

Holocene ENSO-related cyclic storms recorded by magnetic minerals in speleothems of central China

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Extreme hydrologic events such as storms and floods have the potential to severely impact modern human society. However, the frequency of storms and their underlying mechanisms are limited by a paucity of suitable proxies, especially in inland areas. Here we present a record of speleothem magnetic minerals to reconstruct paleoprecipitation, including storms, in the eastern Asian monsoon area over the last 8.6 ky. The geophysical parameter $IRM_{soft-flux}$ represents the flux of soil-derived magnetic minerals preserved in stalagmite HS4, which we correlate with rainfall amount and intensity. $IRM_{soft-flux}$ exhibits relatively higher values before 6.7 ky and after 3.4 ky and lower values in the intervening period, consistent with regional hydrological changes observed in independent records. Abrupt enhancements in the flux of pedogenic magnetite in the stalagmite agree well with the timing of known regional paleofloods and with equatorial El Niño–Southern Oscillation (ENSO) patterns, documenting the occurrence of ENSO-related storms in the Holocene. Spectral power analyses reveal that the storms occur on a significant 500-y cycle, coincident with periodic solar activity and ENSO variance, showing that reinforced (subdued) storms in central China correspond to reduced (increased) solar activity and amplified (damped) ENSO. Thus, the magnetic minerals in speleothem HS4 preserve a record of the cyclic storms controlled by the coupled atmosphere–oceanic circulation driven by solar activity.

storms | paleoprecipitation | speleothems | environmental magnetism | paleoclimate

Carbonate cave deposits are attractive archives for reconstructing changes in past climate because they can provide high-resolution and, in most cases, continuous records. Measurements of oxygen and carbon stable isotopes in speleothems are used routinely to recover information about environmental changes, including monsoon intensity in monsoon-impacted regions (1), although they are highly debated (2–6). Monsoon-driven storms are a common example of extreme precipitation events that can cause widespread flooding and create hazardous conditions for communities and their infrastructure. Identification of storms and elevated precipitation in prehistoric times is thus critical for understanding regional hydrological changes and testing the potential mechanisms that may influence them. Despite their high temporal resolution, speleothems remain an underused tool for assessing regional storm and flood frequency throughout the Holocene, and may offer unique insights into the long-term processes that drive changes in the frequency of such events.

Magnetic minerals, transported by groundwater from soils overlying the cave system, are incorporated into speleothems as they grow, and long-term changes in precipitation can be recorded by magnetic minerals in speleothems (7–11). Recent advances in measurement sensitivity have opened up speleothems as viable archives of magnetic information (7, 11). Here, we examine whether such magnetic records can provide an opportunity to identify extreme precipitation events, such as storms, by measurement of magnetic minerals in a stalagmite from central China,

a region strongly influenced by both the Eastern Asian and Indian monsoon systems. One-hundred-and-fifteen cubic specimens ($2 \times 2 \times 2$ cm) were collected from a 2.5-m-tall stalagmite (HS4) from Heshang Cave ($30^{\circ}27'N$, $110^{\circ}25'E$), central China, for magnetic measurements (Fig. 1; sampling methods are well documented in ref. 10). U–Th dating, combined with layer counting, indicated that the stalagmite grew continuously over the last 9.0 ky (12).

Soil-Derived Magnetic Minerals in Stalagmite HS4 and Their Hydrological Implication

Magnetite, goethite, and hematite/maghemite were identified in our samples. Coercivity unmixing analyses were conducted on specimens from stalagmite HS4 by alternating field (AF) demagnetization of an isothermal remanent magnetization ($IRM_{1.15T}$) induced by a 1.15-T direct current (dc) field (Methods). For each sample, $\sim 90\%$ of the $IRM_{1.15T}$ is carried by a mineral population with an ~ 20 -mT median destructive field (MDF) and a dispersion parameter (DP) of ~ 0.4 (Fig. 2). A small proportion ($<4\%$) of the total remanence is carried by a lower coercivity, or magnetically very “soft,” component with an MDF < 5 mT (Fig. 2), which is likely to be coarse, multidomain magnetic particles. Magnetic particles were extracted from stalagmite HS4 for more detailed mineralogical analyses, including low-temperature magnetic measurements and electronic microscopy (Methods). The presence of magnetite in HS4 was confirmed in all of the magnetic extracts by observation of an abrupt decrease in magnetization at ~ 120 K, identified as the Verwey transition, during low-temperature cycling of thermal remanent magnetization (TRM) (Fig. S1). Low-temperature magnetometry also provided evidence for the

Significance

High-resolution reconstructions of storm history and storms' underlying mechanisms in inland areas are critical but limited by a paucity of suitable paleoproxies. Here we use soil-derived magnetic minerals preserved in a stalagmite as a new paleo-hydrological proxy. This proxy enables us to rebuild decadal resolution storm records in the eastern Asian monsoon area since 8.6 ky. Variance of storms in central China was found to exhibit close correlation with El Niño–Southern Oscillation activity at millennial and centennial time scales, and also occur on a significant 500-y cycle related to periodic solar activity. These findings shed light on the forecasting of future floods and improve our understanding of the potential mechanism of strong precipitation in monsoon regions.

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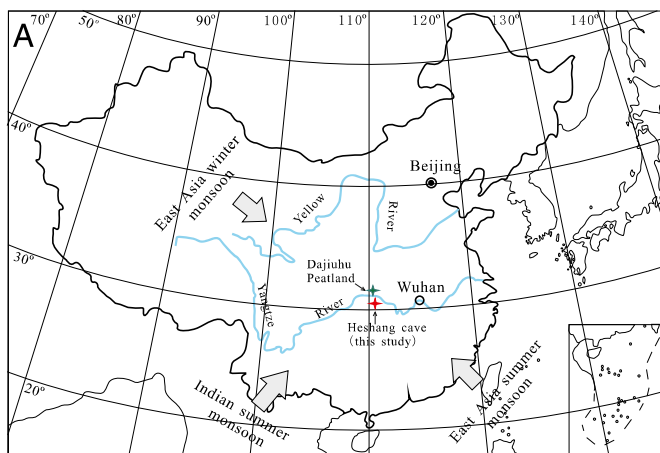


Fig. 1. Location of Heshang cave and the picture of stalagmite HS4. (A) The location of Heshang Cave (red star) and (B) the original figure (right) and the cross section (left) of stalagmite HS4 amended based on ref. 12. Red dashed rectangle denotes the location where the cubic specimens were cut.

presence of high-coercivity goethite and hematite/maghemite in the Heshang specimens (Fig. S1).

Soft magnetic components in our samples were demonstrated to be of soil origin, on the basis of their mineralogy, magnetic coercivity distribution, and morphological characteristics. The coercivity distribution of the dominant soft magnetic component ($MDF \approx 20$ mT and $DP \approx 0.4$) is characteristic of pedogenic magnetite (13, 14), which is commonly transported into caves via drip water and preserved in stalagmites (9, 11) (Fig. 2). The pedogenic magnetite and very soft magnetic component that together dominate the HS4 stalagmite samples were also identified in the soil samples collected immediately above the cave, contributing more than 80% and less than 1.5% of the total $IRM_{1.15T}$, respectively. The ratio of the remanence held by the very soft component to that held by pedogenic magnetite is the same in both stalagmite HS4 and the soil samples (~ 0.2), supporting a soil origin for the soft magnetic minerals in HS4. In contrast, the remanence properties of the carbonate rock that hosts Heshang cave were dramatically different. Less than 20% of

the $IRM_{1.15T}$ of the host rock was demagnetized after a 100-mT AF demagnetization. The bedrock is therefore dominated by higher coercivity, magnetically “hard” magnetic minerals with $MDF > 100$ mT, such as goethite and/or hematite, and is unlikely to be a primary source of soft magnetic minerals to the speleothems in Heshang cave. Furthermore, most of the extracted magnetite grains show evidence of transport and weathering processes, including partially rounded shapes, and etch pits, with an absence of euhedral magnetic grains (Fig. S2), indicating that the magnetite preserved in stalagmite HS4 is likely allochthonous rather than precipitating in situ (9, 15, 16). In addition, based on the past 9 y of in situ monitoring, the pH value of the drip water is always lower than 8.5, and the air temperature of the cave is constantly higher than 15 °C, conditions that are unfavorable for the formation of authigenic magnetites (ref. 16 and references therein). Together, these lines of evidence strongly suggest that the soft magnetic components (mainly magnetite) preserved in stalagmite HS4 are soil-derived.

Increased rainfall can enhance the production of pedogenic magnetite in soil (17), and it promotes the transportation of soil magnetite into the cave via drip water. Discrete intervals with enhanced concentrations of soil-derived particles, including magnetite, in speleothems can be associated with episodic enhanced rainfall (18). We therefore interpret variations in the concentration of soil-derived magnetite identified in the HS4 stalagmite to be indicative of rainfall variability. The amount of soil-derived, soft magnetic minerals (i.e., magnetite and its partially oxidized equivalents) in HS4 can be assessed by measuring $|IRM_{soft}|$ [defined as $0.5 \times (|IRM_{1T}| + |IRM_{-0.3T}|)$; Methods]. This measurement excludes the portion of IRM_{1T} contributed by hard magnetic minerals that originated from the surrounding rocks and authigenic goethite, because hard magnetic minerals such as hematite and goethite are unlikely to be remagnetized by a 300-mT dc field (8). We use the parameter $IRM_{soft-flux}$, whereby $|IRM_{soft}|$ values are normalized by the growth duration of each sample, to trace the variation in the flux of the soft, soil-derived magnetite particles per year.

Although $IRM_{soft-flux}$ can be a proxy for long-term paleo-precipitation, where higher values are associated with increased

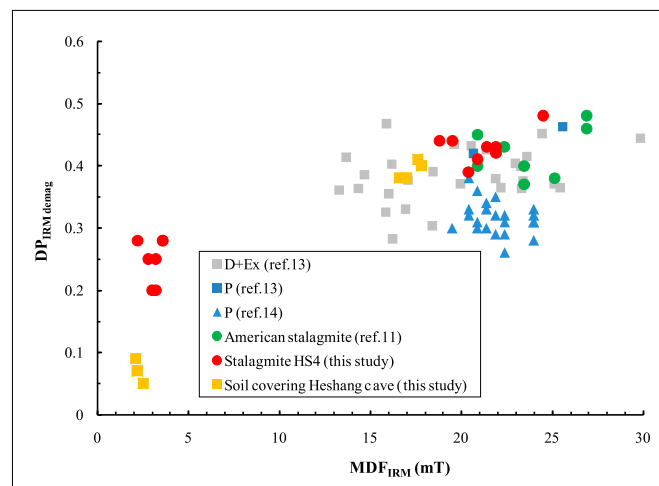


Fig. 2. Magnetic components in soil and stalagmites. Shown are D+Ex, detrital magnetite transported by flowing water and organically formed extracellular, ultrafine magnetite (13) (gray squares); P, pedogenic magnetite observed in the United States (13) and China and Switzerland (13, 14) (blue squares and triangles); the “soft” component from stalagmite BCC-010 (11) (green circles); and the “soft” component from stalagmite HS4 (red circles) and soil capping Heshang Cave (orange squares) (this study). Horizontal and vertical axes represent the MDF and DP, respectively.

rainfall and wetter intervals, it may also record discrete high-intensity precipitation events. In contrast to pedogenic processes that slowly increase the amount of pedogenic ferrimagnetic minerals within soils, large storms can dramatically increase the energy of groundwater in karst regions within a very short time, causing an abrupt increase in the transportation of heavy minerals to the cave system and, in turn, an abrupt increase in the amount of allochthonous particles preserved in stalagmites. We therefore relate abrupt enhancements in $IRM_{\text{soft-flux}}$ to increases in magnetite flux resulting from an increased frequency of extreme precipitation events such as storms.

Long-Term Hydrological Variation and Storms

The mean $IRM_{\text{soft-flux}}$ is relatively low ($5.3 \times 10^{-10} \text{ Am}^2 \cdot \text{y}^{-1}$) between 6.7 ky and 3.4 ky, compared with higher values ($14.9 \times 10^{-10} \text{ Am}^2 \cdot \text{y}^{-1}$) before 6.7 ky and after 3.4 ky (Fig. 3A). This three-interval pattern is in good agreement with local water levels recorded by the accumulation rate of aerobic hopanoids in Dajiuhe peatland, a site about 120 km north of Heshang cave (7) (Fig. 3C). The two wet periods (before 6.7 ky and after 3.4 ky) also agree well with the highest lake water level in the middle and lower reaches of Yangtze River, which occurred between 8.0 ky

and 7.0 ky and after 3.0 ky (19). The carbon isotope composition of the soil-derived acid-soluble organic matter ($\delta^{13}\text{C}_{\text{ASOM}}$) in the HS4 stalagmite (Fig. 3B and ref. 20) also records a pattern comparable to $IRM_{\text{soft-flux}}$ on a millennial scale, with less negative $\delta^{13}\text{C}_{\text{ASOM}}$ values during the two wet periods. These consistencies between the three different records suggest that $IRM_{\text{soft-flux}}$ is a reliable and accurate indicator of regional hydrological changes in central China throughout the Holocene.

A detailed comparison of the magnetic signals and oxygen compositions of carbonate from the HS4 stalagmite is shown in Fig. S3 and does not show any significant correlation. It is well known that $\delta^{18}\text{O}$ in cave sediments is controlled by a variety of factors, including rainfall amount, cave temperature, local evaporation, the $\delta^{18}\text{O}$ of the sources (the Indian Ocean or/and the West Pacific Ocean), and the transport distance from the sources (21, 22). Models and modern observations have shown that variations in vapor sources rather than in the precipitation amount (1) dominated the speleothems $\delta^{18}\text{O}$ records in the East Asian monsoon (EAM) area (2–6). Consequently, converting stalagmite $\delta^{18}\text{O}$ records into a quantitative assessment of past rainfall amount, including that arising from typhoon sources, is very difficult in the EAM region (12), and it is therefore unsurprising that

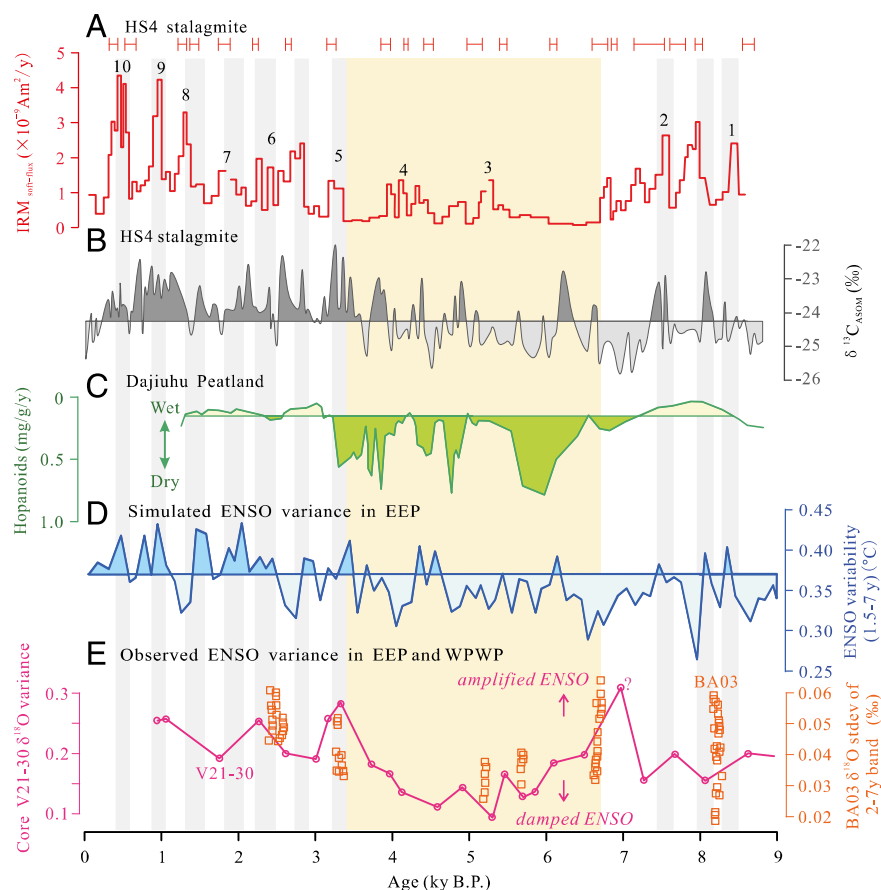


Fig. 3. Hydrological conditions in central China and ENSO strength. (A) $IRM_{\text{soft-flux}}$ in stalagmite HS4. Peaks in $IRM_{\text{soft-flux}}$ indicate intervals with increased extreme precipitation events (this study), numbers 1 through 10 indicate the flooding events reported near the research area (23–28). U–Th dating errors (12) are shown on the top of $IRM_{\text{soft-flux}}$ curve as red line segments. (B) The carbon isotope composition of the acid-soluble soil-derived organic matter ($\delta^{13}\text{C}_{\text{ASOM}}$) of HS4 stalagmite. Although affected by multiple factors, more (less) negative $\delta^{13}\text{C}_{\text{ASOM}}$ was correlated with dry (wet) climate (20). (C) Hopanoid accumulation rate in Dajiuhe peatland. High (low) accumulation rates correlate with dry (wet) intervals (7). (D) Simulated ENSO amplitude in 100-y window based on observation data [shown as SD of Nino3.4 (a region bounded by 5°N to 5°S , from 170°W to 120°W) interannual (1.5 y to 7 y) sea surface temperature variability] (39). (E) Observed ENSO variability from stalagmite BA03 (open squares) (33) and foraminiferal $\delta^{18}\text{O}$ (open circles) (34). BA03 $\delta^{18}\text{O}$ values are calculated based on the SD of the 2- to 7-y band in overlapping 30-y windows and indicate the ENSO variance in WPWP region. Foraminiferal $\delta^{18}\text{O}$ is retrieved from deep-sea sediments in core V21-30 located at EEP region (cold tongue of ENSO activity), and is established on single tests in each 1-cm stratum with an age uncertainty of several hundred years (34). The question mark indicates a questionable $\delta^{18}\text{O}$ value at 7.0 ky (mentioned in ref. 34). Comparison of peaks in $IRM_{\text{soft-flux}}$ and ENSO variance are indicated by gray bars. Vertical yellow bar indicates the regional dry period (6.7–3.4 ky).

there is no significant correlation between the $\delta^{18}\text{O}$ records and our magnetic signal.

The $\text{IRM}_{\text{soft-flux}}$ record shows pulses of abrupt, centennial-scale enhancement throughout the HS4 stalagmite. These abrupt enhancements are particularly strong during the two wet intervals (after 3.4 ky and before 6.7 ky), and are subdued during the dry interval between them. We interpret these pulses of enhanced $\text{IRM}_{\text{soft-flux}}$ to represent intervals of increased storm frequency/strength. For comparison, we compiled previously published histories of flood deposits preserved in the middle reaches of Yangtze River, and 10 paleoflood events could be recognized during the past 9.0 ky, occurring at 9,000–8,400 y B.P. (23, 24), 7500–7200 y B.P. (25), 5500–5000 y B.P. (26), 4200–4000 y B.P. (23, 27), 3200–2800 y B.P. (23–25), 2600–2200 y B.P. (28), 1900–1700 y B.P. (23, 27), 1200 y B.P. (28), 1000–800 y B.P. (26, 28), and 590 ± 50 y B.P. (28), respectively. These paleoflood events are identified in peaks in $\text{IRM}_{\text{soft-flux}}$ in Fig. 3*A* and indicate periods of elevated precipitation (23, 25, 28). Extreme paleofloods became more frequent after 2.2 ky and reached a maximum frequency between 1.0 ky and the present day (29), coincident with the two highest pulses of the $\text{IRM}_{\text{soft-flux}}$ value of HS4 stalagmite. In contrast, the pronounced aridity event induced by the abrupt cooling event in the North Atlantic region at 8.2 ky (30) is coincident with the weak $\text{IRM}_{\text{soft-flux}}$ value of HS4 stalagmite. The $\delta^{13}\text{C}_{\text{ASOM}}$ exhibits a similar variance to $\text{IRM}_{\text{soft-flux}}$ on a millennial scale, but not on a centennial scale; this may be due to the effects of temperature and vegetation on $\delta^{13}\text{C}_{\text{ASOM}}$ in addition to precipitation (20).

Close Correlation Between El Niño–Southern Oscillation and Storms in Central China

The occurrence of modern-day storms in the middle reaches of Yangtze River is related to the strength of El Niño–Southern Oscillation (ENSO) (31), especially the El Niño events (32). Although paleo-ENSO records are difficult to reconstruct, particularly in the early Holocene (33–35), our $\text{IRM}_{\text{soft-flux}}$ record appears to be consistent with paleo-ENSO proxies available for the Holocene. Geological data and climate models document a mid-Holocene reduction in ENSO intensity and fewer El Niño-related flood events (33, 34, 36–38) (Fig. 3*D* and *E*). This finding is consistent with lower $\text{IRM}_{\text{soft-flux}}$ values, which indicates fewer storms between 6.7 ky and 3.4 ky. Although the Holocene ENSO might show different spatial patterns (36), $\delta^{18}\text{O}$ variances of foraminifera (34) (open circles in Fig. 3*E*) and stalagmite BA03 (33) (open squares in Fig. 3*E*) support strong ENSO activity in both the eastern equatorial Pacific (EEP) and western Pacific warm pool (WPWP) regions during the early and later Holocene, which is broadly consistent with the frequent storms before 6.7 ky and after 3.4 ky indicated by the $\text{IRM}_{\text{soft-flux}}$ record. Of particular importance is that, during the relatively strong ENSO periods (the early and later Holocene), $\text{IRM}_{\text{soft-flux}}$ shows an increased frequency of enhancement pulses, and the peaks of $\text{IRM}_{\text{soft-flux}}$ during those periods broadly correspond to the peaks of the observed and modeled ENSO variances in the EEP region (33, 39) (Fig. 3*D* and *E*). This would suggest that stronger ENSO can increase the frequency of extreme precipitation events, such as storms, in central China.

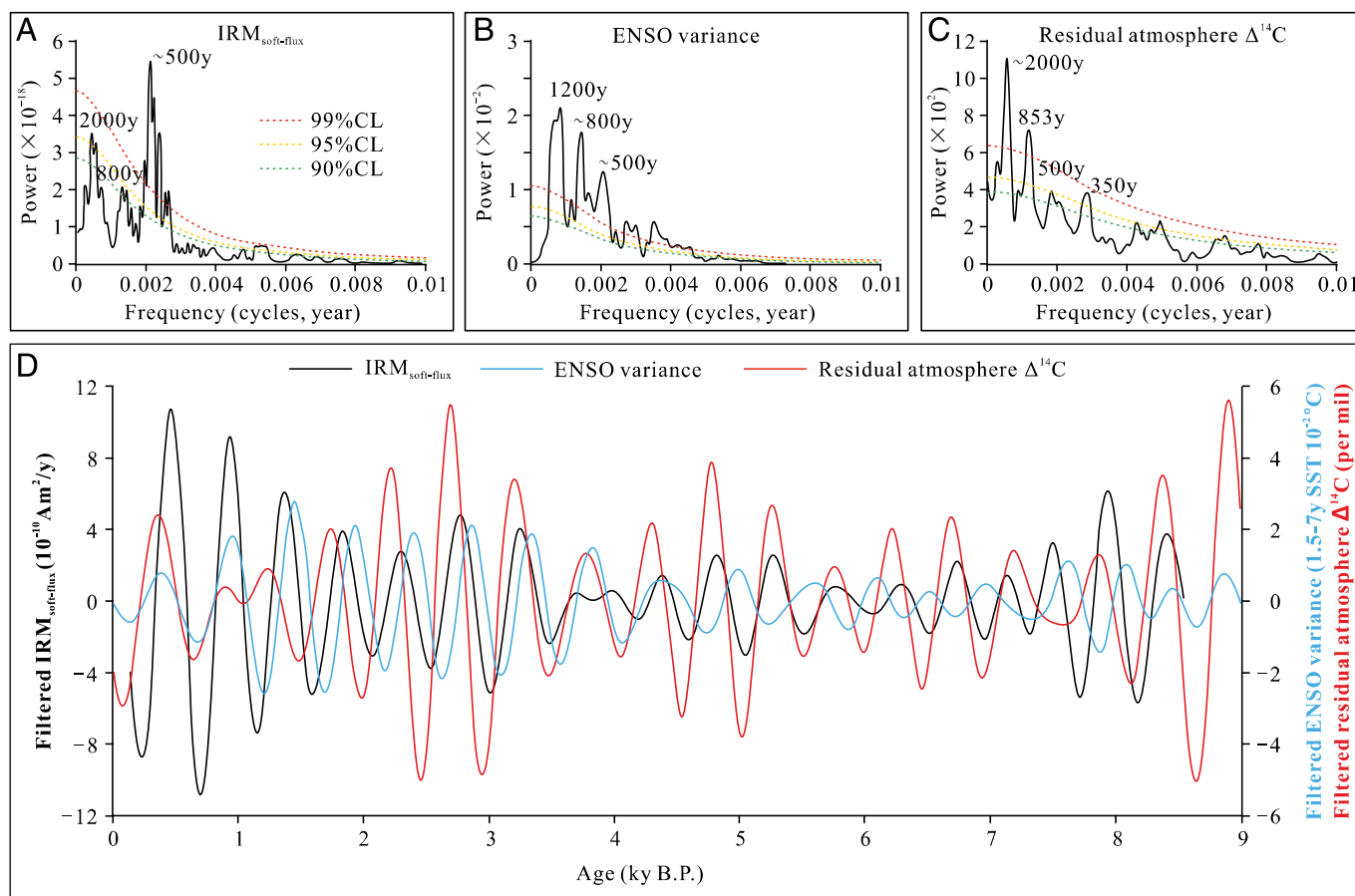


Fig. 4. Power analysis of $\text{IRM}_{\text{soft-flux}}$ of HS4, ENSO variance, and solar irradiance parameter [residual atmospheric $\Delta^{14}\text{C}$ from global tree ring records (46)]. (A–C) Power spectra of (A) $\text{IRM}_{\text{soft-flux}}$ (this study), (B) ENSO variance (39), and (C) residual atmosphere $\Delta^{14}\text{C}$ (46). Red, orange, and green dashed lines are 99%, 95%, and 90% confidence level (CL) respectively. (D) $\text{IRM}_{\text{soft-flux}}$ (black), ENSO variance (blue), and residual atmosphere $\Delta^{14}\text{C}$ (red) after 500-y band-pass filtering. Higher $\Delta^{14}\text{C}$ values correspond to periods of lower solar activity.

Some discrepancies do exist between the two ENSO records (simulated and observed) and the storms inferred by our magnetic record, which might arise from limitations inherent within the simulated and observed ENSO records. The simulated ENSO variability is based on modern observations, which means that the simulation becomes less reliable with increasing age. It might provide a good comparison for variations in frequency, but modeled changes in the amplitude become less reliable with increasing age. This tendency explains why our magnetic record shows similar cyclic variation to the simulated ENSO record (39), but shows inconsistent amplitude variation, particularly in the early Holocene (Fig. 3*D*). Further, the previously published data for the observed ENSO record display a comparatively low temporal resolution and a large uncertainty in age (33, 34). The observed ENSO record cannot provide a detailed comparison, but it does show the same three-interval features observed in the magnetic data (i.e., elevated during 9–6.7 ky, and 3.4–0 ky, but decreased during 6.7–3.4 ky) (Fig. 3*E*). Finally, the compilation of flooding events (23–29) in central China confirms the robustness of our magnetic profile as an ENSO record within the age uncertainties denoted in Fig. 3.

Five-Hundred-Year Periodicity of Storms and Its Forcing

Spectral analysis of the detrended $IRM_{\text{soft-flux}}$ record and modeled ENSO variance data (from ref. 39) reveal that they both exhibit a centennial cycle centered at ~500 y with a confidence level of greater than 99% (Fig. 4*A* and *B*). The 500-y cycle is a significant component of solar activity periodic variations (40, 41) (Fig. 4*C*), which can control Earth surface temperature variability and alter atmospheric and oceanic circulation (42–45). The 500-y cycle of storms in central China is generally in an antiphase relationship with solar activity, indicated by the residual atmospheric $\Delta^{14}C$ from global tree ring records (46) during the past 8.6 ky, where higher $IRM_{\text{soft-flux}}$ values (representing storms) are associated with low solar irradiation (larger $\Delta^{14}C$). The correlation between 500-y periodic storms and the ENSO variance is positive in the wet periods (the early and later Holocene), but it changes to a negative correlation in the dry period (the mid-Holocene) when the ENSO is damped and in a more La Niña-like state according to the observed data (33, 34).

Our results show that strong/frequent storms in central China are broadly consistent with weak solar irradiation as well as strong ENSO activities. Solar activity can modulate the EAM system and thereby affect precipitation in central China. Decreased solar irradiance can weaken the Asian summer monsoon (41, 43), which, in turn, could cause the convergence zone, the Mei-yu Front, where warm–moist, tropical–subtropical air meets the cooler continental air mass, to move southward toward the Yangtze River valley (47) and hover around this area. This southward movement could move preexisting storm centers closer to Heshang cave or, alternatively, increase the frequency of storms regionally. Although there are multiple controls on ENSO (39), its long-term variability is closely related to secular solar activity variations, and both the intensity and frequency of El Niño events, which result in flooding in east and central China (32), are high at secular solar minimum and low at secular solar maximum (48). Thus, solar radiation, ENSO activity, and coupled atmospheric–oceanic variation may therefore control the occurrence of ENSO-related storms in central China. The delayed response of storms in central China to the mature ENSO (48), and/or the different age modes of those two data series, could probably result in the small phase shift between ENSO variations and storms shown in Fig. 4*D*. Reduced ENSO activity during the dry mid-Holocene was not strong enough to affect the storms in central China, which might explain their weak relationship during this interval.

Methods

Sampling. One-hundred-and-fifteen environmental magnetism specimens (2-cm cubes) were cut sequentially from the core of the stalagmite where

growth layers accumulated in a nearly horizontal orientation (10) (Fig. S1). Four soil samples were collected from the B horizon of soil capping Heshang cave. Eight representative HS4 samples distributed across the whole time series were chosen for more advanced magnetic mineralogy analyses.

Extraction of Magnetic Minerals from Stalagmite HS4. To obtain unambiguous magnetic measurements and conduct morphological analyses on the magnetic particles, we dissolved fragments of stalagmite HS4 and extracted the magnetic minerals. The acetate buffer solution [4:1 (vol/vol) of 2 M acetic acid and 1 M sodium acetate] recommended by Perkins (16) was used for stalagmite dissolution. The extracting procedure followed that of Strehlau et al. (49).

$IRM_{\text{soft-flux}}$. All specimens were given an IRM using a 1-T dc field, and their remanences were signed as IRM_{1T} ; subsequently, the specimens were subjected to a 0.3-T dc field in the opposite direction (“backfield”) to produce another remanence signed as $IRM_{-0.3T}$. Then $|IRM_{\text{soft}}|$ is defined as $0.5 \times (|IRM_{1T}| + |IRM_{-0.3T}|)$. To eliminate the effect of variation on the stalagmite’s growth rate, $|IRM_{\text{soft}}|$ measurements were normalized using the age duration of each sample to give $IRM_{\text{soft-flux}}$ (Table S1), which represents the amount of soil-originated magnetic minerals preserved in stalagmite HS4 per year. Remanent magnetizations were imparted using a 2G Enterprises 670M long-core pulse magnetizer and were measured using a 2G U-channel cryogenic SQUID (superconducting quantum interference device) magnetometer.

Magnetic Mineralogy Analyses. IRMs were imparted to eight HS4 samples and four soil samples using a 1.15-T dc field and then progressively degaussed in AFs up to a maximum of 170 mT. The first derivatives of the demagnetization data were used to perform coercivity unmixing analyses to identify magnetic mineral components on the basis of their MDF and DP, calculated following the methods described in ref. 50. The contribution of the very soft component is represented by the portion of the IRM demagnetized following application of a 5-mT AF, and that of the pedogenic magnetite is indicated by the component of the IRM demagnetized between the application of a 20-mT and 5-mT AF. The ratio of the two soft components’ relative contributions was then calculated, and is 0.2 for almost all of the stalagmites samples and for all of the soil samples.

The low-temperature magnetic behavior of the samples extracted was examined using a Quantum Designs Magnetic Properties Measurement System (MPMS-55). We used the goethite-pretreatment low-temperature measure sequence designed by Guyodo et al. (51) to target weak magnetic minerals such as goethite and hematite. Detailed protocols are provided in Fig. S1.

Electron Microscopy Analyses. The morphology of the extracted magnetic particles was observed using a scanning electron microscope (SEM) and a transmission electron microscope (TEM). The extracted magnetic particles were deposited onto carbon-coated adhesive tape, and then analyzed with a JSM-35CF SEM (Japanese Electronics Co., Ltd.). The elemental composition of magnetic minerals was measured using energy dispersive spectroscopy (EDS). For TEM analysis, magnetic particles were suspended in a solution of collo-dion and isoamyl acetate [1:4 (vol/vol)] via 10 min of ultrasonic vibrating, and then two to three drops of the suspension were dropped into a bowl of pure water. A thin film then formed on the surface of the water, and a portion of this film was then applied to a TEM support grid, dried, and carbon-coated for TEM analyses using a Philips CM12/S TEM. Further elemental analysis was provided by Philips PV9760 Energy Dispersive X-ray Analyzer attached to the Philips CM12/S TEM. Photomicrographs collected from the SEM and TEM were obtained at operating voltages of 1.5 and 1.2 kv, respectively.

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