

RECENT ADVANCES IN OBSIDIAN HYDRATION DATING

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Major advances have been made in the past decade in understanding and applying the basic science of obsidian hydration dating (OHD). Drawing on advances in geochemistry and glass science, mathematical models for OHD have been developed and published which are based on the physics and chemistry of obsidian hydration. If these improved methods are used, OHD is capable of reliably placing obsidian artifacts within the correct archaeological period and of answering interesting anthropological questions. In this article, we address four significant aspects of these advances: the form of the age equation; the effects of temperature on hydration; the effect of obsidian structural water on the hydration rate; and the ability to compute age accuracy in addition to age itself. Finally, we briefly summarize the limitations to the current method and the outlook for future research.

Obsidian hydration dating (OHD) is a method of computing an archaeological age based on measuring the depth of diffused water in the near surface region of obsidian artifacts. Although currently less accurate than radiocarbon dating, it is also less expensive and hence larger data sets are feasible. Unlike dating by projectile point typology, OHD can be used on debitage as well, and it is the only chronometric method that can directly date obsidian artifacts. It is often the only option for chronometric assessments of sparse desert sites, where radiocarbon or dendrochronology specimens are typically lacking. Its ability to determine ages for non-diagnostic artifacts makes it useful in studies of trade and exchange by correlating obsidian compositional data from a number of sites. Unlike radiocarbon or dendrochronology, obsidian hydration is primarily controlled by post-depositional temperature history, so great care is needed in controlling for environmental effects.

Here we provide background on OHD and briefly review four recent advances in the field: the form of the age equation; the effects of temperature and methods of controlling for temperature; the effects of obsidian structural (intrinsic) water content on the hydration process; and a recently developed method for computing the accuracy of an OHD age. Finally, we give a summary of the state of the art and a description of current research topics.

HISTORY AND CURRENT METHOD OF OBSIDIAN HYDRATION DATING

Obsidian hydration dating as a discipline dates from the original article by Friedman and Smith (1960). They correctly identified the physical process involved, the mathematical form of the hydration law, and other fundamental properties of hydration. Subsequently, other advances were made, primarily by researchers in glass science, geochemistry, and diffusion studies. The field of archaeology has gone through periods of great optimism (e.g., Friedman and Long 1976) and of complete disillusionment (e.g., Anovitz et al. 1999;

Ridings 1996). In recent years, the field of OHD has benefited greatly from the rigorous application of physics, geochemistry, and glass science, so that the basic physics and mathematical models are now understood, and are the basis for the present treatment. The history of OHD has recently been summarized by Liritzis and Laskaris (2011) and by Rogers and Stevenson (2020).

The OHD method described here is based on usual archaeological practice in the western United States. The obsidian specimens are grouped by geochemical source, and a hydration rate is ascribed to the source. The hydration rate is not adjusted for the intrinsic water content of the individual specimen. Hydration measurements are made by optical microscopy, and temperature corrections are made by calculation from meteorological records or on-site temperature measurements. Both age and age standard deviation are computed by physics-based mathematical models.

Analytical methods that are not general archaeological practice at present are not addressed here. Some of these include Secondary Ion Mass Spectrometry (Anovitz et al. 1999, 2004; Liritzis and Laskaris 2012; Riciputi et al 2002); Fourier Transform Infrared spectroscopy (Newman et al. 1986) and Infrared Photo-Acoustic Spectroscopy (Stevenson and Novak 2011).

SCIENCE OF OBSIDIAN HYDRATION DATING

Hydration and its Measurement

In its most basic aspect, “obsidian hydration” describes the process by which water is absorbed by obsidian, and involves both physical and chemical changes in the glass (Anovitz et al. 2008; Doremus 2002; Kuroda and Tachibana 2019; Kuroda et al. 2018, 2019). When a fresh surface of obsidian is exposed to air, water molecules adsorb on the surface. Adsorption is a chemical bonding process, not simply condensation, and the adsorbed layer may be many molecules deep (Kuroda and Tachibana 2019; Kuroda et al. 2018, 2019). Some of the adsorbed water molecules, as well as other water molecules impinging directly from the atmosphere, are absorbed into the glass and diffuse into the interstices in the glass matrix. The process occurs when a water molecule has sufficient energy to stretch the glass matrix and enter one of the interstices.

Some of the diffusing H₂O molecules react with the silica or alumina in the glass, forming hydroxyl (OH) and causing an increase in volume and openness of the hydrated region. Since the hydrated region is expanded and the non-hydrated region is not, a stress region exists between the two. The stress region is visible under a polarizing microscope due to stress birefringence. As time passes, the region of increased water concentration progresses into the glass, its rate of progress being a function of the initial openness of the glass, temperature, and the dynamics of the process itself. When the hydrated layer becomes thick enough, typically greater than 20 μ , the accumulated stresses cause the layer to spall off as perlite.

The depth of water penetration is measured by determining the depth of the stress zone. A thin slice is cut from the margin of an artifact with a diamond saw, mounted on a microscope slide, polished to transparency, and observed under a polarized light microscope. The thickness of the hydrated layer (the “rim” or “rind”) is on the order of microns, so typically a petrographic microscope system with an optical magnification of 400X or more is used. The rate of hydration can be determined by any number of methods (Rogers and Stevenson 2020), the equation relating penetration depth to time is known, and thus age can be computed. The age accuracy, measured by age standard deviation, can also be computed. The resulting ages and standard deviations are accurate enough to place an artifact in the correct archaeological period and answer interesting anthropological questions (Rogers and Yohe 2014, 2020).

Obsidian Mineralogy

Obsidian is an aluminosilicate glass, formed by rapid cooling of rhyolitic magma. Like any other glass, obsidian is not a crystal and lacks the lattice structure typical of crystals at the atomic level, but it does possess a matrix-like structure exhibiting some degree of short-range spatial order (Doremus 1994:27, Figure 2; 2002:59-73). Obsidians are typically about 74% silica (SiO₂) and about 14% alumina (Al₂O₃) by weight, the remainder being matrix modifiers (mostly alkaline oxides) and trace elements (mostly rare earth elements), some of which are source-specific (Doremus 2002:109, Table 8.1; Hughes 1988; Stevenson et al. 2019; Zhang et al. 1997). The trace elements provide the means for geochemical sourcing for provenance studies. The anhydrous composition (chemical composition independent of water) of obsidians from a wide variety of sources has been shown to be remarkably consistent, within a few tenths of a weight percent (Zhang et al. 1997). The minute interstices within the glass matrix, on the order of 0.1-0.2 nanometers in diameter, are where water penetration takes place.

All obsidians also contain small amounts of natural water, known as intrinsic water or structural water, resulting from the incomplete degassing of the magma during its ascent from the magma chamber. The amount is generally < 2 wt.% in natural obsidians, although cases of somewhat higher concentration are occasionally encountered (Newman et al. 1986; Stevenson et al. 2019). The effects of water on hydration rate are discussed below.

FOUR RECENT ADVANCES

Form of the Age Equation

Many forms of the age equation have been published over the years (e.g., Basgall 1990; Bettinger 1989; Friedman and Long 1976; Pearson 1994), but only the equation of Friedman and Long (1976) is based on the physics of the process. Hydration of obsidian is a diffusion process (Doremus 2000, 2002); by definition, diffusion is a process in which mass is transported due to a concentration gradient (Crank 1975). All laboratory data (e.g., Rogers and Duke 2011; Stevenson and Scheetz 1989; Stevenson et al. 1998, 2019) and theory (Crank 1975; Doremus 2002; Ebert et al. 1991) indicate that the position of the stress zone due to hydration progresses into the obsidian such that depth is proportional to t^n , where t is time and $n = 0.5$ within limits of experimental error. Thus, the age equation which should be employed is

$$t = r^2/k \quad (1)$$

where t is age in calendar years, r is rim thickness in microns, and k is the hydration rate in μ^2/year (Rogers 2007, 2012). No other equations for age are valid. If data from an archaeological site seem to conform better to another equation, it is because of experimental errors in the data; archaeological data are not sufficiently accurate to question equation (1) (Rogers 2006).

Controlling for Temperature Effects

The hydration rate (k in equation (1)) is a strong function of temperature, which in the archaeological case is time-dependent. The temperature dependence is described by the familiar Arrhenius equation,

$$k = A \exp [-E/(RT)]. \quad (2)$$

If k is in μ^2/year , then A is a constant in the same units, E is the activation energy of the diffusion process in J/mol , R is the universal gas constant ($8.315 \text{ J/mol } ^\circ\text{K}$), and T is absolute temperature in Kelvins (Kelvin = Celsius + 273.15). Thus, since the temperature undergoes both annual and diurnal variation, k varies as well. The key concept in controlling for temperature in archaeological chronometrics is the *Effective Hydration Temperature* (EHT), which is a single temperature that yields the same hydration results as the actual varying temperature over the same time. Due to the mathematical form of equation (2), EHT is always higher than the mean temperature (except in the uninteresting case of a constant temperature, in which case they are the same). Further discussion is in Rogers (2007, 2012).

The EHT is computed by integration of equation (4) over a mathematical model of the time-varying temperature. Details of the computation are in Rogers (2007, 2012) and the application of this method to controlling for temperature is described in Rogers and Stevenson (2020). A simple equation for archaeological use has been developed, which yields EHT values within 0.25°C of the more complex computation:

$$\text{EHT} = T_a + 0.0062*(V_a^2 + V_d^2) \quad (3)$$

The annual average temperature is T_a ; the annual variation, V_a , describes the variation of monthly average temperatures through the year; the mean diurnal variation, V_d , describes the daily hot and cold cycle. All temperatures in equation (3) are in $^\circ\text{C}$. These parameters can be determined from meteorological records or from temperature sensors. The V_a and V_d terms also need to be corrected for burial depth (Rogers and Stevenson 2020).

To yield a valid age, both k and t in equation (1) must be for the same EHT. The EHT computed from equation (3) is used to adjust the measured hydration rim data to the same EHT as the hydration rate, after which age and age accuracy can be computed (Rogers and Stevenson 2020).

Water Content and Hydration Rate

Obsidian anhydrous chemistry has traditionally been regarded as having a major influence on hydration rate (see attempts to determine a chemical index to hydration (e.g., Friedman and Long 1976; Stevenson and Scheetz 1989). However, Stevenson et al. (1998, 2000) found no consistent influence of anhydrous chemistry on hydration rate. Behrens and Nowak (1997) and Zhang and Behrens (2000) found the effect of anhydrous chemistry to be negligibly small, although Karsten et al. (1982) reported that Ca^{2+} concentration may influence hydration rate to a very slight extent. It now appears that anhydrous chemistry has a negligible effect on hydration rate and attempts to predict hydration rate from anhydrous composition are unlikely to succeed.

Intrinsic water, on the other hand, has a profound affect on hydration rate since it impacts the openness of the glass structure during formation of the glass from a melt (Behrens and Nowak 1997; Delaney and Karsten 1981; Karsten et al. 1982; Lapham et al. 1984; Rogers 2015; Stevenson et al. 1998, 2000; Zhang and Behrens 2000; Zhang et al. 1991). Shelby (2005:145ff.) described the effects of adding network modifier atoms to a glass melt. A melt of silica and alumina is a liquid, but as the temperature is lowered the silica-alumina network starts to form. If there are no modifier ions present, the network forms with the interatomic spacing characteristic of its composition, approximately 0.086 nanometers for glass (Doremus 2002:67). If

modifier ions such as water are present the glass has to form interstices around them which are larger than the interstices in the modifier-free case (Shelby 2005:145). The radius of a water molecule is in the range of 0.138 to 0.233 nanometers (Doremus 2002:63), so the diameter is roughly 0.4 nanometers. This leads to much larger interstices than for the water-free case (Ambrose and Stevenson 2004; Garofalini 2020; Stevenson et al. 2019). These larger interstices represent voids in the glass matrix, which can be more easily penetrated by water molecules. Thus, increasing the structural water content leads to increased hydration rate (Kuroda and Tachibana 2019; Kuroda et al. 2018, 2019).

Stevenson et al. (1993) analyzed the intrinsic water content of obsidians from the Coso Volcanic Field (CVF) source in eastern California. The CVF was known to have four geochemically distinct sub-sources (Hughes 1988), and Stevenson demonstrated that the mean intrinsic water content of the sub-sources varied between sub-sources, and that there was significant variation within each sub-source. The variation in intrinsic water within a geochemical source or sub-source leads to variations in hydration rate, which in turn increases the uncertainty (statistical error) in computed ages (Rogers 2015). The effects of these errors have been analyzed in detail in Rogers (2008, 2010).

From a practical standpoint, controlling for geochemical source functions as a proxy for controlling for intrinsic water (Stevenson et al. 2000), albeit rather poorly (Rogers 2008; Stevenson et al. 1993). Geochemical sourcing controls for the mean value (central tendency) of intrinsic water in the obsidian from that source; the uncontrolled intra-source variation in water content contributes to uncertainty in the age and is reflected in the standard deviation of age.

Age Accuracy

In obsidian hydration dating, the primary error sources affecting the computed age are: obsidian rim thickness measurement; errors in the hydration rate ascribed to a source; intra-source rate variability due to uncontrolled intrinsic water in the obsidian (Ambrose and Stevenson 2004; Rogers 2008, 2010; Stevenson et al. 1993, 2000; Zhang and Behrens 2000; Zhang et al. 1991); errors in reconstructing the temperature history (EHT) (Rogers 2007, 2012); and errors in association or temperature history caused by site formation processes (Schiffer 1987). The mathematics to compute standard deviation of age were developed in Rogers (2010).

Recently, an accurate but simplified method of computing age accuracy has been developed, based on typical values of error sources (Rogers and Yohe 2021). This analysis shows that the standard deviation of age (σ_t) is

$$\sigma_t = t * \text{sqrt}[(0.16/r_m)^2 + 0.007*k - 0.0581] \quad (4)$$

where t is the age computed from equation (1), r_m is the measured hydration rim value in microns, and k is hydration rate in $\mu^2/1000$ years at 20°C. If this method is used, any uncertainty due to site formation processes must be added separately.

AREAS OF FUTURE RESEARCH

Despite its current utility, there are limitations to the OHD method. We have effective mathematical models for the effects of intrinsic water and temperature; the primary limitations to OHD accuracy and

precision occur because these models are not developed for the specimen but for the environment. For example, the temperature model is developed for the site and burial depth, but not for the specific obsidian specimen, and humidity can only be accounted for statistically. Similarly, hydration rates are computed for geochemical sources, but do not account for the intrinsic water in a particular obsidian artifact. As a result, OHD dates typically have relatively poor precision (age uncertainties of ~10-25%, with faster hydrating obsidians giving poorer accuracy). It is unlikely that further refinement of the present process will lead to dramatic improvements in precision with the current approach to controlling for temperature and intrinsic water.

Looking to the future, there are four areas of research that promise significant improvements. The first is a method for measuring the intrinsic water content of the individual specimen cheaply, quickly, and without damage to the specimen. Such a method would enable the hydration rate to be computed for the individual artifact rather than being ascribed to the geochemical source and would lead to a dramatic improvement in age precision.

A second improvement would be a method to determine the temperature history an artifact has experienced by a measurement on the artifact itself, or “intrinsic EHT.” Possible phenomena being investigated are the water speciation process in glass and index of refraction. Much more research is needed in this area.

A third area for research is to develop a quantitative understanding of hydration at the molecular level. The present diffusion models are macro-level, phenomenological models. They are based on understanding and including molecular-level effects, but the models themselves are higher level. The goal of research in this area is a mathematical model which starts with the structure and composition of the glass matrix and allows computing both a hydration rate and the speciation reaction.

A final area of research is much less glamorous, but is sorely needed: a method for measuring the extent of hydration without damage to the artifact. Current methods are consumptive, in that they require cutting a small piece of obsidian from the margin of an artifact, and land management agencies and museums are often unwilling to allow damage to artifacts. The method would need to be fast, cheap, non-damaging, and applicable to a wide range of artifact types and sizes. A method meeting these criteria does not currently exist.

SUMMARY OF RECENT ADVANCES

It has been nearly 60 years since the original obsidian hydration dating work of Friedman and his colleagues, and their work has stood the test of time. In succeeding years, other researchers, primarily in glass science and geochemistry, developed the field further. The archaeological advances over the past decade, which are the subject of this article, have been chiefly the result of applying numerical modeling to the known physical and chemical basis of hydration. Key points are discussed here are:

- First, the appropriate archaeological age equation for OHD is equation (1). No other mathematical form is valid, and should be avoided.
- Second, the primary environmental factor affecting OHD is temperature. Its effects are summarized by the Effective Hydration Temperature, which can be computed from equation (3). Methods for applying EHT in age computation have been developed and published.
- Third, the primary compositional factor affecting hydration rate is the intrinsic water content of the obsidian, whose causes and effects are now understood. Geochemical sourcing of the obsidian, and

ascribing a rate to the source, controls for mean value of water content; the effects of intra-source variation in water content are included in the accuracy computation in equation (4).

- Fourth, both age and age accuracy can be easily computed for OHD. Current models enable computing both an age and a standard deviation of the age, and sources of uncertainty contributing to the standard deviation are known.

Thus, at present, the mechanism of hydration is well understood at a macro level and can be successfully applied to archaeological problems.

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