Hydration Rate of Obsidian

New experimental techniques allow more precise dating of archeological and geological sites containing obsidian.

Irving Friedman and William Long

Since Friedman and Smith in 1960 (*1*) described a new dating method using obsidian, many investigators have attempted to apply the technique, not only to archeology (*2–4*) but also to geology (*5*). The method depends on the fact that obsidian (rhyolitic volcanic glass) absorbs water from the atmosphere to form a hydrated layer, which thickens with time as the water slowly diffuses into the glass. The hydrated layer can be observed and measured under the microscope on thin sections cut normal to the surface.

To convert the measured hydration thickness to an age, the equation relating hydration thickness to time must be known—not only the form of the equation (functional dependence), but also the constants in it. In 1960 the only way to determine rates was to measure the hydration thickness on obsidian artifacts found in sites that could be dated historically or by the use of carbon-14. On the basis of a limited number of such artifacts from different parts of the world, Friedman and Smith concluded that (i) the hydration thickness increased as the square root of time, (ii) the rate was independent of relative humidity but increased with temperature, and (iii) the chemical composition of the obsidian appeared to affect the rate.

In 1966 Friedman *et al.* (*6*) published the results of an experimental hydration of obsidian from the Valles Mountains, New Mexico, at 100°C and 1 atmosphere steam pressure. Pieces of obsidian were exposed to these conditions for varying lengths of time, up to 4 years, and the hydration thicknesses were measured. Figure 1 shows that the hydration followed the rate equation

$$T = kt^{1/2} \qquad (1)$$

where T is the thickness of the hydration layer, t is time, and k is a constant.

Over the years, some of these conclusions have been challenged on the basis of archeological evidence (*3*). We have conducted additional experiments to check the original assumptions and to derive hydration rates as a function of both temperature and the chemical composition of the volcanic glass. It is now possible to calculate hydration rates (i) if the ground temperature where the obsidian has been buried is known by measurement or can be estimated from weather records, and (ii) if the silica content (percentage of SiO_2) or refractive index of the glass is known.

Experimental Hydration of Obsidian Samples

Obsidian samples of different compositions from 12 localities were hydrated at elevated temperatures and water pressures for various periods of time. After the experimental hydration, thin sections were cut and the hydration thickness was measured under the microscope. Data for obsidian from one location are shown in Fig. 2 as plots of the hydration thickness (micrometers) squared against time for temperatures of 95°, 150°, 195°, and 245°C. The hydration front tends to be more diffuse the higher the temperature (and water pressure) of hydration. The measurements are precise to ± 0.2 μm on samples hydrated at 95° and 150°C and ± 0.3 μm on samples hydrated at 195° and 245°C. On material hydrated at surficial temperatures, the hydration rind can be measured to ± 0.1 μm.

Figure 2 illustrates that the hydration, at temperatures from 95° to 245°C, proceeds at a rate that is proportional to the square root of time. The rate appears to be independent of water pressure, at least from 10 to 50 pounds per square inch (psi) at 150°, 50 to 200 psi at 195°, and 200 to 2000 psi at 245°C (1 psi = 6.80×10^{-2} atm).

Results

Using the hydration rates for each obsidian source at 95°, 150°, 195°, and 250°C, we can solve the Arrhenius equation relating the hydration rate to temperature

$$k = Ae^{-E/RT} \qquad (2)$$

where k is the hydration rate (micrometers squared per 10^3 years), A is a constant, E is the activation energy of the hydration process (calories per mole), R is the gas constant (calories per degree per mole), and T is absolute temperature (degrees Kelvin).

The plot of ln k against $1/T$ for the Icelandic samples is shown in Fig. 3. The slope of the line is equal to $-E/R$, and the intercept on the abscissa gives ln A. Table 1 gives the values of E/R and A for the 12 obsidian types investigated. Figure 4 shows plots of the extrapolated hydration rate for 12 of the obsidian samples as a function of temperature.

The rate of hydration clearly differs among the samples. For example, at 10°C the hydration rate for samples from Love Quarry is 0.22 μm^2 per 1000 years, whereas the rate for Obsidian Cliff samples is 5 μm^2 per 1000 years, a factor of about 20 difference between the slowest and fastest rates of the 12 samples investigated. The chemical analyses and refractive indices of the samples investigated are given in Table 2. Hydration rate should be related to chemical composition, and several authors have suggested relationships between rate and silica and alumina coupled with alkali (*7*) and the ratio of potash (K O) to alumina (Al_2O_3) (*4*). We do not observe any systematic relation between rate and these components, but do find a relation between rate and SiO_2 content and between rate and refractive index of the nonhydrated glass (Figs. 5 and 6). Other components of the glass also appear to affect the hydration rate. According to our data, increased SiO_2 content increases the hydration rate, but increased CaO and MgO reduce the rate. The Al_2O_3, FeO, Na_2O, and K_2O contents appear to have little effect on the rate, at least within the ranges found in our samples. A combination of components expressed as SiO_2-45(CaO + MgO)-20H_2O^+ (all percentages by weight; the H_2O is water liberated above 110°C) is more closely related to hydration rate (see Fig. 7) than is silica content alone. We refer to this parameter as the chemical index. This chemical index was derived empirically by varying the coefficients to fit the smoothest curve to the experimental hydration rates.

Obsidian from the East Lake Flow at Newberry Craters hydrates at a rate that appears to be too high, in light of its chem-

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ical composition. The reason for the apparently anomalous behavior is unknown.

Using the data collected in this study, we drew graphs of hydration rate plotted against temperature for obsidians of various chemical indices (Fig. 8). The curves are generalized and do not refer to obsidian samples from any specific locality. In constructing these curves, the data from the East Lake obsidian were not used.

In principle, if we can measure the temperature to which an obsidian sample has been exposed and know its chemical composition we can calculate its average rate of hydration.

An obsidian exposed to atmospheric temperature, either by being at the surface or buried in the ground at very shallow depth (~ 1 to 10 cm), will experience wide diurnal temperature fluctuations. Since the hydration rate varies as a power function of the temperature and not linearly, an average or mean temperature cannot be used to calculate a rate. Instead, Eq. 2 must be solved for k using a temperature that has acted for a small time interval, and then all the k's must be averaged to get an average rate for the total time.

In contrast, an obsidian that has been buried sufficiently deep that the diurnal temperature wave is damped out will be exposed to attenuated seasonal temperature variations, or even to a steady temperature. A single temperature can then be used to determine an average rate. The depths of burial to yield these conditions vary with soil diffusivity, albedo, snow cover, climate, and so forth, but usually samples buried to depths of at least 10 m will experience an extremely small seasonal variation; samples buried to depths of 0.5 m will experience almost no diurnal variation and a seasonal variation that is about one-third the variation of the average air temperature.

A comparison of the mean annual air temperature with the mean annual soil temperature at depths greater than 0.6 m measured at the same sites (*8*) shows that, in general, they agree to within 1°C for sites having little or no snow cover. For snow-covered sites the mean annual ground temperature is 1° to 3°C higher than the mean annual air temperature.

As an example of the use of direct measurement of soil temperatures to calculate

Table 1. Constants for the Arrhenius equation (Eq. 2) relating hydration rate to absolute temperature. Obsidian localities are: Obsidian Cliff, Yellowstone National Park, Wyoming; Cosco Hot Springs, U.S. Naval Ordnance Test Station, Inyo County, California; Grassy Lake, Grassy Lake quadrangle, Teton County, Wyoming (Welded tuff–Lava Creek Tuff); American Falls, Power County, Idaho (Welded tuff–Walcott Tuff); Panum Dome, Mono Craters, Mono County, California; East Lake Flow, Newberry Craters, Deschutes County, Oregon (east flow of East Lake flow); Xalpazquillo, Mexico; Kerlingerfjoll, Iceland; Medicine Lake, Big Glass Mountain, Siskiyou County, California; Clear Lake, south of Tower Lake, Lake County, California; Big Obsidian Flow, Newberry Craters, Deschutes County, Oregon; Love Quarry, outcrop on east side near summit of Teton Pass, Teton County, Wyoming.

Obsidian locality	A (μm^2 per 10^3 years)	ln A	E (cal/mole)	E/R (°K)
Obsidian Cliff	6.457×10^{15}	36.40	19,580	–9,851
Cosco Hot Springs	3.017×10^{15}	35.64	19,260	–9,687
Grassy Lake	4.601×10^{15}	36.07	19,710	–9,923
American Falls	9.000×10^{15}	36.74	20,140	–10,140
Panum Dome	6.279×10^{15}	36.38	20,020	–10,070
East Lake Flow	1.429×10^{15}	34.90	19,220	–9,668
Xalpazquillo	1.832×10^{16}	37.45	20,970	–10,550
Kerlingerfjoll	8.928×10^{15}	36.73	20,700	–10,410
Medicine Lake	7.708×10^{15}	36.58	20,740	–10,430
Clear Lake	1.494×10^{16}	37.24	21,210	–10,670
Big Obsidian Flow	1.488×10^{16}	37.24	21,370	–10,750
Love Quarry	1.172×10^{16}	37.00	21,650	–10,900

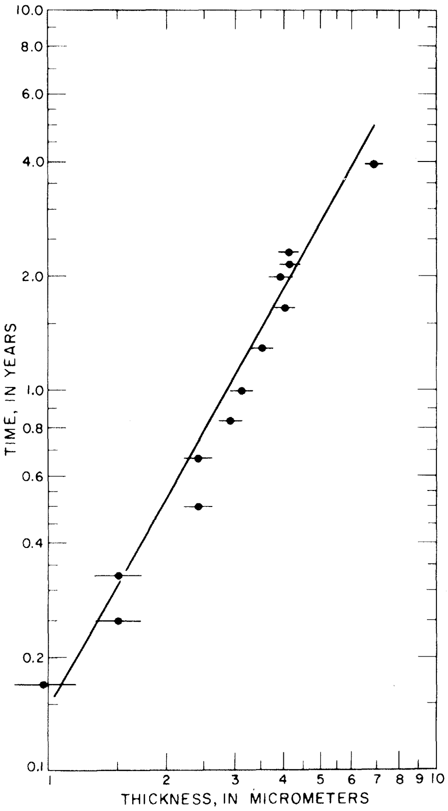


Fig. 1. Experimental hydration rate curve for obsidian at 100°C. [Reprinted from (*6*)]

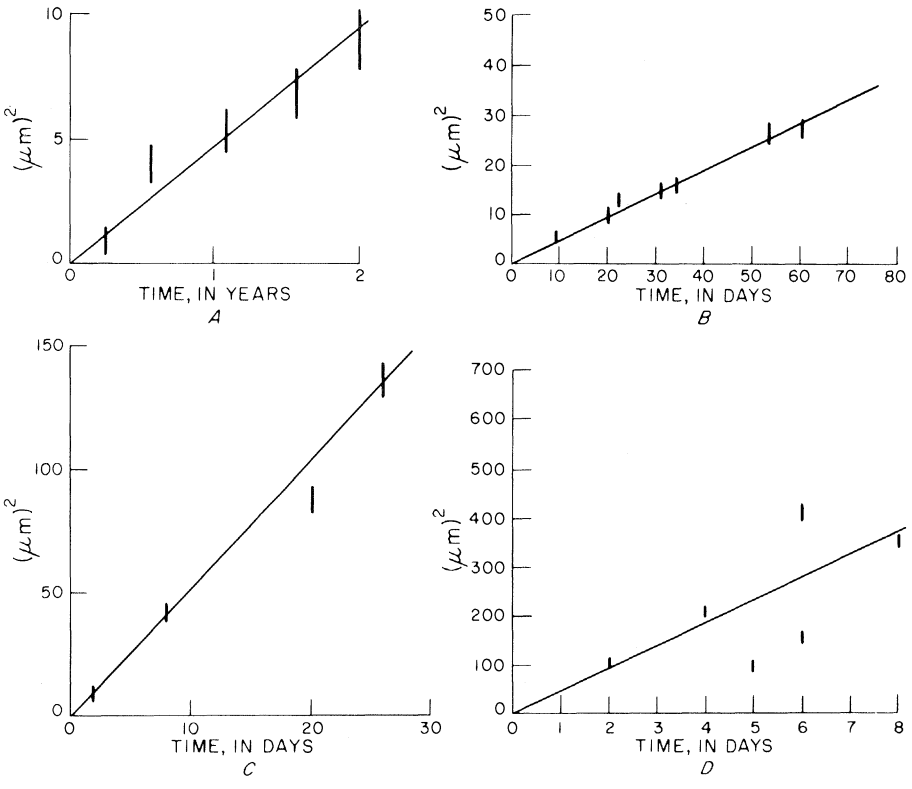


Fig. 2. Experimental hydration curves for obsidian from Kerlingerfjoll, Iceland. Hydration conditions were: (A) 95°C and 1 kg/cm^2 H_2O pressure; (B) 150°C and 3.5 kg/cm^2; (C) 195°C and 14 kg/cm^2; and (D) 245°C and 350 kg/cm^2.

effective hydration temperatures, we measured the ground temperature at several depths and the temperature of the surface of a fist-sized piece of black obsidian that was exposed to the sun. These measurements were made at several sites, and temperatures were recorded frequently for periods of more than 1 year. The sites for which we now have complete records are Gardiner, Montana; West Yellowstone, Montana; Pine Mountain Observatory (30 km east of Bend), Oregon; and Gutierrez Zamora, State of Veracruz, Mexico. In each case the temperature was read every 2 hours (for obsidian exposed to the sun) or every 5 days for soil temperatures, which changed slowly. These temperatures were then substituted into the Arrhenius equation (Eq. 2) using the constants (A and E) for the "local" obsidian. The average rates that we calculate are given in Table 3.

A piece of obsidian exposed to the sun will hydrate much more rapidly than buried obsidian (Table 3). This disparity in rate is much greater at high elevations, where solar heating is most effective, especially where snow cover is minimal.

The site near Gardiner, Montana, is about 100 m from a small hot spring. Our soil-temperature measurements show the presence of geothermal heat at the site.

Effective Hydration Temperature

The most satisfactory method of determining effective hydration temperature (EHT) is direct measurement at the archeological or geological site. A less accurate but still satisfactory method is estimation from mean air temperature measured at the site, or at stations having a similar temperature regime. Estimation from weather records is particularly successful in tropical climates, where the temperature varia-

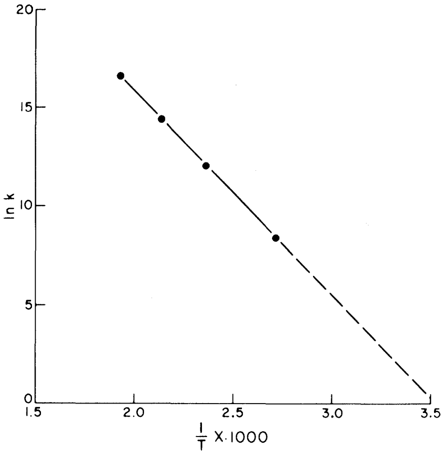


Fig. 3. Arrhenius plot for the experimental data for obsidian from Kerlingerfjoll, Iceland. The values of k are derived from the slopes of the curves in Fig. 2.

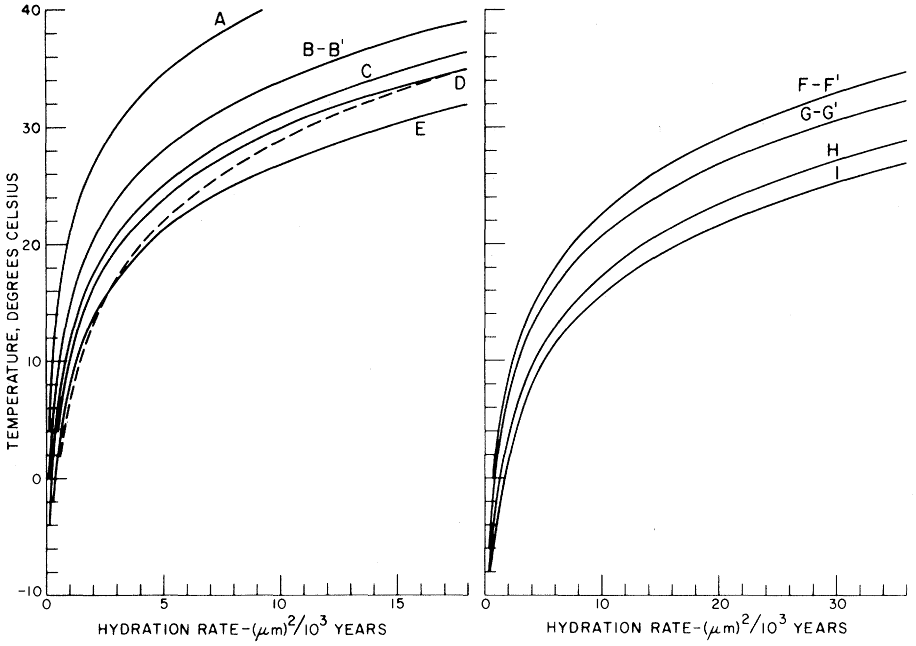


Fig. 4. Hydration rate as a function of temperature for the 12 obsidian samples: A, Love Quarry; B-B', Clear Lake and Big Obsidian Flow; C, Medicine Lake; D, Kerlingerfjoll; E, Xalpazquillo; F-F', East Lake and Panum Dome; G-G', Grassy Lake and American Falls; H, Cosco Hot Springs; and I, Obsidian Cliff. The dashed curve is from Suzuki (*4*).

Table 2. Chemical analyses and refractive indices of obsidian samples. The Big Obsidian Flow sample was analyzed by a single solution procedure described by Shapiro (*12*); the analyst was L. Mei. The other samples were analyzed by the methods described by Shapiro and Brannock (*13*), supplemented by atomic absorption; analysts were P. L. D. Elmore, J. L. Glenn, H. Smith, and J. Kelsey. All analyses were performed in the Rapid Rock Analysis Laboratory under L. Shapiro. Chemical components are expressed as weight percentages.

Chemical components	Locality											
	East Lake Flow	Big Obsidian Flow	Obsidian Cliff	Grassy Lake	Love Quarry	American Falls	Cosco Hot Springs	Clear Lake	Medicine Lake	Panum Dome	Kerlingerfjoll	Xalpazquillo
SiO_2	73.2	72.2	76.7	76.3	74.5	76.3	76.9	75.2	73.7	75.8	73.2	74.5
Al_2O_3	14.3	14.6	12.5	12.7	14.4	12.4	13.2	13.4	14.0	12.9	13.8	14.4
Fe_2O_3	0.55	0.72	0.43	0.59	0.29	0.52	0.16	0.19	0.57	0.36	0.84	0.00
FeO	1.5	1.7	0.88	1.1	0.92	0.80	0.76	1.1	1.3	0.76	1.6	0.68
MgO	0.28	0.32	0.09	0.09	0.18	0.10	0.02	0.18	0.32	0.07	0.13	0.07
CaO	1.0	0.92	0.48	0.58	1.3	0.52	0.45	1.0	1.2	0.60	0.99	0.50
Na_2O	4.5	5.3	3.3	3.4	3.9	3.2	4.0	3.3	4.0	3.9	4.7	4.2
K_2O	3.8	4.0	4.6	4.6	3.5	4.6	3.9	4.3	3.8	4.2	3.5	3.7
H_2O^+	0.54	0.31	0.24	0.11	0.35	0.29	0.34	0.21	0.23	0.23	0.13	0.92
H_2O^-	0.06	0.02	0.06	0.06	0.08	0.04	0.06	0.06	0.04	0.10	0.04	0.08
TiO_2	0.24	0.21	0.07	0.14	0.07	0.22	0.05	0.25	0.28	0.06	0.19	0.02
P_2O_5	0.04	0.06	0.00	0.00	0.03	0.02	0.00	0.03	0.04	0.00	0.02	0.04
MnO	0.05	0.06	0.03	0.04	0.07	0.05	0.04	0.03	0.04	0.05	0.07	0.13
CO_2	<0.05	0.01	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Sum	100	100	99	100	100	99	100	99	100	99	99	99
Refractive index	1.493	1.494	1.483	1.488	1.490	1.486	1.485	1.489	1.491	1.486	1.494	1.487

tions, both diurnal and seasonal, are small compared to those in temperate, arctic, and especially continental climates.

Several techniques have been used, or suggested, for measuring EHT. The first has already been discussed—namely, periodic measurement of the temperature of a buried sensor and reduction of the temperature data to EHT by use of the appropriate Arrhenius equation for the obsidian that has undergone hydration.

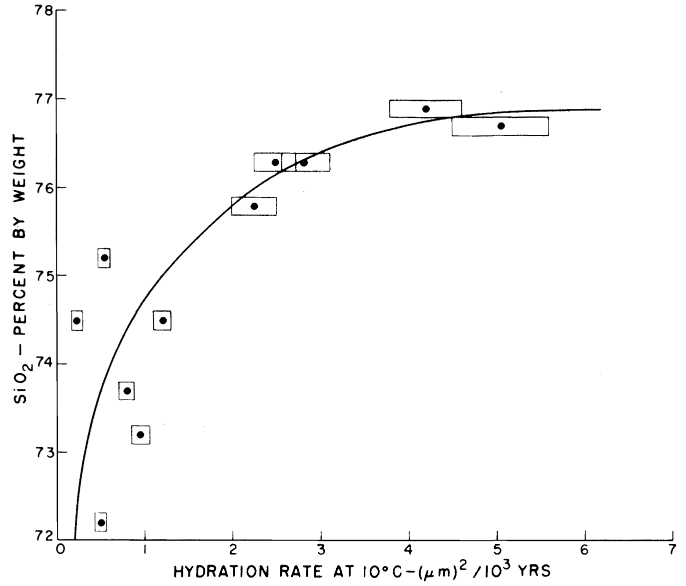
Another method is to use the Pallman technique of temperature integration (*9*). This method makes use of the fact that a solution of sucrose will slowly invert to two other sugars, D-glucose and D(–)-fructose. This reaction can be made to proceed slowly, so that it will not be completed during the 1-year period of the measurement. The reaction is monitored with a polarimeter, since the rotation of polarized light changes during the course of the reaction. The rate of reaction is temperature dependent, and the reaction therefore integrates the temperature it has been exposed to, at low cost. This technique has been used to determine mean annual temperatures of soils to 0.3°C and its use for determining EHT is now being investigated.

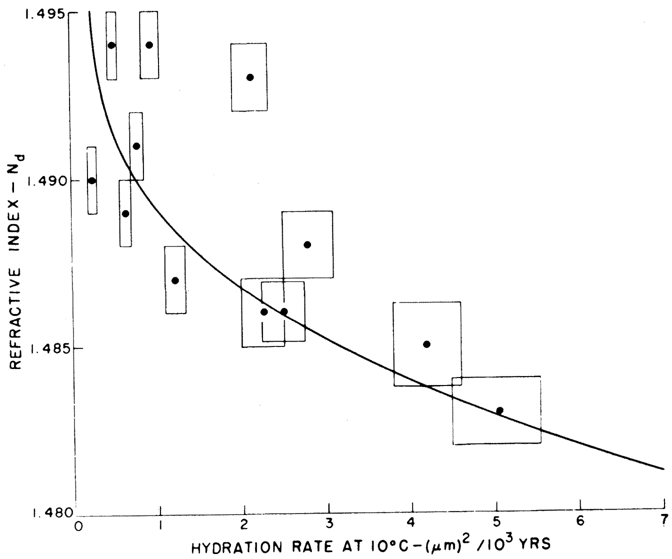
A third method is due to Ambrose (*10*), who uses a small sealed hollow plastic sphere filled with molecular sieve. This sphere is immersed in water, and the whole unit is then buried at the site for 1 year. Water slowly diffuses into the sphere and is adsorbed by the molecular sieve. The rate of diffusion of water through the plastic is temperature dependent. At the end of the year the sphere is reweighed. The gain in weight (gain of water) can be related to EHT.

Effective hydration temperatures have been estimated from published records of mean annual air and soil temperatures at stations having a similar temperature regime to the site in question. An example of the use of such data follows.

Comparison of Measured and Calculated Rate

The obsidian buried in the Hopewell Mounds in Ohio has been found, by an analysis of its minor element composition, to come from Obsidian Cliff, Yellowstone National Park, Wyoming. Mean annual ground temperature 20 cm under a bluegrass sod cover measured at a site in the vicinity of the archeological site that contained obsidian artifacts is 10°C. The mean annual air temperature at this site is 10.3°C. The estimated ground temperature





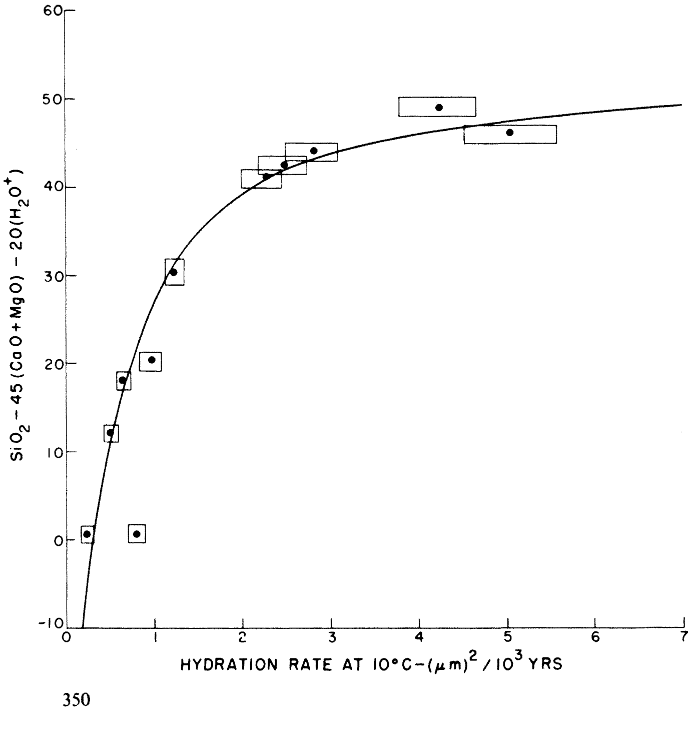


Fig. 5 (top left). Hydration rate at 10°C as a function of SiO_2 content for the 12 obsidian samples. Fig. 6 (top right). Hydration rate at 10°C as a function of refractive index (N_d) for the 12 obsidian samples. Fig. 7 (bottom). Hydration rate at 10°C as a function of the combination of components SiO_2-45(CaO+MgO)-$20H_2O^+$. The curve is drawn as a best fit to the experimental data.

at depths of 2 to 10 m (commensurate with the depths of burial of the obsidian artifacts) is 9.5°C. The calculated rate of hydration for obsidian of this composition at 9.5°C is 4.7 μm^2 per 10^3 years. The "measured" rate, from ^{14}C dating, is 4.0 to 5.2 μm^2 per 10^3 years, in reasonable agreement with the calculated rate.

The relationship between mean annual temperature and EHT is complex. The hydration process is an exponential function of temperature, while mean annual temperatures are linear averages of daily minimums and maximums. However, since the extremes of temperature at a depth of 2 m are small, mean annual soil temperature and EHT are close. Figure 9 is a plot of the measured soil temperatures for West Yellowstone, Montana (44°40′N, elevation 2025 m, and a very continental climate), and Gutierrez Zamora, Mexico, adjacent to the Santa Luisa archeological site (20°32′N, elevation < 10 m, and tropical). The mean annual soil temperature at a depth of 1.8 m for West Yellowstone is 5.7°C, while the EHT for this site (1.8 m) is 6.0°C. In the tropical site at Gutierrez Zamora, the mean annual soil temperature (1.8 m) is 24.8°C and the EHT is 24.8°C. At both the tropical and the north temperate, continental site, the mean annual ground temperatures are very close to EHT. It is necessary to be cautious in using mean annual air temperatures to approximate EHT in northerly regions with extensive winter snow cover. For example, the mean annual air temperature for West Yellowstone, Montana, is 1.7°C and the EHT at 1.8 m is 6.0°C.

Obsidian Hydration Rate

Measuring the hydration rate of a specific obsidian by hydrating it at an elevated temperature and water pressure appears to be satisfactory, in spite of the extrapolation that is necessary from high to low (surficial) temperatures. A nuclear reaction technique has been developed (*11*) to measure the penetration of water into obsidian. This permits the use of lower temperatures of hydration, because it allows the measurement of much thinner hydration rinds that can be measured optically.

Ambrose (*10*) has hydrated powdered obsidian at low temperatures (20° to 40°C) and measured the rate of uptake of water by monitoring the weight change of the sample. To convert these measurements into hydration rates, Ambrose measures the surface of the powdered obsidian by the standard gas adsorption (Brunauer-Emmett-Teller) technique. The use of the powdered obsidian has the advantage of small sample size and relative rapidity. The errors involved in the surface area determinations are 5 to 10 percent, probably no greater than the errors involved in the first technique arising from extrapolation from the relatively high temperatures of 95° to 245°C to ambient temperatures.

Table 3. Hydration rates (micrometers squared per 10^3 years) and effective temperatures (degrees Celsius) calculated from measured temperatures.

Temperature measured	Locality and obsidian type used for rate				
	West Yellowstone, Montana	Gardiner, Montana	Pine Mountain, Oregon		Gutierrez Zamora, Mexico
	Obsidian Cliff	Obsidian Cliff	Big Obsidian Flow	East Lake Flow	Obsidian Cliff
Air	6.4 (12°)		0.7 (12.8°)	3.0 (11.9°)	
Obsidian in sun	16.0 (19.7°)	22.6 (22.7°)	3.0 (24.3°)	9.9 (22.5°)	66 (32.6°)
Soil					
Depth 0.3 m			0.5 (10.8°)	2.5 (10.4°)	
Depth 0.6 m			0.5 (10.0°)	2.3 (9.8°)	
Depth 0.9 m	3.4 (6.9°)	3.6 (7.3°)			31.7 (25.8°)
Depth 1.8 m	3.1 (6.0°)	4.5 (9.0°)			28.3 (24.8°)
Mean annual air	(1.7°)	(6.6°)	(4.9°)		(25°)
Mean annual soil					
Depth 0.9 m	(6.0°)				(24.9°)
Depth 1.8 m	(5.7°)				(24.8°)

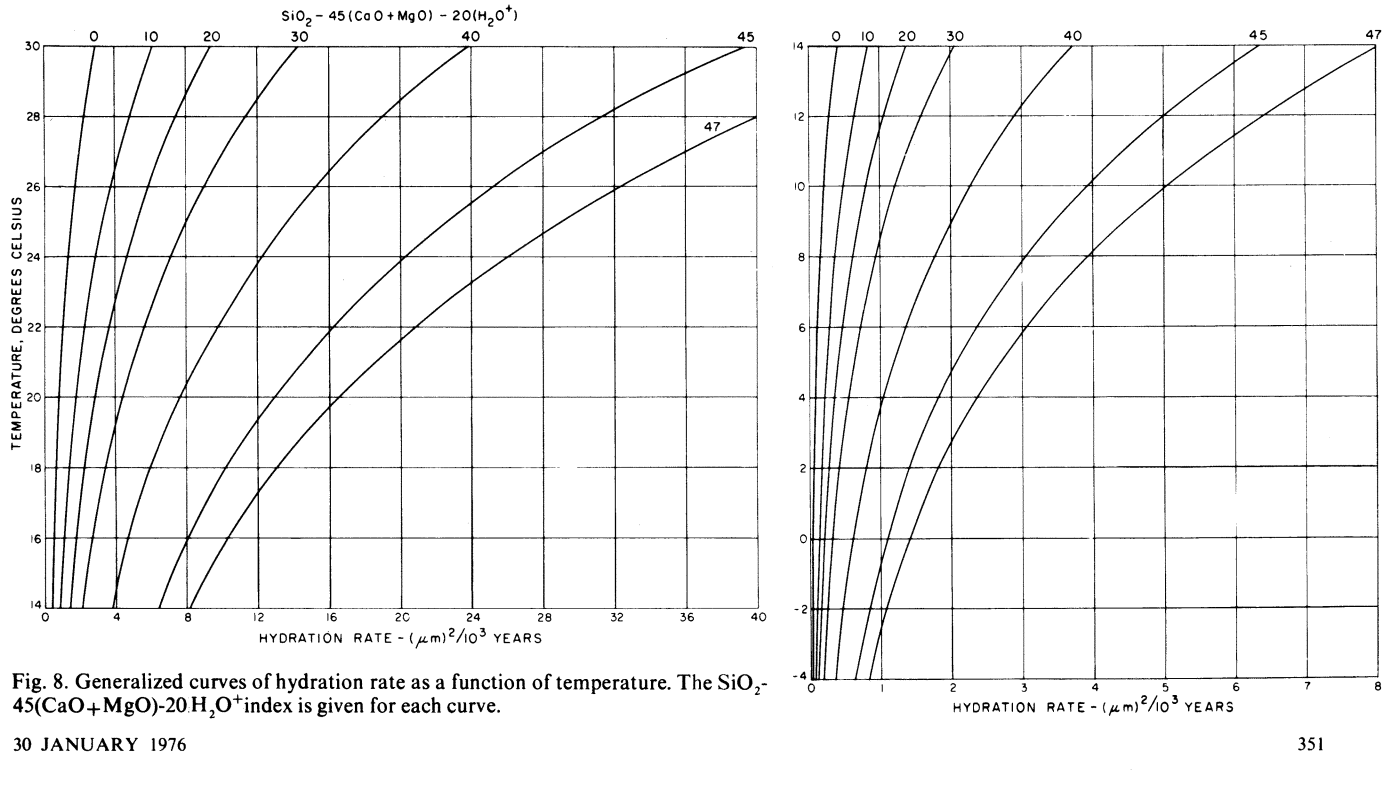


Fig. 8. Generalized curves of hydration rate as a function of temperature. The SiO_2-45($CaO+MgO$)-20H_2O^+index is given for each curve.

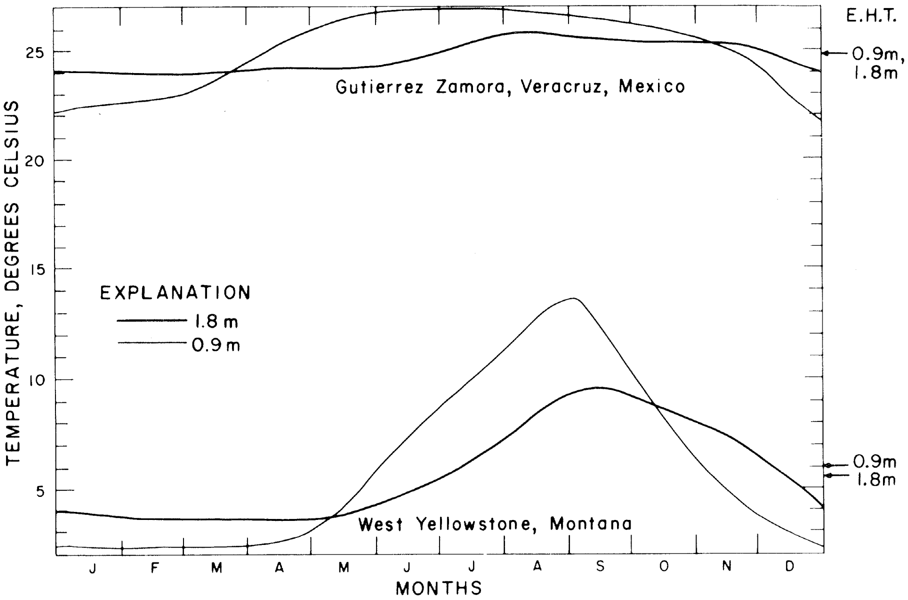


Fig. 9. Measured temperatures at 0.9 and 1.8 m in soil at a tropical site, Gutierrez Zamora, Veracruz, Mexico, and a temperate continental site, West Yellowstone, Montana. The calculated effective hydration temperature for each depth is also shown.

Suzuki (*4*) published data on the rate of hydration as a function of temperature for obsidians from Japan. He determined the hydration rate at various ^{14}C-dated sites and estimated the EHT from climate records. Suzuki discussed the effect of composition on the hydration rate. He suggested as a first approximation that all of his obsidians, containing 67.3 to 77.2 percent SiO_2, followed the same rate-temperature equation, $K = 6.76 \times 10^{13} \exp [-8927(1/T)]$ (Fig. 4).

We do not have any direct evidence on the effect of the freezing of water on the hydration rate. However, we have found that hydration of obsidian in two flows in Yellowstone Park—the Obsidian Cliff flow (179,000 years ago) and the adjacent Crystal Springs flow (69,000 years ago), both of which were covered with ice for most of their history—proceeded during these cold periods. Other rhyolite flows in the vicinity that were not covered with ice, but were undoubtedly below freezing for long periods of time, also show amounts of hydration that can be best explained by hydration if continued during the time when the obsidian was below 0°C. If our picture of the hydration process is correct—namely, rapid adsorption of water on the surface of the obsidian followed by slow diffusion of water into the obsidian—then as long as the vapor pressure of water is sufficient to saturate the surface, the hydration rate will be independent of water pressure.

If the vapor pressure of water over obsidian has any resemblance to that over silica gel, this limiting water pressure will be extremely low—much lower than that of the vapor pressure over ice at temperatures as low as -10°C. Therefore hydration should not be affected by water changing to ice and should proceed at low surficial temperatures.

Summary

The hydration rates of 12 obsidian samples of different chemical compositions were measured at temperatures from 95° to 245°C. An expression relating hydration rate to temperature was derived for each sample. The SiO_2 content and refractive index are related to the hydration rate, as are the CaO, MgO, and original water contents.

With this information it is possible to calculate the hydration rate of a sample from its silica content, refractive index, or chemical index and a knowledge of the effective temperature at which the hydration occurred. The effective hydration temperature can be either measured or approximated from weather records. Rates have been calculated by both methods, and the results show that weather records can give a good approximation to the true EHT, particularly in tropical and subtropical climates.

If one determines the EHT by any of the methods suggested, and also measures or knows the rate of hydration of the particular obsidian used, it should be possible to carry out absolute dating to ± 10 percent of the true age over periods as short as several years and as long as millions of years.

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