Supplemental Material

New chronology of the Chinese loess-paleosol sequence by leaf wax δD records during the past 800 k.y.

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1. Materials and Methods

Site description. The Xifeng loess profile is located at the central Chinese Loess Plateau (35°40'N, 107°39'E; Fig. S1). The mean annual air temperature is 8.7 °C and the mean annual precipitation is 547 mm over the last 50 years. The modern vegetation of this region is dominated with typical temperate steppe. Loess samples were collected from two sites about 3 km apart at the Xifeng village: 166 samples with 20 cm increments above the L1 layer and 5 cm increments in S1 horizon from a 16 m-deep shaft and 158 samples with 25 cm increments of L8–L2 layers from a 64 m core.

Magnetic susceptibility and grain size analysis. Magnetic susceptibility was measured using a MS₂ Bartington magnetic susceptibility meter at the Institute of Earth Environment, Chinese Academy of Sciences. For medium grain size analysis, samples were pretreated to remove organic matter and calcium carbonate using 30% hydrogen peroxide (H₂O₂) and 6N hydrochloric acid, respectively, and then dispersed by sonification with 10 ml 10% (NaPO₃)₆ solution. Grain size distribution was carried out on a Malvern 2000 laser instrument with an analytical error of within 2% at the Institute of Earth Environment, Chinese Academy of Sciences.

δD_{wax} analysis. Each sample (~30 g) was extracted with a mixture of dichloromethane (DCM) and methanol (MeOH) (9:1, v/v) under 100°C heat and 1500 psi pressure for 1 hour using Accelerated Solvent Extraction (ASE 350, DionexTM). Total extracted lipid compounds were transported *via* a soft N₂ stream. The *n*-alkane fractions were separated by silica gel chromatography using hexane eluents. Gas chromatography (GC) was

performed using an Agilent 7890 Series instrument equipped with a split-injector, HP-1ms GC column (60 m length; 0.32 mm i.d.; 0.25 μ m film) and a flame ionization detector. The samples were injected in split mode at an inlet temperature of 310°C and a column flow rate of 1.2 ml/min. The analysis was run under the following temperature ramps: held for 1 min at an initial temperature of 40°C; heated to 150°C at 10°C/min; and then heated to 310°C at 6°C/min and held for 20 min. The δD_{wax} were measured using a Delta-V IRMS (Thermo-Finnigan). The temperature ramps were the same as those used in the GC analysis. The *n*-alkanes were converted to hydrogen gas using a high-temperature pyrolysis reactor at 1430°C. Most of samples were analyzed in duplicate or more, and the standard deviation was usually < 3‰. Mixed laboratory standards of *n*-alkanes (odd carbon number *n*-alkanes; C₂₁–C₃₃; Sigma-Aldrich) were measured after every four injections to monitor the external precision, and the standard deviation of the working standard was < 2‰. The working standard was calibrated using Indiana University FAME reference substances. The GC analyses and δD_{wax} analyses were performed at the Stable Isotope Laboratory of the Institute of the Earth Environment, Chinese Academy of Sciences.

Chronologies. The chronologies of the loess–paleosol sequences were established by the orbital parameter tuning method (Kukla et al., 1988; Ding et al., 1994). The tuned age is calculated on the weighted interpolation method reported by Kukla et al. (1988).

$$T_m = T_1 + (T_2 - T_1) \left(\sum_{i=1}^m a_i p_i \right) / \left(\sum_{i=1}^n a_i p_i \right)$$

where T_m is the tuned age of given level (m), T_1 and T_2 are age of control points, n is the total sampling level between T_1 and T_2 ; a_i and p_i are the accumulation rate and value of tuned-proxy at level i . The traditional magnetic susceptibility tuned chronology were established on 2 cm incremental data, the loess core magnetic susceptibility curves for this

study were then compared with the previous Xifeng records (Guo et al., 2009). For the new chronology on loess δD_{wax} , the age control points up to L8 loess layer were transferred from boreal insolation maximum by correlating them with low value peaks of δD_{wax} .

2. Comparison of grain size, magnetic susceptibility and δD_{wax} records from S1 layer and up in the central and south of the CLP

Samples in the Xifeng profile were collected from the bottom of the S1 layer upward, and their grain size, magnetic susceptibility and δD_{wax} of *n*-alkanes were measured and then compared with those collected from the Weinan profile (Thomas et al., 2016). δD_{wax} records show almost the identical characteristics of three low-value peaks in the S1 paleosol horizon (Fig. S1). It means that the precipitation isotopes may be completely synchronous in the central and south of CLP. On the other hand, detailed δD_{wax} values vary off phase with magnetic susceptibility and grain size indexes at the same depth.

3. Comparison of grain size, magnetic susceptibility and δD_{wax} records of S2 layer in the central and south of CLP

The comparison of δD_{wax} in the S2 layer between the lower-resolution record of Xifeng (Wang et. al., 2018) and higher-resolution record of Weinan (Thomas et al., 2016) also shows the consistency of paleo-precipitation isotope changes among CLP sites (Fig. S2). Moreover, similar to the characteristics of the S1 layer, peaks of δD_{wax} in the S2 layer are not synchronous with peaks of magnetic susceptibility and grain size records. The δD_{wax} peaks that are tuned by using magnetic susceptibility and grain size proxies lag behind the

peaks of stalagmite $\delta^{18}\text{O}$ proxy in central China (Cheng et al., 2016) and the boreal summer insolation (Laskar et al., 2004).

4. Spectrum analysis of loess $\delta\text{D}_{\text{wax}}$ record

The wavelet power spectrum analysis indicates that the $\delta\text{D}_{\text{wax}}$ variation in the central CLP since the last interglacial have clear periodicity of 23-ka and 41-ka under the new time-scale, while the magnetic susceptibility and grain size in the same region do not record those cycles (Fig. S3a). Below the S1 stratigraphy, due to the lower sampling resolution, the 23-ka cycle of loess $\delta\text{D}_{\text{wax}}$ becomes weak (Fig. S3b). Even so, the comparison result is sufficient to confirm that the loess $\delta\text{D}_{\text{wax}}$ proxy reflects periodic changes on sub glacial-interglacial scale. It means that loess-paleosol sequences indicated by loess $\delta\text{D}_{\text{wax}}$ proxy are consistent with the periodicity of other important global records.

5. The differences between two age-models in paleosol and loess layers

Intense summer monsoon precipitation could enhance the leaching process causing high degree of dissolution of magnetic minerals and translocation of fine-grained loess particles during the interglacial period. Therefore, in theory, the age uncertainty of traditional loess chronology in interglacial-scale paleosol layers may be greater than that in loess layers. However, the current results do not support this hypothesis, and the age differences do not differ statistically between paleosol and loess forming intervals (Fig. S4). We believe that the lower resolution of the data below L2 layer may introduce additional error to prove this hypothesis. It is necessary to carry out more high-resolution work in the future.

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DR Figures:

Fig. S1. Comparison of loess records from S1 layer and up in central and south of CLP. a, Location of the Xifeng profile site, 1, CLP: Xifeng and Weinan profiles in the CLP; 2, HL: Hulu cave; 3, SB: Sanbao cave; 4, XBL: Xiaobailong cave; 5, KS: Kesang cave; b, grain size in Xifeng (orange line) and Weinan (gray line); c, magnetic susceptibility; d, δDwax of n -alkanes in Xifeng and δDwax of n -acid in Weinan, dash lines indicate the peaks of δDwax . Data of Weinan profile were cited from Thomas et al. (2016).

Fig. S2. Comparing summer insolation with δD_{wax} of S2 layer in central and south CLP. a, magnetic susceptibility records of Xifeng (green line, this study; red line, Guo et al., 2009) and Weinan (gray line, Thomas et al., 2016); b grain size records of Xifeng (red line, Guo et al., 2009) and Weinan (gray line, Thomas et al., 2016); c δD_{wax} of Xifeng (green line, Wang et al., 2018) and Weinan (gray line, Thomas et al., 2016); d stalagmite $\delta^{18}O$ in central China (gray line, Cheng et al., 2016) and summer insolation of $65^{\circ}N$ (yellow line, Laskar et al., 2004); e, marine benthic $\delta^{18}O$ stack (Lisiecki and Raymo, 2005).

Fig. S3. Comparison of wavelet power spectrum between CLP δDwax and other global records. a, wavelet power spectrum during 0-150 ka; b, wavelet power spectrum since the last 800 ka. Marine $\delta^{18}\text{O}$: marine benthic $\delta^{18}\text{O}$ stack (Lisiecki and Raymo, 2005); EDC δD : δD of EDC ice core in Antarctica (Jouzel et al., 2007); Tropical SST: sea surface temperature of ODP 1146 core in South China Sea (Herbert et al., 2010); CLP MS and CLP GS: magnetic susceptibility and grain size in the central CLP under the traditional tuning time-scale (Guo et al., 2009); CLP δDwax : loess δDwax in the central CLP under the new chronology, this study; Stalagmite: stalagmite $\delta^{18}\text{O}$ in central China (Cheng et al., 2016); Inso 65°N Jul: Boral summer insolation of 65°N (Laskar et al., 2004). The green lines show the periods of 23 ka, 41 ka and 100 ka, respectively. MATLAB software is used for wavelet power spectrum analysis followed the method reported by Torrence and Compo (1998).

Fig. S4. The age difference between two age-models in the paleosol and loess layers. The box shows the median value and the standard deviation, the gray dots are age differences in the high resolution sequence above S1 layer.