Testing rehydroxylation dating of ceramics

Elisabeth Schnepp, Roman Leonhardt

Well dated archaeological ceramics provide a rich source of geomagnetic intensities, which can be measured with palaeomagnetic methods. If historical documents are missing, precise age dating of such material is still challenging. Rehydroxylation (RHX) dating is a method proposed to obtain the age of ceramics, which relies on the nature of clay minerals to accumulate crystal water caused by the slow progressive chemical recombination of the fired-clay with environmental water. The Conrad Observatory provides stable environmental conditions in the tunnel for testing this method. Here we present the first tests from a collection of bricks and shards.

A very attractive dating method that could be applied to ceramics was proposed by Wilson et al. (2009 and 2014, Proc. R. Soc.). During production of ceramics, clay looses weakly bound molecular water at low temperatures. Above ~450°C water is removed from the octahedral sheets by chemical dehydroxylation or other minerals are formed, but the reactions are not complete. The second process can be used for age dating. Measurements comprise two steps: 1. After heating to 105°C for several hours the increasing weight of the sample is measured until the content of molecular water is re-established. 2. After heating to 550°C the RHX process is observed and the obtained RXH rate constant is determined. Dating is then obtained from the mass lost between 550 and 105°C and the RHX rate, which is claimed to be proportional to the fourth root of time ($t^{0.25}$).

The main aims of our experiment are to test, (1) if conditions in the tunnel are suitable for such dating experiments and (2) whether the suggested general trends can be confirmed. The measurement setup is very simple: A balance (Kern 410-11, resolution 0.1 mg) is installed within a cabinet in the tunnel, which provides constant temperature and humidity conditions (8.8°C, 86%). This balance is sensitive enough to resolve the RHX effect, although for precise dating measurements 1 µg or better would be required. 16 specimens comprising bricks or brick-like kiln fragments and one potshard with ages ranging from medieval to 2003 are tested; 3 have ages known from historical documents. The mass of the specimens lies between 2.5 and 12 g. Specimens were heated 40.5 h in Gams laboratory and again at Trafelberg. After cooling the weighting experiment has been started in the tunnel. Step 1 of the experiment is not yet finished, although the experiment is in progress for almost one year. Preliminary results are shown in Fig. 1. The results are much more scattered as those found in references (e.g. LeGoff & Gallet 2014, Quat. Geochr.).

The observed curves show 4 different types. Type Ia found in only 4 specimens is similar to the results of Wilson et al. (2009). After a rapid increase a stabilization of mass is observed. For Type Ib (9 specimens) the mass is still slowly increasing. This absent state of saturation was also observed by e.g. Gallet & LeGoff (2015, J. Am. Ceram. Soc.). Saturation is reached only in 4 cases within a few hours. For all other samples of groups Ia and Ib several hundred hours or even 1400 h are observed.
Type II shows a progressive increase which is almost linear with $t^{0.25}$. This law is observed for the RHX process, which seems to be very strong in these specimens (2). Finally, there is one example (Type III) showing a decrease in mass followed by stabilisation.

These preliminary results strongly support other findings (e.g. LeGoff & Gallet) that RHX dating is not that simple as proposed by Wilson.

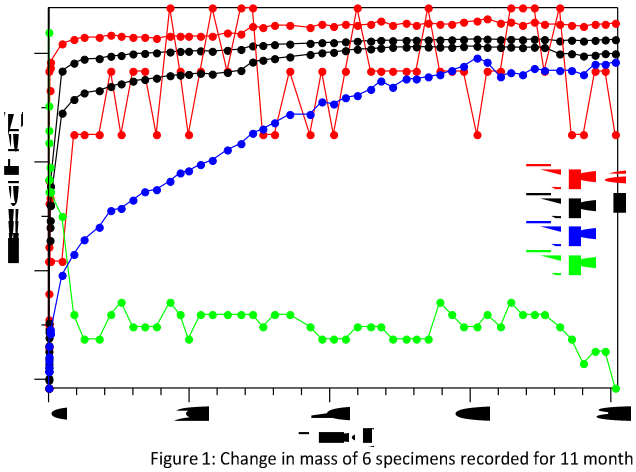

Figure 1: Change in mass of 6 specimens recorded for 11 month

Author:
E. Schnepp[1] R. Leonhardt[2]
1) Montanuniversität Leoben, Leoben, Austria
2) Central Institute for Meteorology and Geodynamics, Vienna, Austria

Corresponding author:
Dr. Elisabeth Schnepp
Montanuniversität Leoben, Lehrstuhl für Geophysik, Paläomagnetiklabor Gams, Gams 45, A-8130 Frohnleiten, Austria.
Tel.: +43 (0) 3842 402 2643
e-mail: elisabeth.schnepp@unileoben.ac.at