S., Dubbert, M., Dyckmans, J., Gallagher, B., Gralher, B., Herbstritt, B., Hervé-Fernández, P., Hissler, C., Koeniger, P., Legout, A., Macdonald, C. J., Oyarzún, C., Redelstein, R., Seidler, C., … McDonnell, J. J. (2018). Inter-laboratory comparison of cryogenic water extraction systems for stable isotope analysis of soil water. *Hydrology and Earth System Sciences*, *22*(7), 3619–3637. https://doi.org/10.5194/hess-22-3619-2018

Orlowski, N., Breuer, L., & McDonnell, J. J. (2016). Critical issues with cryogenic extraction of soil water for stable isotope analysis. *Ecohydrology*, *9*, 1–5. https://doi.org/10.1002/eco.1722

Orlowski, N., Frede, H.-G., Brüggemann, N., & Breuer, L. (2013). Validation and application of a cryogenic vacuum extraction system for soil and plant water extraction for isotope analysis. *Journal of Sensors and Sensor Systems*, *2*(2), 179–193. https://doi.org/10.5194/jsss-2-179-2013

Orlowski, N., Pratt, D. L., & McDonnell, J. J. (2016). Inter-comparison of soil pore water extraction methods for stable isotope analysis. *Hydrological Processes*, *30*(19), 3434–3449. https://doi.org/10.1002/hyp.10870

Orlowski, N., Winkler, A., McDonnell, J. J., & Breuer, L. (2018). A simple greenhouse experiment to explore the effect of cryogenic water extraction for tracing plant source water. *Ecohydrology*, *11*, e1967. https://doi.org/10.1002/eco.1967

Palacio, S., Azorín, J., Montserrat-Martí, G., & Ferrio, J. P. (2014). The crystallization water of gypsum rocks is a relevant water source for plants. *Nature Communications*, *5*, 4660. https://doi.org/10.1038/ncomms5660

Peters, L. I., & Yakir, D. (2008). A direct and rapid leaf water extraction method for isotopic analysis. *Rapid Communications in Mass Spectrometry*, *22*(18), 2929–2936. https://doi.org/10.1002/rcm.3692

Pinheiro, J., Bates, D., DebRoy, S., Sarkar, D., & R Core Team. (2021). *nlme: Linear and nonlinear mixed effects models*. R Package Version 3, 1–153. https://CRAN.R-project.org/package=nlme

Qiu, X., Zhang, M. J., Wang, S. J., Evaristo, J., Argiriou, A. A., Guo, R., Chen, R., Meng, H. F., Che, C. W., & Qu, D. Y. (2019). The test of the ecohydrological separation hypothesis in a dry zone of the northeastern Tibetan plateau. *Ecohydrology*, *12*, e2077. https://doi.org/10.1002/eco.2077

R Core Team. (2021). *R: A language and environment for statistical computing*. R Foundation for Statistical Computing. https://www.R-project.org/

Revesz, K., & Woods, P. H. (1990). A method to extract soil water for stable isotope analysis. *Journal of Hydrology*, *115*(1–4), 379–406. https://doi.org/10.1016/0022-1694(90)90217-L

Rodríguez-Robles, U., Arredondo, J. T., Huber-Sannwald, E., Yépez, E. A., & Ramos-Leal, J. A. (2020). Coupled plant traits adapted to wetting/drying cycles of substrates co-define niche multidimensionality. *Plant, Cell & Environment*, *43*(10), 2394–2408. https://doi.org/10.1111/pce.13837

Schultz, N. M., Griffis, T. J., Lee, X. H., & Baker, J. M. (2011). Identification and correction of spectral contamination in $^{2}H/^{1}H$ and $^{18}O/^{16}O$ measured in leaf, stem, and soil water. *Rapid Communications in Mass Spectrometry*, *25*(21), 3360–3368. https://doi.org/10.1002/rcm.5236

Schwendenmann, L., Veldkamp, E., Moser, G., Hölscher, D., Köhler, M., Clough, Y., Anas, I., Djajakirana, G., Erasmi, S., & Hertel, D. (2010). Effects of an experimental drought on the functioning of a cacao agroforestry system, Sulawesi, Indonesia. *Global Change Biology*, *16*(5), 1515–1530. https://doi.org/10.1111/j.1365-2486.2009.02034.x

Song, L., Yang, B., Liu, L. L., Mo, Y. X., Liu, W. J., Meng, X. J., Lu, H. Z., Li, Y., Zakari, S., Tan, Z. H., Fan, Z. X., & Zhang, Y. J. (2022). Spatial-temporal differentiations in water use of coexisting trees from a subtropical evergreen broadleaved forest in Southwest China. *Agricultural and Forest Meteorology*, *316*, 108862. https://doi.org/10.1016/j.agrformet.2022.108862

Stock, B. C., & Semmens, B. X. (2013). *MixSIAR GUI user*. https://doi.org/10.5281/zenodo.6159

Stoffel, M. A., Nakagawa, S., & Schielzeth, H. (2017). rptR: Repeatability estimation and variance decomposition by generalized linear mixed-effects models. *Methods in Ecology and Evolution*, *8*(11), 1639–1644. https://doi.org/10.1111/2041-210X.12797

Tang, Y. K., Wu, X., Chen, C., Jia, C., & Chen, Y. M. (2019). Water source partitioning and nitrogen facilitation promote coexistence of nitrogen-fixing and neighbor species in mixed plantations in the semiarid loess plateau. *Plant and Soil*, *445*, 289–305. https://doi.org/10.1007/s11104-019-04301-9

Thielemann, H., Gerjets, R., & Dyckmans, J. (2019). Effects of soil-bound water exchange on the recovery of spike water by cryogenic water extraction. *Rapid Communications in Mass Spectrometry*, *33*(5), 405–410. https://doi.org/10.1002/rcm.8348

Tsuruta, K., Yamamoto, H., Katsuyama, M., Kosugi, Y., Okumura, M., & Matsuo, N. (2019). Effects of cryogenic vacuum distillation on the stable isotope ratios of soil water. *Hydrological Research Letters*, *13*(1), 1–6. https://doi.org/10.3178/hrl.13.1

Turner, J. V., & Gailitis, V. (1988). Single-step method for hydrogen isotope ratio measurement of water in porous media. *Analytical Chemistry*, *60*(11), 1244–1246. https://doi.org/10.1021/ac00162a030

von Freyberg, J., Allen, S. T., Grossiord, C., & Dawson, T. E. (2020). Plant and root-zone water isotopes are difficult to measure, explain, and-predict: Some practical recommendations for determining plant watersources. *Methods in Ecology and Evolution*, *11*(11), 1352–1367. https://doi.org/10.1111/2041-210X.13461

Walker, G. R., Woods, P. H., & Allison, G. B. (1994). Interlaboratory comparison of methods to determine the stable isotope composition of soil water. *Chemical Geology*, *111*(1–4), 297–306. https://doi.org/10.1016/0009-2541(94)90096-5

Wassenaar, L. I., Ahmad, M., Aggarwal, P., van Duren, M., Pöltenstein, L., Araguas, L., & Kurttas, T. (2012). Worldwide proficiency test for routine analysis of $\delta^{2}H$ and $\delta^{18}O$ in water by isotope-ratio mass spectrometry and laser absorptionspectroscopy. *Rapid Communications in Mass Spectrometry*, *26*(15), 1641–1648. https://doi.org/10.1002/rcm.6270

Wassenaar, L. I., Hendry, M. J., Chostner, V. L., & Lis, G. P. (2008). High resolution pore water $\delta^{2}H$ and $\delta^{18}O$ measurements by H_2O(liquid)-H_2O(vapor) equilibration laser spectroscopy. *Environmental Scince Technology*, *42*(24), 9262–9267. https://doi.org/10.1021/es802065s

Wen, X. F., Yang, B., Sun, X. M., & Li, X. H. (2016). Evapotranspiration partitioning through in-situ oxygen isotopemeasurements in an oasis cropland. *Agricultural and Forest Meteorology*, *230–231*, 89–96. https://doi.org/10.1016/j.agrformet.2015.12.003

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

West, A. G., Patrickson, S. J., & Ehleringer, J. R. (2006). Water extraction times for plant and soil materials used in stable isotope analysis. *Rapid Communications in Mass Spectrometry*, *20*(8), 1317–1321. https://doi.org/10.1002/rcm.2456

White, J., Cook, E., Lawrence, J., & Broecker, W. (1985). The D/H ratios of sap in trees-implications for water sources and tree-ring D/H ratios. *Geochimica et Cosmochimica Acta*, *49*(1), 237–246. https://doi.org/10.1016/0016-7037(85)90207-8

Xiao, W., Lee, X. H., Wen, X. F., Sun, X. M., & Zhang, S. C. (2012). Modeling biophysical controls on canopy foliage water ^{18}O enrichment in wheat and corn. *Global Change Biology*, *18*(5), 1769–1780. https://doi.org/10.1111/j.1365-2486.2012.02648.x

Yang, B., Dossa, G. G. O., Hu, Y. H., Liu, L. L., Meng, X. J., Du, Y. Y., Li, J. Y., Zhu, X. A., Zhang, Y. J., Singh, A. K., Yuan, X., Wu, J. E., Zakari, S., Liu, W. J., & Song, L. (2023). Data from: Uncorrected soil water isotopes through cryogenic vacuum distillation may lead to a false estimation on plant water sources. *Dryad Digital Repository*, https://doi.org/10.5061/dryad.fxpnvx0w1

Yang, B., Meng, X. J., Singh, A. K., Wang, P. Y., Song, L., Zakari, S., & Liu, W. J. (2020). Intercrops improve surface water availability in rubber-based agroforestry systems. *Agriculture, Ecosystems and Environment*, *298*, 106937. https://doi.org/10.1016/j.agee.2020.106937

Yang, B., Wen, X. F., & Sun, X. M. (2015). Seasonal variations in depth of water uptake for a subtropical coniferous plantation subjected to drought in an east Asian monsoon region. *Agricultural and Forest Meteorology*, *201*, 218–228. https://doi.org/10.1016/j.agrformet.2014.11.020

Zhu, Q. Z., Sun, Q., Su, Z. G., Xie, M. M., Song, J. Y., Shan, Y. B., Wang, N., & Chu, G. Q. (2014). A soil water extraction method with accelerated solvent extraction technique for stable isotope analysis. *Chinese Journal of Analytical Chemistry*, *42*(9), 1270–1275. https://doi.org/10.1016/S1872-2040(14)60766-0

SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

Appendix A: Figures S1–S3, Table S1.

Appendix B: Supplementary Information Text.

How to cite this article: Yang, B., Dossa, G. G. O., Hu, Y.-H., Liu, L.-L., Meng, X.-J., Du, Y.-Y., Li, J.-Y., Zhu, X.-A., Zhang, Y.-J., Singh, A. K., Yuan, X., Wu, J.-E., Zakari, S., Liu, W.-J., & Song, L. (2023). Uncorrected soil water isotopes through cryogenic vacuum distillation may lead to a false estimation on plant water sources. *Methods in Ecology and Evolution*, *14*, 1443–1456. https://doi.org/10.1111/2041-210X.14107

2041210x, 2023, 6, Downloaded from https://besjournals.onlinelibrary.wiley.com/doi/10.1111/2041-210X.14107 by Xishuangbanna Tropical Botanical Garden, Wiley Online Library on [03/08/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License