

Holocene wet episodes recorded by magnetic minerals in stalagmites from Soreq Cave, Israel

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ABSTRACT

This study demonstrates the feasibility of speleothem magnetism as a paleo-hydrology proxy in speleothems growing in semi-arid conditions. Soil-derived magnetic particles in speleothems retain valuable information on the physicochemical conditions of the overlying soil, and changes in bedrock hydrology. Yet, the link between magnetic and isotopic proxies of speleothems has been only partly established. We reveal strong coupling between the in-flow of magnetic particles (quantified using the magnetic flux index, IRM_{flux}) and $\delta^{13}C$ in two Holocene speleothems from Soreq Cave (Israel). The stalagmite record spans from ca. 9.7 to ca. 5.4 ka, capturing the warm-humid conditions associated with the early Holocene and the transition to mid-Holocene wet-dry cycles. Extremely low IRM_{flux} during the early Holocene, indicating minimal contribution from the overlying soil, is accompanied by anomalously high $\delta^{13}C$ (approaching bedrock values) hypothesized to be caused by high rainfall and soil erosion. By contrast, IRM_{flux} during the mid-Holocene covaries with the saw-tooth cyclicity of $\delta^{13}C$ and $\delta^{18}O$, interpreted as rapid fluctuations in rainfall amount. The peaks in IRM_{flux} precede the negative (wet) $\delta^{13}C$ peaks by ~60–120 yr. The apparent lag is explained as a rapid physical translocation of overlying soil particles via groundwater (high IRM_{flux}) as a response to increasing rainfall, compared with slower soil organic matter turnover rates (10–10² yr).

INTRODUCTION

Speleothem paleoenvironmental proxy records focus on stable oxygen and carbon isotope time series ($\delta^{18}O$ and $\delta^{13}C$, respectively) (e.g., Fairchild and Baker, 2012; Bar-Matthews et al., 2019). Coupling these data with other climate proxies increases the accuracy and precision of paleoclimate interpretations (Fairchild and Baker, 2012). These other proxies include nontraditional isotopes, trace element geochemistry, and, more recently, mineral magnetism (e.g., Bourne et al., 2015; Zhu et al., 2017). The underlying assumption is that speleothem magnetic properties are controlled by ferromagnetic particles flushed into the cave and encapsulated within calcite laminae (Lascu and Feinberg, 2011). These particles can be detrital in origin (dominated by regionally ubiquitous magnetic minerals such as magnetite, titanomagnetite, and hematite), or authigenic (mainly pedogenic magnetite and goethite) (Lascu and Feinberg, 2011; Bourne et al.,

2015; Jaqueto et al., 2016). The concentration, mineralogy, and size of the magnetic particles can be measured indirectly using non-destructive methods, providing a proxy for the physicochemical properties of the overlying soil and the cave hydrology (Lascu and Feinberg, 2011; Feinberg et al., 2020). However, the interpretation of the magnetic data is not straightforward because the link between climate and magnetic parameters is controlled by the local geohydrology. For example, data from mid-latitude stalagmites suggest that more magnetic particles are delivered into a cave as rainfall increases (e.g., Bourne et al., 2015; Zhu et al., 2017), while the opposite relationship is observed in tropical caves, where magnetic particles are delivered primarily during dry periods (Jaqueto et al., 2016; Fu et al., 2021). Thus, adaptation of speleothem magnetism in paleoclimate models requires a site-specific multiproxy analysis that includes both magnetic and geochemical proxies.

Our study examines the coupling between magnetic parameters and $\delta^{13}C$ values over centennial to millennial timescales in two Holocene stalagmites from Soreq Cave, Israel (31°45'21"N, 35°01'20"E). The stalagmite record spans the time interval from ca. 9.7 ka to ca. 5.4 ka, with an overlap between ca. 7.0 and ca. 6.4 ka covering the early-Holocene pluvial conditions associated with the deposition of Sapporopel layer 1 (S1) in the eastern Mediterranean Sea (Bar-Matthews et al., 2000), followed by the transition to mid-Holocene wet-dry cycles (Bar-Matthews and Ayalon, 2011). We demonstrate the interplay between magnetic and isotopic proxies during regional climate change derived from speleothems deposited under two drip-sites with different hydrochemical settings. This is part of an effort to further develop speleothem magnetism as a standardized paleoenvironmental proxy.

THE SOREQ CAVE

Soreq Cave (Fig. 1A) is located on the west-ern slopes of the Judean Mountains, 40 km east of the Mediterranean Sea at an elevation of ~400 m above mean sea level. The cave area is ~5000 m², hosted within Cenomanian dolomitic limestone. Bedrock above the cave varies between 10 m to >50 m. The overlying soil occurs as 30–100-cm-thick pockets covering ~50% of the present-day surface (Figure S1 and Section S1 in the Supplemental Material¹). Flora above the cave are intermingled coniferous forest and C₃ shrubland (Danin, 1992). Mean high annual rainfall is 500¹⁰³⁴₁₉₀ mm with >95% of rain limited to October through May (Ayalon et al., 1998).

Soreq Cave is situated on the boundary between the Mediterranean semi-arid climate zone

¹Supplemental Material. Extended site description, methods, age model, and results, including data tables. Please visit <https://doi.org/10.1130/G49383.1> to access the supplemental material, and contact editing@geosociety.org with any questions.

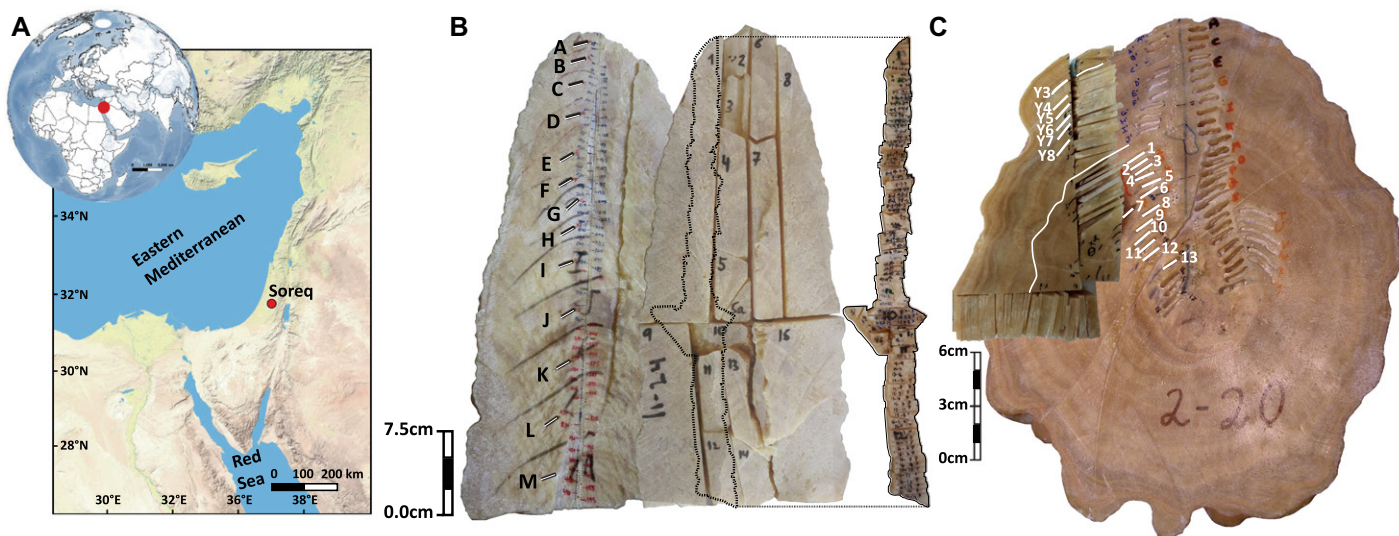


Figure 1. (A) The eastern Mediterranean, with the location of Soreq Cave, Israel ($31^{\circ}45'21''\text{N}$, $35^{\circ}01'20''\text{E}$, WGS84). (B,C) Stalagmites 11-24 and 2-20 superimposed with the slices used for magnetic analysis. Black lines (on 11-24) and white lines (on 2-20) mark the locations of U-Th sampling (denoted by letters and numbers). Small holes (numbered from top to bottom, in intervals of five) mark the stable isotope measurements.

to the north and the arid desert environment to the south, thus acting as a sensitive recorder of changes in hydroclimate and seasonality (Bar-Matthews et al., 2019). The composite isotope record from Soreq secondary calcite speleothems spans ~ 180 ka and is the quintessential proxy time series for the eastern Mediterranean (Bar-Matthews et al., 2019, and references therein; Section S1 in the Supplemental Material). In addition, Soreq data bracket the timing of the terrestrial occurrence of sapropel S1 (Almogi-Labin et al., 2009) and reveal mid-Holocene wet-dry cycles simultaneous with cultural transitions in the Levant (Bar-Matthews and Ayalon, 2011).

METHODS

Stalagmite 11-24 (from zone 11 in the cave) is a 425-mm-long stalagmite cut along its growth axis. Bar-Matthews and Ayalon (2011) studied one half of the stalagmite for reconstructing paleoclimate, and the second half is used here for paleomagnetic analysis (Fig. 1B; Fig. S2). Stalagmite 2-20 (from zone 2) was cut perpendicular to its growth axis near its base. The ~ 122 -mm-long semi-major axis of the horizontal plane was used for paleoclimate reconstruction (Bar-Matthews et al., 2003; Grant et al., 2012) as well as for paleomagnetic analysis (Fig. 1C, Figs. S2 and S3). The isotopic time series in our study was constructed from 502 drilled samples in stalagmite 11-24 and 162 drilled samples in stalagmite 2-20. The age-depth models for the two stalagmites were recalculated with the StalAge R code (Scholz and Hoffmann, 2011; code is available in Comas-Bru et al. [2020]) using 14 of the published U-Th ages from stalagmite 11-24 (Bar-Matthews and Ayalon, 2011) and 16 U-Th ages from stalagmite 2-20 (12 published ages [Grant et al., 2012] and

4 new ages) (Fig. S3; see Section S2 for the age-depth model).

We performed paleomagnetic experiments in the paleomagnetic laboratory at the Institute of Earth Sciences, The Hebrew University of Jerusalem, using a 2G-RAPID cryogenic sample rock magnetometer equipped with in-line AF demagnetization coils and an ASC pulse magnetizer. The samples are thin slices, 2–5 mm thick with a median thickness of 4.2 mm. Stalagmite 11-24 was cut into 82 slices, and stalagmite 2-20 was cut into 44 slices (Figs. 1B and 1C). The samples were washed with distilled water to remove possible contamination from the saw, and a 1.5 T (tesla) DC field was applied to all specimens to amplify the samples' weak natural remanent magnetization (NRM) resulting from a low concentration of magnetic minerals. The isothermal remanent magnetization (IRM) was normalized both to mass (IRM_{mass}) and time (IRM_{flux}) (Zhu et al., 2017). IRM_{mass} is a measure of the relative concentration of magnetic minerals, while IRM_{flux} provides the flux of magnetic particles encapsulated in the calcite per unit time, and compensates for growth rate changes. We used coercivity unmixing to characterize the magnetic components in 11 samples: five samples from stalagmite 11-24, three samples from stalagmite 2-20, two samples from the overlying Terra Rossa Mediterranean soil (at depths of 5 and 30 cm), and one sample from clay collected inside the cave. The procedure included 95 alternating field (AF) demagnetization steps from 1 mT to 250 mT after applying the 1.5 T field. Unmixing data were analyzed using the MAX UnMix program (Egli, 2004; Maxbauer et al., 2016).

A 2×3 cm slice cut from the middle of stalagmite 11-24 (Fig. 1B) was used for electron microscopy using an extreme high resolution

scanning electron microscope (XHR-SEM; Magellan 400L). The slice was cleaned but not polished, to preserve the magnetic particles occurring between calcite crystals. See Section S3 of the Supplemental Material for the extended methods.

ORIGIN OF MAGNETIC PARTICLES

Electron microscopy coupled with energy dispersive spectroscopy (EDS) analysis shows the presence of $0.5 \mu\text{m}$ to $\sim 2 \mu\text{m}$ anhedral ferromagnetic grains occurring at the interstices of speleothem calcite grains (Fig. S5). The morphology of the sub-micrometer particles resembles pedogenic magnetite (Strauss et al., 2013), where the larger ones may be weathered detrital particles. Coercivity spectra of the stalagmites samples (Fig. S6a) broadly agree with values reported in previously published speleothem studies (Lascu and Feinberg, 2011; Osete et al., 2012; Bourne et al., 2015; Zhu et al., 2017) and are in the range of pedogenic and detrital magnetite (Egli, 2004). The IRM unmixing data, interpreted with two end-member populations (high-coercivity [HC; median destructive field, MDF, >25 mT] and low-coercivity [LC; MDF <15 mT]), show a general agreement between the stalagmites, the overlying soil, and the clay material found in the cave. The contribution of the HC component in the soil and the clay is lower than that of the stalagmites (Section S2 and Table S2), indicating that the grains incorporated into stalagmites were filtered toward smaller grain sizes when infiltrating through the karst. The IRM unmixing of local dolomite bedrock, characterized by a weak IRM of $<10^{-7} \text{Am}^2$, revealed a significant (30%–50%) component with much higher coercivity (>250 mT), which could not be fully demagnetized with standard AF methods (Fig. S6b). This dolomite-driven

component is not incorporated in the stalagmites. Our observations support the underlying assumption that the magnetic minerals in the speleothems are micrometer- to sub-micrometer-sized particles derived from overlying soil and filtered through the karst system.

MAGNETO-ISOTOPIC TIME SERIES

The interval spanning ca. 9.7 to ca. 5.4 ka (Fig. 2) includes the absolute maxima and minima in the entire Holocene $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ records of Soreq (Bar-Matthews et al., 2019), representing the most extreme climate variations of the interglacial. Inspection of the overlap-

ping time series between ca. 7.0 and ca. 6.4 ka shows that IRM_{mass} values of stalagmite 2-20 are approximately half an order of magnitude higher than those of stalagmite 11-24 (Fig. 2E). This difference is explained by the drip site-specific, nonlinear nature of karst hydrology (Treble et al., 2013) associated with the stalagmites that formed in different parts of the cave (Fig. S2). Specifically, the geochemical model for Soreq Cave (Burstyn, 2013; Section S1) estimates a higher fraction of soil-derived elements and a significant fraction of vadose flow in the shallower locations, where stalagmite 2-20 was formed. By contrast, IRM_{flux} values of the two

stalagmites are within the same value range, and their mean IRM_{flux} is indistinguishable within 1σ uncertainty. This suggests that IRM_{flux} is a potential normalized index for comparing multiple samples from the same cave (Section S4 and Tables S3–S6).

EARLY-HOLOCENE WET PERIOD

The early-Holocene recorded in stalagmite 2-20 is marked by a rapid decrease in IRM_{flux} to minimum Holocene values (Fig. 2D). The low IRM_{flux} coincides with a distinct isotopic event between ca. 9.5 and ca. 7.1 ka, where $\delta^{13}\text{C}$ values are uniquely high (-8‰ to -4‰ ; Fig. 2C)

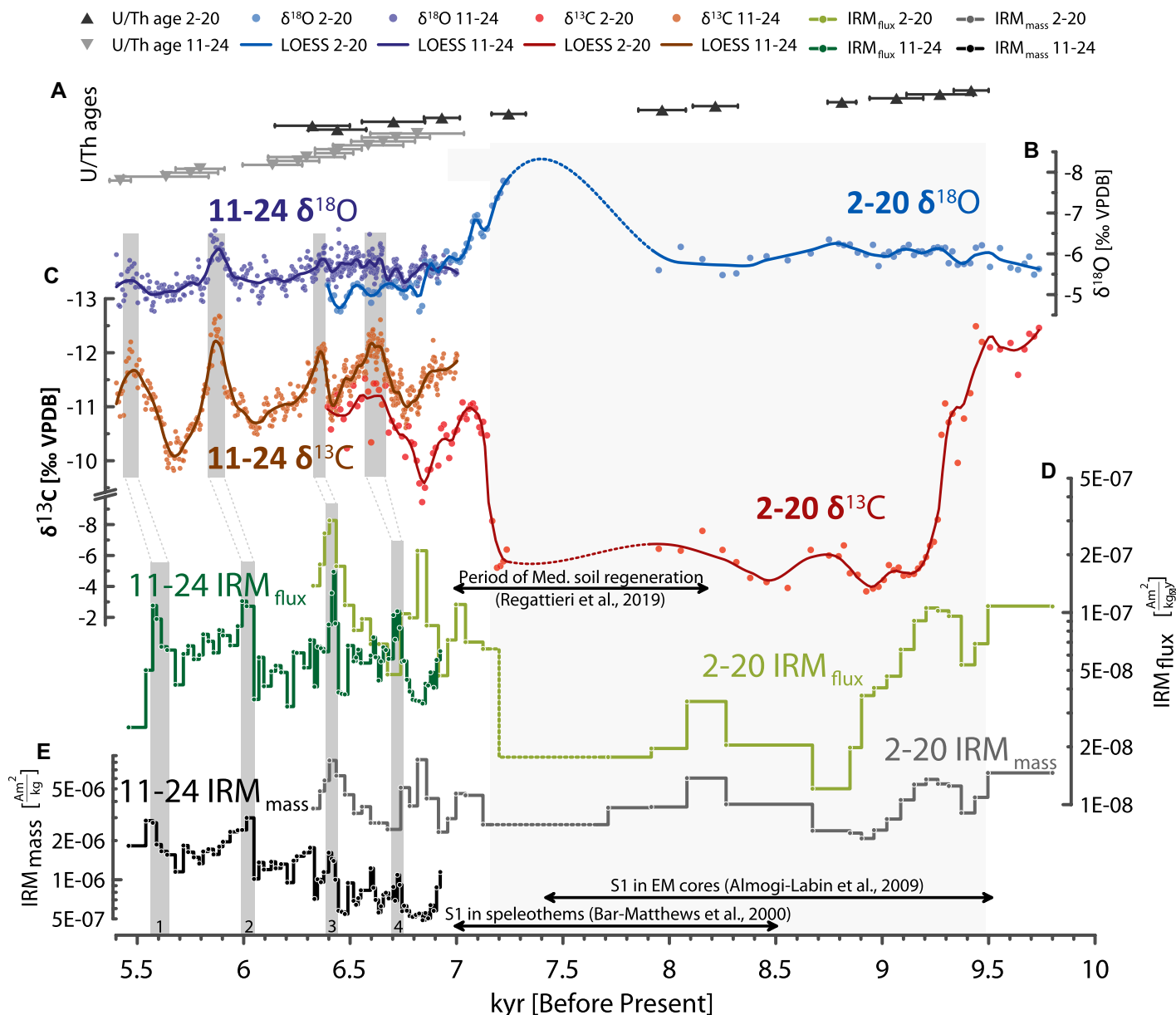


Figure 2. Composite multi-proxy time series of stalagmites 2-20 and 11-24 from Soreq Cave, Israel. (A) U-Th ages used for the age model. (B,C) $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ time series superimposed with a locally estimated scatterplot smoothing (LOESS) function with a Gaussian kernel and second-order locally weighted polynomial. Light gray shaded area is the isotopic excursion defining sapropel layer S1. Dark-gray vertical bands in C and E highlight four mid-Holocene wet-dry cycles (see text for details). VPDB—Vienna Pee Dee belemnite. (D,E) Isothermal remanent magnetization normalized to flux (IRM_{flux}) and to mass (IRM_{mass}). Sapropel 1 (S1) timing is marked by black arrowed lines in E and by light-gray shading throughout the plot. Timing of Mediterranean (Med.) soil regeneration in D is marked by black arrowed line (Regattieri et al. (2019).

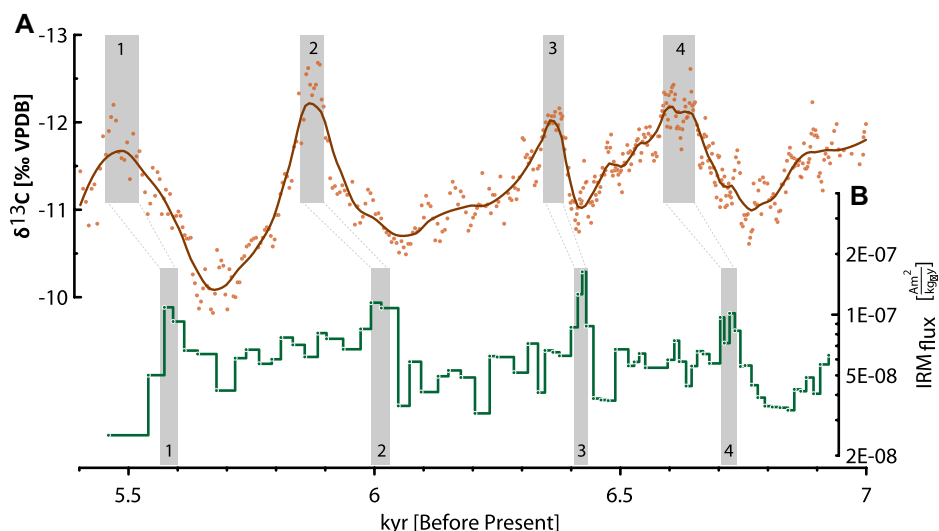


Figure 3. Correlation between $\delta^{13}\text{C}$ (A) and isothermal remanent magnetization normalized to flux (IRM_{flux}) (B) in stalagmite 11-24 from Soreq Cave, Israel during mid-Holocene wet-dry cycles. Cycles are highlighted here by dark-gray vertical bands numbered 1 through 4. Colors of dots and lines are as in Figure 2. VPDB—Vienna Pee Dee belemnite.

relative to the Soreq overall mean, approaching bedrock values of 2‰ (Section S1). Conversely, the termination of early-Holocene conditions is observed between ca. 7.2 ka and ca. 7.1 ka as stalagmite 2-20 $\delta^{13}\text{C}$ values decreased back to C_3 -like values (−10‰ to −12‰), simultaneous with IRM_{flux} returning to pre-sapropel values (Figs. 2C and 2D). This excursion in the stalagmite record corresponds with sapropel layer S1 in the eastern Mediterranean sea (striped gray bar in Fig. 2), thought to be warm and humid (Nehme et al., 2015; Bar-Matthews et al., 2019; Goldstein et al., 2020). Bar-Matthews et al. (2003) explained the $\delta^{13}\text{C}$ excursion as meteoric water reaching the cave after little interaction with overlying soil due to either (1) recently eroded soils, as water percolating through minimal soil cover will be undersaturated in low- $\delta^{13}\text{C}$ soil CO_2 (Genty et al., 2001), or (2) high runoff fraction, circumventing the soil pathway altogether. Both hypotheses are supported by a coeval drop in $^{87}\text{Sr}/^{86}\text{Sr}$ and initial ($^{234}\text{U}/^{238}\text{U}$) ratios toward bedrock ratios (Ayalon et al., 1999). The IRM_{flux} as a soil contribution proxy may help differentiate between these hypotheses.

Thus, the entire early Holocene is bracketed between the ca. 9.5 ka weakening of the soil-related isotopic component and the large decrease in IRM_{flux} , followed by the ca. 7.1 ka rehabilitation of the C_3 isotopic signal and IRM_{flux} values, which is coupled with evidence of soil regeneration in the circum-Mediterranean region (Regattieri et al., 2019). We believe that the structure of the low- IRM_{flux} event (rapid decrease/increase) and the regional evidence of soil regeneration in the termination of that event supports the hypothesis that a change in soil cover or soil stability resulted in a change in $\delta^{13}\text{C}$ (and IRM_{flux}) values. Apparently, the post-glacial

increase in rainfall destabilized soils, perhaps coupled with a “shut down” of soil production due to a decrease in dust input during the “green Sahara” phase (see Rohling et al., 2015).

MID-HOLOCENE WET-DRY CYCLES

The post-sapropel mean IRM_{flux} values suggest that the soil overlying Soreq Cave was regenerated and stabilized by the mid-Holocene. The four saw-tooth cycles in IRM_{flux} between ca. 7.0 and ca. 5.4 ka (numbered in Figures 2E and 3) are also evident in the $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ records (Section S4). These cycles were attributed to wet-dry events (Mayewski et al., 2004; Bar-Matthews and Ayalon, 2011). Bar-Matthews and Ayalon (2011) interpreted low $\delta^{18}\text{O}$ values as an increase in regional rainfall superimposed with strong annual variations, while the $\delta^{13}\text{C}$ cyclicity was smoothed and amplified by complex soil processes, as they represent changes in soil organic carbon. Thus, the post-sapropel regeneration of soil is linked to the emergence of a positive relationship between IRM_{flux} and rainfall amount, as also observed in other mid-latitude sites (Zhu et al., 2017; Regattieri et al., 2019; Feinberg et al., 2020).

The mid-Holocene record also highlights an apparent phase shift between the physical flux of magnetic particles into Soreq Cave and the chemical processes affecting the $\delta^{13}\text{C}$ of drip water (Fig. 3). The IRM_{flux} peaks precede $\delta^{13}\text{C}$ minima by ~60 to 120 yr, where a maximum correlation factor between the two records is achieved by a time-shift of 60 yr (Fig. S7). This lag can be explained by the mechanisms governing the two proxies: (1) An increase in rainfall typically results in increased production of pedogenic magnetic minerals (Maxbauer et al., 2017, and references within) simultaneously en-

hancing physical flushing of soil particles into the cave. (2) In contrast, the change in soil water content, temperature, vegetation cover, and soil activity that affect biogenic soil- CO_2 concentrations may require several decades to develop and affect $\delta^{13}\text{C}$ values (Genty et al., 2001). Another hypothesis is that a rise in rainfall increases vegetation cover to a certain threshold, inhibiting transfer of fine-grained magnetite (e.g., Chen et al., 2019) and increasing the time lag between peak values of each proxy.

CONCLUSIONS

Soreq Cave demonstrates two opposite responses of the magnetic flux parameter to increasing rainfall. During the early-Holocene, low- IRM_{flux} and high- $\delta^{13}\text{C}$ indicate minimal contribution from soils, adding to the isotopic-based hypothesis of soil removal and regeneration during the prevailing warm-humid climate conditions of sapropel S1 (e.g., Goldstein et al., 2020). During the mid-Holocene, couplets of high- IRM_{flux} and low- $\delta^{13}\text{C}$ are associated with increased precipitation of wet-dry cycles. A phase shift between the couplets may elucidate soil and vegetation dynamics, crucial to interpreting subannual-resolution climate data (e.g., Orland et al., 2014). We show that IRM_{flux} in speleothems is a valuable proxy for regional changes in soil-related processes. IRM_{flux} could be used to resolve controversies in interpreting the complex behavior of isotopes in geological records, and could hold the potential to constrain soil carbon turnover response to climate events.

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