Obsidian hydration: A new paleothermometer

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ABSTRACT

The natural hydration of obsidian was first proposed as a dating technique for young geological and archaeological specimens by Friedman and Smith (1960), who noted that the thickness of the hydrated layer on obsidian artifacts increases with time. This approach is, however, sensitive to temperature and humidity under earth-surface conditions. This has made obsidian hydration dating more difficult, but potentially provides a unique tool for paleoclimatic reconstructions. In this paper we present the first successful application of this approach, based on combining laboratory-based experimental calibrations with archaeological samples from the Chalco site in the Basin of Mexico, dated using stratigraphically correlated ^{14}C results and measuring hydration depths by secondary ion mass spectrometry. The resultant data suggest, first, that this approach is viable, even given the existing uncertainties, and that a cooling trend occurred in the Basin of Mexico over the past 1450 yr, a result corroborated by other paleoclimatic data.

Keywords: obsidian, hydration, secondary ion mass spectrometry, Pachuca, Basin of Mexico, paleoclimate, paleotemperature.

INTRODUCTION

Obsidian hydration dating (Friedman and Smith, 1960; Friedman et al., 1966; Friedman and Long, 1976; Ambrose, 1976; Michels et al., 1983; Anovitz et al., 1999, 2004; Riciputi et al., 2002) is based on the idea that a freshly broken obsidian surface begins to absorb water from its environment almost immediately. Absorption continues with time, generating a hydrated layer whose thickness is proportional to the time the glass surface was exposed. While this approach is conceptually simple, the technique has, generally, not proven successful. In part this is because a number of factors affect the absorption rate, some of which are difficult to determine in field-based studies. Key factors include the temperature and relative humidity of the environment. Even if modern temperatures are measured accurately, under typical soil conditions paleotemperature variations of only a few degrees are sufficient to measurably change the predicted hydration thickness, and thus to dramatically affect derived dates.

This environmental sensitivity has, however, potential benefits. If both the hydration rate as a function of temperature and relative humidity and the ages of the samples are known, the average conditions over the lifetime of the sample can be calculated. Obsidian hydration can, therefore, potentially provide a direct monitor of paleoclimate.

To test this hypothesis we have compared experimentally calibrated hydration rates for obsidian from the Pachuca source (Tenorio et al., 1998; Lighthart, 2001), located in the Sierra Madre Oriental in Mexico, with archaeological artifacts of the same obsidian. Pachuca obsidian is green and of very high quality, and thus is relatively easily identified in archaeological deposits. It was commonly used for tools and widely traded in ancient Mesoamerica (e.g., Moholy-Nagy, 1999; Ponomarenko, 2004), and thus this study can potentially be expanded to evaluate regional climatic trends. It sometimes shows a schiller due to microscopic vesicles, but materials were selected for our experiments that were largely free of macroscopic inclusions.

The naturally hydrated samples selected for this analysis were obtained from the Chalco site, located along the southeastern shore of Lake Chalco in the Basin of Mexico. This site provides a relatively high-quality data set with which to test and calibrate this approach. Archaeological materials naturally form a significant reservoir from which to draw samples for this sort of analysis. However, this technique is not limited to them, and well-dated nonanthropogenic samples should also provide useful data.

The ancient city-state of Chalco was the principal city of the Chalca during Early Postclassic time (750–1200 CE), and one of 40 major urban centers in the Basin of Mexico during the Late Postclassic (1200–1521 CE; Sanders et al., 1979; Parsons et al., 1982; Hodge, 2006). The artifacts analyzed here were recovered during the 1992 excavation of Mound 65 by Hodge and coworkers. Analysis of ceramic artifacts revealed two major occupations. The first dates to the Epiclassic and spans the period 750–950 CE (Sanders et al., 1979). The second spans the Aztec period (Sanders et al., 1979), from 1150 to 1519 CE. Mound 65 was also relatively rich in samples suitable for radiocarbon dating, and the selected obsidian samples were stratigraphically correlated with high-quality ^{14}C results (Elam, 1993). The radiocarbon dates indicated occu-

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TABLE 1. DERIVED TEMPERATURES FOR ARCHAEOLOGICAL SAMPLES FROM CHALCO, MEXICO

Sample	Median intercept date*	Temperature	2σ uncertainty	~Depth below surface
	(CE)	(°C)	(°C)	(m)
CHO110	1525	21.63	2.75	1.5
CHO110	1525	23.00	2.77	1.5
CHO086	1365	20.28	2.72	0.9
CHO081	1355	21.16	2.74	0.95
CHO081	1355	21.96	2.76	0.95
CHO120	1245	20.21	2.72	1.95
CHO015	1155	20.79	2.73	1.0
CHO018	1155	21.36	2.74	1.05
CHO047	645	25.84	2.83	1.55
CHO055	560	25.42	2.82	1.2
CHO051	560	26.41	2.84	1.3
CHO051	560	25.74	2.83	1.3

*Median intercept dates are based on reported ^{14}C analyses.

pations dating from 400 to 1620 CE with a hiatus (abandonment) from 900 to 1100 CE.

NATURAL AND EXPERIMENTAL DATA

In order to calculate paleotemperatures from hydrated obsidians, at least three factors must be known: the ages of the samples, the hydration profile in the samples, and the temperature/time coefficients of the diffusion equation. For this test of the method, the first two were obtained from our investigations of samples from Chalco. Samples were chosen for analysis for which stratigraphically equivalent ^{14}C determinations were available. Depth profiles were then obtained from these selected artifacts using secondary ion mass spectrometry (SIMS; Anovitz et al., 1999, 2004; Riciputi et al., 2002).

In addition to the results obtained from the obsidian samples, an additional temperature-time point can be obtained by measuring current average yearly temperatures at the Chalco site. As the hydration rates for buried samples represent soil, rather than air temperatures, modern soil temperatures are needed. For archaeological applications, soil temperatures and relative humidities are commonly measured using Ambrose cells (Ambrose, 1976, 1980; Trembour et al., 1988). Four pairs of such cells were buried in Mound 65 for 1 yr (1994–1995) at depths of 25, 50, 100, and 200 cm below the surface of the mound (Riciputi et al., 2002). Of these, the uppermost cell, buried 25 cm below the surface of the mound, yielded a slightly higher temperature (20.3 °C) and lower relative humidity (RH) (95%) than the other three (18.9–19.3 °C, 97%–100% RH). This suggests that there may be a small thermal gradient in the upper few centimeters of the soil profile at this site, but that it does not extend far below the surface. The depths from which the naturally hydrated samples were recovered are shown in Table 1. All samples were recovered well below this surface zone, although they may have spent part of their burial history at near-surface conditions.

The coefficients of the diffusion equation were obtained from a series of isothermal experiments run at 30, 50, 75 and 150 °C for times ranging from 1 day to several years. The method used for these experiments (Anovitz et al., 2004) involved hydration of small polished obsidian blocks in closed vessels with the samples suspended in the vapor phase to prevent dissolution. Hydrogen depth profiles for both the naturally hydrated and experimental samples were obtained using SIMS.

Figure 1 shows the hydration depths of our experimental and natural samples as a function of time. The extent of hydration in these samples is represented in this figure by the half-fall distance (H_F), the distance at which the concentration (C_{HF}) is the average of the maximum (or surface) and background values [$C_{HF} = (C_{max} + C_{background})/2$]. Numerical analysis has shown that this datum accurately captures the diffusion rate (Riciputi et al., 2002; Anovitz et al., 2004).

As can be seen from Figure 1, the experimental results at each temperature (T) are close to log-linear. The experimental data set can be fitted to the expression:

$$\begin{aligned}\mathrm{Log}_{10}(H_F) = {} & 2.470(\pm 0.031) \\ & - 1911.402(\pm 20.191)/T(\mathrm{K}) \\ & + 0.4235(\pm 0.0078)\log t(\mathrm{s}),\end{aligned} \tag{1}$$

with an R^2 value of 0.9951 and standard error (1σ) of 0.030 (the values in parentheses are the 1σ errors of the coefficients). Because the standard error in this equation is relatively small, it can be used to calculate paleotemperatures with reasonable accuracy. In addition, an interesting aspect of this equation is that the time exponent is not equal to 0.5, and the difference is statistically significant. The assumption of square-root-of-time behavior is common in many diffusive systems (cf. Crank, 1975), and has been the standard assumption in obsidian hydration dating (cf. Friedman and Smith, 1960). However, as previously pointed out (Anovitz et al., 1999, 2004; Riciputi et al., 2002), the complexities involved in the obsidian hydration process at temperatures below the glass transition are sufficient to question this assumption, and the experimental data appear to confirm its inaccuracy.

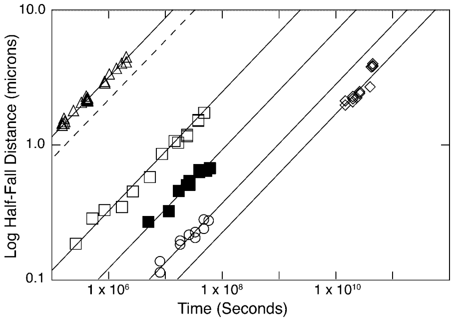

Figure 1. Hydration depth of Pachuca obsidian as function of time and temperature. Data shown are derived from laboratory experiments and archaeological samples. Solid lines were fitted to the experimental data (equation 1). Triangles—150 °C, open squares—75 °C, solid squares—50 °C, circles—30 °C, diamonds—Chalco samples. Dashed line shows experimentally determined shift in half-fall distance when relative humidity at 150 °C is reduced from 100% to 20%.

PALEOCLIMATIC RESULTS AND DISCUSSION

Equation 1 can be used to obtain temperatures for the naturally hydrated samples of Pachuca obsidian if, as in the case of the Chalco samples, the ages of the samples are known and the half-fall depths of their hydration profiles have been measured. Table 1 shows the temperatures obtained from these samples. The results fall into two groups. Assuming 100% relative humidity, artifacts manufactured between 450 and 900 yr ago yield an average temperature of 21.3 ± 0.33 °C, while those manufactured between 1350 and 1450 yr ago yield 25.9 ± 0.21 °C. The uncertainties quoted are 1σ values about the mean, and the differences between the two data sets are clearly statistically significant.

However, because these temperatures are obtained using a diffusion process that began at the moment of artifact manufacture and continued from then to the present, the results obtained represent characteristic temperatures (cf. Shewmon, 1963; Chakraborty and Ganguly, 1991) over the entire time period, not time-specific values. A characteristic temperature is the integral of the temperature-time curve, divided by the total time. Thus, the effects of variations in seasonality, daily fluctuations, or even an abnormally hot decade followed by an abnormally cold decade or

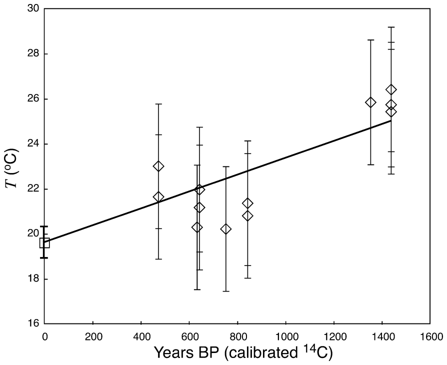

Figure 2. Evolution of soil temperature (*T*) as function of time in the Basin of Mexico. These temperatures have been calculated assuming that relative humidity of soil remained at 100% over time. Magnitude of uncertainties is 2σ error in fit to experimental data (equation 1). Curve shows fitted temporal evolution (equation 2). Note that data may also suggest a more complex nonlinear thermal pattern.

other changes over the time period of interest only affect the result insofar as they change the total integral. For two samples of the same age and different detailed temperature-time paths, the results will be identical as long as the integral remained unchanged. Thus, the key to obtaining more detailed temperature-time data is the acquisition and analysis of samples of different ages. For example, in the alternating hot decade–cold decade scenario this excursion may be discernable by examining glasses deposited before, during, and after these episodes.

Figure 2 shows the characteristic temperatures at Chalco as a function of time. The error brackets for the modern soil temperature represent the spread in the Ambrose cell results (including the value nearest the surface), while those for the archaeological specimens represent the 2σ error in the fit to the experimental data. Given the uncertainties and absence of a compelling reason for an alternate choice, a linear equation is the simplest and most reasonable mathematical form with which to fit the temperature-time data. With the intercept fixed at the Ambrose cell average this yields:

$$T\,(^\circ\mathrm{C}) = 19.6 + 1.1877\mathrm{E} - 10(1.2293\mathrm{E} - 11) \times t(\mathrm{s}), \quad (2)$$

where the value in parentheses is the 1σ uncertainty in the slope, and the overall fit has an R^2 value of 0.89 and a standard error of 1.33 °C. The t statistic of the slope (equal to the coefficient divided by its standard error) is 9.66. Thus the value of the slope is statistically nonzero at a rather high confidence level. Examination of the individual data points may also indicate a more complex evolution of temperature with time. This suggests that more specific paleoclimatic events may be discernable using this technique by examining results from suites of samples, but additional samples are required to verify this pattern.

The uncertainties described here only consider the fitting error in equation 1. Two additional sources of error are the depths of the SIMS profiles and the ^{14}C dates. The former add a temperature uncertainty of ~0.44° for the younger samples and ~0.26° for the older (1σ errors). Assuming that the association between the ^{14}C dates and the obsidian samples are correct, the reported errors in the ^{14}C dates (Riciputi et al., 2002) add 1σ uncertainties of 0.89° and 0.57° for the younger and older samples, respectively.

These uncertainties must be combined to ascertain whether the temperature change observed is statistically significant. Student's t-test shows that, for the combined uncertainty, the mean of the older data set differs from the modern value at the 99.985% confidence level. Thus, while the absolute magnitude of the trend could be refined, the conclusion that soil temperatures have cooled over the past 1450 yr is relatively robust.

While our data clearly show a decrease in apparent soil temperature in the Basin of Mexico from 1450 yr ago to the present, the absolute value of this decrease, ~5 °C, appears rather high, although tree-ring results (cf. Cronin, 1999) commonly suggest temperature variations of this magnitude. The magnitude of the suggested temperature change may, in part, be attributable to the uncertainties in the fit to, and extrapolation of, the experimental data. If we consider the highest value obtained from the modern Ambrose cell measurements (20.3 °C), and the constraining value at the lower 2σ confidence level from the older Chalco samples (23.5 °C at 560 CE.), the result still suggests significant temperature change. Adding the uncertainties in the older archaeological samples due to depth measurements and ^{14}C ages further reduces the limiting value of the older samples, but does not eliminate the trend.

The temperatures derived from this technique are time-integrated or characteristic, not isotemporal values. Conversion of these integrated soil temperatures into temperature-time profiles involves a nonunique convolution, as the total integral is path independent. Nonetheless, some approximations of the actual temperatures can be obtained using the characteristic temperature concept. If we assume that the characteristic temperature for the past 430 yr has been 21.3 °C, then the characteristic temperature from 1480 to 430 B.P. becomes 27.7 °C, and if we reduce only the characteristic temperature of the older samples by 2σ (to 23.8 °C), the characteristic temperature of the older time segment becomes 24.8 °C, which still robustly leads to the conclusion that soil temperatures in the Basin of Mexico have, on average, decreased over the past 1450 yr.

EFFECTS OF RELATIVE HUMIDITY

All of the above calculations are based on the assumption that the relative humidity in the environment around the hydrating sample was a constant 100%. It is well known, however, that variations in relative humidity can affect the diffusion rate. Thus the thermal decline discussed here could reflect changes in relative humidity as well as temperature (Mazer et al., 1991; Friedman et al., 1994). In general, lowering the relative humidity reduces the overall hydration rate. Thus, the archaeological samples could reflect an average hydration environment that was either warmer or wetter than today.

Measurements of the effect of relative humidity (RH) on the hydration of Pachuca obsidian (Fig. 1, dashed line) suggest, however, that this effect is relatively small. Measured half-fall distances decrease by only 30% with a decrease in RH from 100% to 20%. In addition, natural data (Mazer et al., 1991; Friedman et al., 1994), suggest that, even in relatively dry environments, relative humidities are near 100% below 50 cm of the surface, while values in the upper layers are typically above 65%. This is consistent with the Ambrose cell results at Chalco. Adjusting the temperature differences between the older and younger Chalco samples on the basis of RH alone would require an average RH for the younger samples of 46%, assuming that the older samples experienced a constant RH of 100%. This is clearly unreasonable, and it is likely that most of the observed change represents thermal, rather than RH variations. Thus, while paleoclimatic reconstructions based on obsidian hydration undoubtedly reflect integrated changes in temperature and relative humidity, the effects of the latter appear to be relatively minor.

COMPARISON WITH OTHER SOURCES OF PALEOCLIMATIC DATA

The data obtained here mirror other results in the Basin of Mexico. Several lines of evidence (isotopic, chemical, palynological, ostracod) suggest that a severe period of aridity occurred between ~800 and 1000 CE, approximately correlated with the period of abandonment at Chalco (Metcalfe et al., 2000; Hodell et al., 2001). If this was a warm and dry event, it would explain the warmer average temperatures recorded by the older artifacts. Data for paleoclimatic variations in the

Northern Hemisphere during the past 1000 yr (Mann and Jones, 2003) also suggest a general cooling trend until the last 100–150 yr, although data from Mexico suggest that there may have been several cycles of aridity and increased precipitation during this period, including a 500 yr dry period from ~1200 to 1700 CE followed by wetter conditions over the past 220 yr. Population collapse during the sixteenth century may have been related to a severe drought during this period (Soto et al., 2002; Cleaveland et al., 2003). The cooling of soil temperatures suggested by our data thus correlates well with hemispheric trends.

FUTURE DIRECTIONS

The data presented here are the first ever obtained in which obsidian hydration results have been used to constrain paleoclimate. This success is based on improved analytical techniques, a careful experimental calibration of the hydration process, and natural samples in which ^{14}C or other dates and obsidian samples can be correlated with reasonable certainty. Acquisition of further data of this type and quality should allow refinement of the technique, including analysis of isotemporal and polytemporal suites of artifacts in delineating detailed temperature-time variations, and provide a new tool for paleoclimatic reconstructions.

Successful applications of this technique will, however, depend on the sampling strategy and experimental and analytical protocols used. First, high-precision hydration profiles such as those provided by SIMS analysis are clearly necessary. Second, results strongly depend on the quality of the experimental calibration, and experiments should cover a sufficient temperature-time range to reduce the uncertainties to a meaningful level. Third, the artifacts must be securely dated by whatever means (e.g., ^{14}C) are available. Finally, the larger the number of artifacts, and the more thoroughly they cover the time period of interest, the more detailed and reliable the results are likely to be.

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