Quaternary Geochronology 20 (2014) 89–98

Contents lists available at ScienceDirect

Quaternary Geochronology

journal homepage: www.elsevier.com/locate/quageo

Research paper

Evaluation of the rehydroxylation dating method: Insights from a new measurement device

Maxime Le Goff*, Yves Gallet

Equipe de Paléomagnétisme, Institut de Physique du Globe de Paris, Sorbonne Paris Cité, Univ. Paris Diderot, UMR 7154 CNRS, F-75005 Paris, France

ARTICLE INFO

Article history:
Received 2 May 2013
Received in revised form
2 October 2013
Accepted 3 December 2013
Available online 11 December 2013

Keywords:
Rehydroxylation dating
Ceramics
Heating duration
Archeology
Archeomagnetism

ABSTRACT

The recently published rehydroxylation (RHX) dating method applicable to baked clay artifacts potentially represents a major tool for research in geoarcheology and archeomagnetism. We report on a new experimental device customized to conduct RHX experiments on series of ten samples without any operator manipulation. We applied the rehydroxylation method on precisely dated French archeological fragments. Our device provides adequate environmental experimental conditions, yet our observations identified several difficulties. First, based on the published protocol, the "archeological mass" of a sample should be determined following an initial drying at 105 °C when the slope of the mass over time is zero. In all our experiments on ~60 samples, no stabilization of the sample mass is reached even after several weeks of monitoring. This is always true whether the heating at 105 °C was short (a few hours) or long (several days), which may indicate that a slow diffusion process is ongoing even after a low-temperature heating. Second, the initial sample mass following the heating step at 105 °C or 500 °C remained dependent on the duration of heating even though both short and long heating were applied. We demonstrate that the duration of heating at both 105 °C and 500 °C is a critical parameter for the RHX dating method. Further methodological improvements, including the selection of suitable fired clay fragments, are thus required so that the RHX dating method becomes reliable and efficient.

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1. Introduction

Much effort is currently devoted to the construction of reliable regional and global geomagnetic field models ranging over the past few millennia (e.g. Pavón-Carrasco et al., 2009; Korte et al., 2011; Licht et al., 2013). These reconstructions rely on large compilations of directional and intensity magnetic data obtained from archeological, volcanic and sedimentary materials, for which the parameters of dating are clearly of major importance (e.g. Genevey et al., 2008; Donadini et al., 2009). In particular, the (archeomagnetic) data obtained from archeological baked clays, i.e. from kiln walls, pottery, bricks or tiles are recognized as very suitable for geomagnetic modeling because they often benefit from dating constraints provided by their archeological contexts of discovery, further including the typology of the studied objects, historical accounts and/or by radiocarbon (^{14}C) counting.

The dating accuracy, together with the temporal homogeneity of the samples when several fragments collected from different objects (for instance ceramics) are used to define a single dated archeomagnetic site are very restrictive criteria but essential to ensure the reliability of archeomagnetic data. All themes of research in archeomagnetism, such as the detection and characterization of rapid multidecadal-scale geomagnetic fluctuations (e.g. the archeomagnetic jerks; Gallet et al., 2003, 2009a), the potential link of the latter with atmospheric processes or their use as a dating tool for archeological purposes (e.g. Le Goff et al., 2002; Lanos, 2004; Gallet et al., 2009b) depend on the dating uncertainties attached to the analyzed objects. For this reason, the archeological dating method based on rehydroxylation processes in baked clays recently proposed by Wilson et al. (2009, 2012) offers an exceptional range of applications in archeomagnetism, in particular because the very same objects could both provide archeomagnetic and dating results, and more generally for all fields of research in (geo)archeology. One of the most important aspect of this new dating method, referred below to as the RHX (ReHydroXylation) dating method, is that it would also give the possibility to perform straightforward relative dating between fragments collected from the same archeological sites.

The RHX dating method as described by Wilson et al. (2009), however, is still challenging due to the lack of sufficient testing. There are no other successful dating results than those, not numerous, already proposed by M. Wilson and colleagues (10 data

* Corresponding author.
E-mail address: legoff@ipgp.fr (M. Le Goff).

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http://dx.doi.org/10.1016/j.quageo.2013.12.001

reported in 2009 and 5 others in 2012), while Bowen et al. (2011) were unable to obtain satisfactory results from 4 samples and Burakov and Nachasova (2013) underlined complexities from 6 dating attempts. For this reason, it appears essential to explore further the method, its validity considering samples of different ages and origins and its applicability to the domain of archeomagnetism. If successful, the rehydroxylation dating method would allow us to strengthen our collaboration with archeologists and would significantly contribute to the improvement of our knowledge of the geomagnetic field behavior over the past millennia.

The paper is organized as follows. After the present introduction, Section 2 describes the RHX dating method as proposed by Wilson et al. (2009). Section 3 describes a new experimental system we built in order to perform mass gain measurements for series of ten samples at the time in a temperature and relative humidity controlled environment. We present in Section 4 the results of our investigations using samples of different origins and different ages. From these experiments, we address in Section 5 several encountered difficulties, such as the systematic lack of stabilization of the mass of the samples after their heating at 105 °C, even after several weeks of monitoring, or the fact that the initial sample mass following the heating step at 105 °C and 500 °C remained dependent on the heating duration even though both short (a few hours) and long (up to a week) heating were applied. These difficulties, which prevent us to obtain satisfactory RHX dating estimates, are finally summarized in Section 6.

2. Description of the rehydroxylation dating method

Such as described by Wilson et al. (2009), the overall principles of the rehydroxylation dating method seem to be relatively simple, self calibrated and apparently easy to implement. We present below a rough description of the method but a more thorough discussion on the underlying processes can be found in Wilson et al. (2009, 2012) (see also Savage et al., 2008; Hall et al., 2011; Hamilton and Hall, 2012). When the clays used for making pottery, bricks, etc. are heated to high temperatures (>500 °C), they undergo a complete dehydroxylation, which results in the loss of all water components (e.g. Wilson et al., 2012). This stage defines what can be referred to as the archeological “zero age” to be determined. When the baked clays are subsequently cooled at ambiant temperature, they rapidly undergo (in a few hours) an adsorption phase which principally corresponds to the gain of capillary water (defining the brief “stage I” in RHX experiments), followed by a very slow rehydroxylation process, i.e. the diffusive transport of OH groups from the environment into the material (defining “stage II”), with both stages leading to moisture expansion and to a gain of mass of the material (e.g. Hamilton and Hall, 2012). The fundamental observation of Wilson et al. (2003, 2009) (see also Savage et al., 2008) lies in the fact that the increase in mass of a baked clay sample is a function of time following a power law with exponent 1/4 over centennial and millennial time scales (red dashed lines, Fig. 1). According to Wilson et al. (2009), such a behavior may reflect a diffusion process in a restricted environment (see also discussion in Hamilton and Hall, 2012). Using the notation given in Fig. 1, the law is:

$$m = m_{RX}\left(1 + \alpha t^{1/4}\right) \quad (1)$$

or, in terms of fractional mass gain relative to m_{Rx} (fmg_{RH}):

$$fmg_{RX} = (m - m_{RX})/m_{RX} = \alpha t^{1/4} \quad (2)$$

The RHX dating method derived from the observations above hence comprises the following steps (Wilson et al., 2009, 2012):

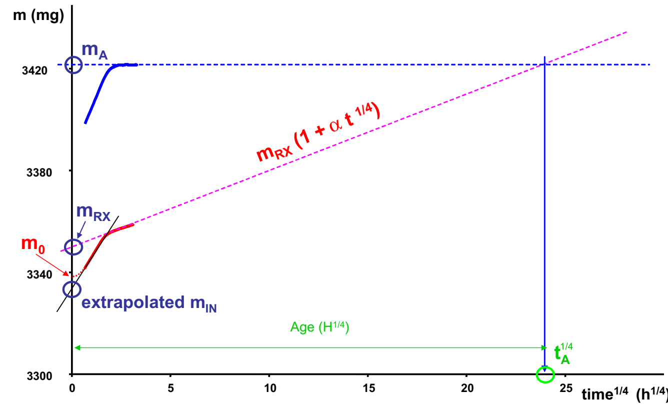

Fig. 1. Principles of the rehydroxylation dating method. The blue (resp. red) curve represents the mass data obtained for one sample after its heating to 105 °C (resp. 500 °C) and while it remains in constant environmental conditions (temperature and relative humidity). The data are plotted against time$^{1/4}$. After the first heating at 105 °C, the mass data become constant after ~2.5 hours$^{1/4}$, so defining the sample archeological mass (m_A). After the second heating at 500 °C, the mass values show a linear trend after ~2 hours$^{1/4}$, i.e. after the so-called stage I (see text), in accordance with a slow rehydroxylation process (stage II, see text); the extrapolation of this behavior to m_A gives its archeological age (t_A, in hours$^{1/4}$). The two values m_{IN} and m_{Rx} are the initial sample mass respectively derived from stage I and stage II-mass variations extrapolated to time$^{1/4}$ = 0; m_0 is the “true” initial mass value after heating of the sample to 500 °C (Wilson et al., 2009). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

a) Heating the sample of a few grams at 105 °C to constant mass in order to remove the capillary water from the pores and weakly bound adsorbed water (Wilson et al., 2012).
b) Weighing the sample in imposed and constant conditions of temperature and relative humidity (RH) until reaching a constant mass, hereafter referred to as the archeological mass (m_A).
c) Heating the sample at high temperature (500 °C; Wilson et al., 2012) during several hours, until reaching a constant mass, in order to perform a complete dehydroxylation of the sample.
d) Repeatedly weighing the sample during several weeks in the same constant environmental conditions as those imposed for step “b” until the mass gain rate becomes constant and can be precisely determined (Wilson et al., 2013).
e) Defining from RHX mass data of step “d” fitted to a time ¼ power law the parameters α and m_{Rx}, which allows one through extrapolation to m_A to determine the time spent since the original (archeological) heating of the sample. This defines t_A with:

$$t_A = ((m_A - m_{RX})/\alpha m_{RX})^4 \quad (3)$$

It is worth noting that Bowen et al. (2011) used a single power law function to fit all the mass data encompassing both stages I and II. Until now, however, no satisfactory RHX dating results could be derived using this alternative approach (see below).

In the method proposed by Wilson et al. (2009), the stringent parameter is that the kinetic of rehydroxylation after high-temperature heating (α) strongly depends on the temperature experienced by the sample (but not on humidity). To obtain a dating, the temperature used for the experiments must be chosen identical or very close to that experienced by the sample during its archeological history (referred to as the mean lifetime temperature by Wilson et al., 2009). Beyond the experimental constraints, which this imposes (no disturbance in temperature and humidity in a climatic chamber during several days up to several weeks), it is thus necessary to precisely know the average climate (average

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