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Technical Note

A guideline for sample preparation in modern tree-ring stable isotope research

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ABSTRACT

The comprehensive procedure of wood sample preparation, including tree-ring dissection, cellulose extraction, homogenization and packing for stable isotope analysis, is labour intensive and time consuming.

Based on a brief compilation of existing methods, we present a methodological approach from pre-analyses considerations to wood sample preparation, semi-automated chemical extraction of cellulose from tree-ring cross-sections, and tree-ring dissection for stable isotope ratio mass spectrometry: the Cross-Section Extraction and Dissection (CSED) guideline. Following the CSED guideline can considerably increase efficiency of tree-ring stable isotope measurement compared to classical methods < ABS-P > We introduce a user-friendly device for cellulose extraction, allowing simultaneous treatment of wood cross-sections of a total length of 180 cm (equivalent to 6 increment cores of 30 cm length) and thickness of 0.6–2.0 mm. After cellulose extraction, tree-ring structures of 10 tree species (coniferous and angiosperm wood) with different wood growth rates and tree-ring boundaries, largely remained well identifiable.

Further, we demonstrate that tree rings from cellulose cross-sections can be dissected at annual to intra-seasonal resolution, utilizing simple manual devices as well as sophisticated UV-laser microdissection microscopes in a way that sample homogenization is no longer necessary in most cases.

We investigate seasonal precipitation signals in high-resolution intra-annual $\delta^{18}O$ cellulose values from African baobab, performed by using UV-laser microdissection microscopes.

1. Introduction

Tree-ring stable isotope records are powerful proxies in palaeoclimatic (e.g. Brienen et al., 2012; Heinrich et al., 2013; Konter et al., 2014; Loader et al., 2010; Treydte et al., 2006) and plant physiological studies (e.g. Gessler et al., 2013; Helle and Schleser, 2004; Simard et al., 2012). Different wood components (wood, lignin or cellulose) can be used in the analysis of carbon, oxygen or hydrogen stable isotopes. Cellulose, a primary carbohydrate, is often the preferred sample material because of its short synthesis pathway, singular chemical composition and physical immobility within the stem (McCarroll and Loader, 2004).

Recent advancements in the methodology of tree-ring stable isotope analysis have permitted the efficient measurement of a large number of samples using minimal sample amounts (few micrograms) (Gori et al., 2013; Kornexl et al., 1999; Koziet, 1997; Loader et al., 2014; Saurer et al., 1998). However, such analytical developments complicate sample preparation, as they require very high sample purity and homogeneity. Furthermore, tree-ring dissection and chemical preparation (cellulose extraction from wood tissue) are time consuming, prompting research into improving the efficiency of these procedures for stable isotope analysis (Anchukaitis et al., 2008; Brendel et al., 2000; Cullen and MacFarlane, 2005; Gaudinski et al., 2005; Green, 1963; Harada et al., 2014; Hook et al., 2015; Laumer et al., 2009; Leavitt, 2010; Leavitt and Danzer, 1993; Loader et al., 1997; Rinne et al., 2005; Sheu and Chiu, 1995; Wieloch et al., 2011). As indicated in the overview of key procedures of tree-ring stable isotope analysis (Fig. 1), all of these approaches start with sampling tree rings by using

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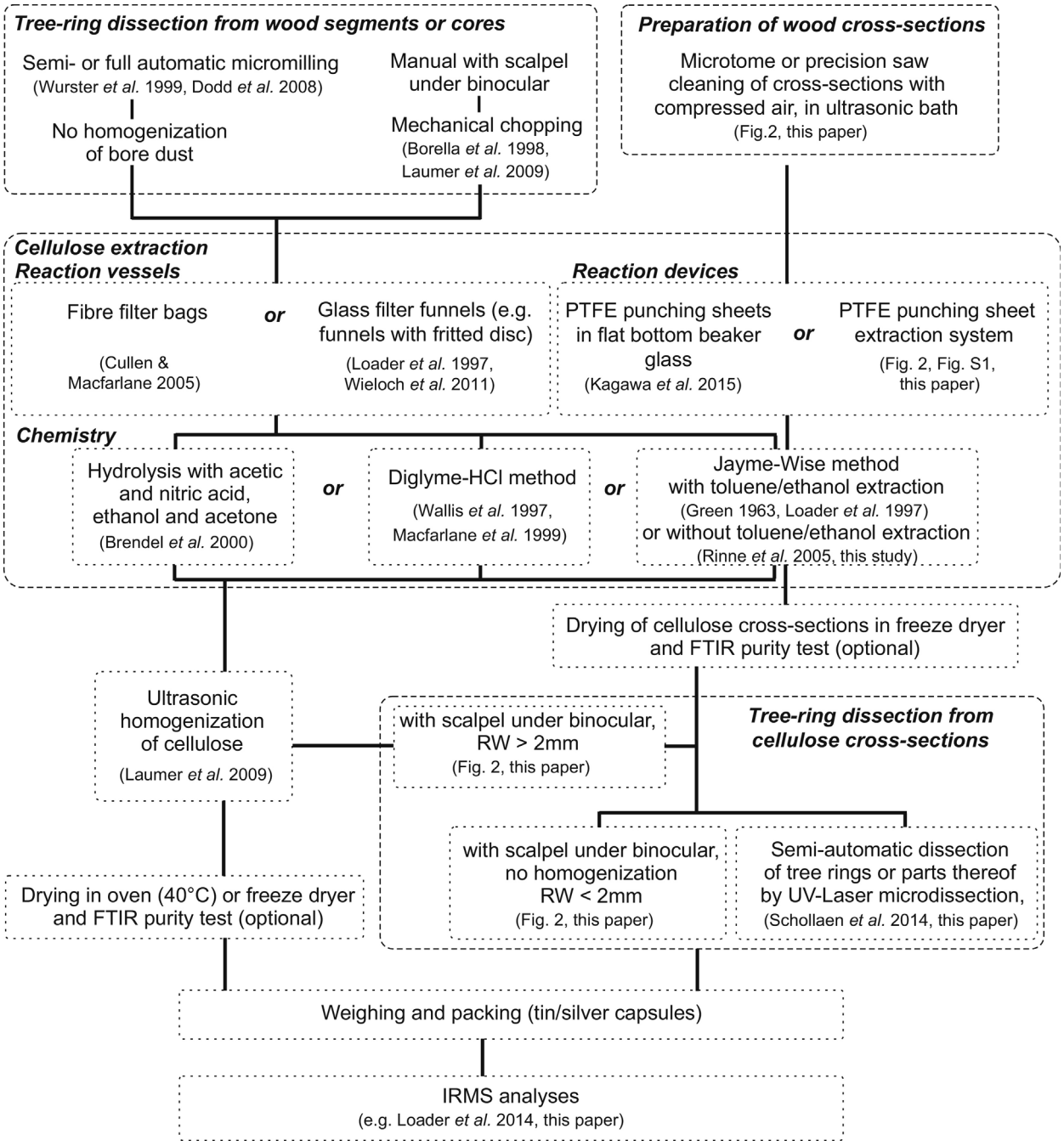

Fig. 1. Overview of procedures of tree-ring stable isotope analysis involving cellulose extraction. For technical details cf. text, table S2, as well as key references given in that figure and citations therein.

manual subdivision by scalpel, microtome or micromilling devices (e.g. Dodd et al., 2008; Wurster et al., 1999) prior to cellulose extraction. Despite novel technique developments, including the simultaneous chemical treatment of several hundred micro-samples (Wieloch et al., 2011) and individual homogenization (grinding or ultra-sonic treatment) (Laumer et al., 2009), chemical treatment of individual tree-ring samples was still required.

In order to reduce the cutting-grinding-chemical processing for each individual tree ring, Loader et al. (2002) made a first attempt to extract cellulose directly from standard increment cores (5 mm Ø). More recently, Li et al. (2011) reported a technique to extract α-cellulose directly from wood cross-sections using perforated U-channel PTFE casing to prevent cellulose spline from breaking apart. They conducted high-resolution intra-annual $\delta^{13}C$ and $\delta^{18}O$ analyses on a 3 mm wide rings from a 3.5–4.0 mm thick α-cellulose cross-section, revealing no discrepancies from the usual method. A breakthrough in high sample throughput was achieved by Nakatsuka et al. (2011) with methodological improvements by Kagawa et al. (2015), where a container made of teflon (PTFE, polytetrafluoroethylene) punching sheet was designed to prevent disintegration of cellulose laths (see also Xu et al., 2011). The basic principle allows cellulose extraction from wood laths in a single batch, enabling the same chemical conditions for all samples, while significantly reducing time needed for cellulose preparation and retaining the wood cell structure.

Building upon on the idea of extracting cellulose from wood laths, we designed an improved semi-automated cellulose extraction system. The performance of this high-throughput cellulose extraction device was evaluated to provide precisely dissected, homogenous tree-ring