Radiocarbon dating of the Pleistocene/Holocene climatic transition across the Chinese Loess Plateau

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A R T I C L E I N F O

Article history:
Received 8 March 2016
Received in revised form 6 June 2017
Accepted 20 June 2017
Available online 23 June 2017

Keywords:
Radiocarbon  
\( ^{18} \text{O} \)
Land snails
Pleistocene/Holocene transition
Chinese Loess Plateau

A B S T R A C T

The magnetic susceptibility (MS) of Chinese loess deposits is extensively used to reconstruct climate changes from centennial to earth orbital time scales. The approach assumes that the MS is sensitive to the variations of the Asian Summer Monsoon (ASM), and thus that changes in MS are synchronous across the Chinese Loess Plateau. However, this inference has not been fully confirmed due to the limitations of both chronological control of Chinese loess sequences and the climatic proxies of the ASM. Here, we present the results of radiocarbon dating of the shifts in the oxygen isotopic composition of fossil land snail shells (\( ^{18} \text{O} \)) and in MS during the Pleistocene/Holocene transition at various sites in Chinese Loess Plateau. The results show that the age of shift in MS at Pleistocene/Holocene transition at Mangshan, Heshui, and Huanxian sections is 10.43 ka, 8.55 ka, and 7.13 ka BP, respectively. Thus, the shift is time-transgressive with age decreasing from southeast to northwest across the plateau. However, the \( ^{18} \text{O} \) record, which is directly related to the precipitation delivered by the ASM, is not significantly time-transgressive, indicating that the ASM strengthened almost simultaneously across the plateau. We suggest that the time-transgressive nature of the shifts in MS may result from the low amplitude of the ASM strengthening during the Pleistocene/Holocene transition. Overall, our results demonstrate a regional difference in the insensitivity of MS to low amplitude climatic changes, and they challenge the previously-held assumption that the ages of climatic boundary based on MS stratigraphy are time-equivalent in Chinese loess.

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1. Introduction

The thick loess-paleosol sequences of the Chinese Loess Plateau (CLP) provide an unrivalled continuous terrestrial record of climatic change extending back to over 22.0 Ma (Guo et al., 2002). The records from climatic proxies, such as the magnetic susceptibility (MS) and the sediment grain-size distribution of Chinese loess, have been widely used to reconstruct and interpret the dynamics of global and regional climatic change (An, 2000; Balsam et al., 2005; Derbyshire et al., 1995; Ding et al., 1995; Guo et al., 1996; Hao et al., 2012; Porter and Zhisheng, 1995; Sun et al., 2006). Much or all of this work is based on the chronologies established by correlating the MS record or grain-size distributions of loess with the astronomically tuned oceanic \( ^{18} \text{O} \) record (Imbrie et al., 1984; Martinson et al., 1987). The methods used include proxy-based (Kukla et al., 1988), statistical correlation to the \( ^{18} \text{O} \) record (Bloemendal et al., 1995), independent astronomical tuning (Ding et al., 2002), and direct correlation to the \( ^{18} \text{O} \) record (Balsam et al., 2005). These methods are based on two key assumptions: 1) the loess records are continuous and highly resolved, and the resolution of the climate signals has not been significantly impacted by post-depositional diagenesis; and 2) the major changes inferred from the proxy records (e.g. the MS) are synchronous not only within the CLP, but also with those of the marine oxygen isotope record. However, recent work, such as the chronological studies based on Optically Stimulated Luminescence (OSL), challenge the validity of these two assumptions (Lai and Wintle, 2006; Lu et al., 2006; Stevens et al., 2006), especially during the Pleistocene/Holocene transition.

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http://dx.doi.org/10.1016/j.quageo.2017.06.003
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The results of OSL dating of loess sequences at Beiguoyuan, Xifeng, Shiguanzhi, and Huanxian sites indicate that sedimentation was episodic at suborbital time scales, and that unconformities, cessation of eolian deposition, and mixing of sediments are common (Lu et al., 2006; Stevens et al., 2006). Discontinuities within Chinese loess sequences are also evidenced by welding and truncations of paleosol, erosion surfaces (Derbyshire et al., 1997), and wind deflation (Zhengtang and Dongsheng, 1996). The OSL age of the S0/L1 boundary defined by the MS shift at Xifeng and Shiguanzhi (ca. 20 ka) indicates a significant pedogenic overprinting of the late glacial loess (Stevens et al., 2006). Similar post-depositional diagenesis has also been detected at the Beiguoyuan and Yuanbao sites (Lai and Wintle, 2006; Stevens et al., 2007). With regard to the second assumption, the Pleistocene/Holocene boundary defined by a rapid shift in MS is dated at around 10.5 ka, 8.5 ka and 7.5 ka in the Yaxian, Jinchuan, and Huanxian sections, respectively, indicating that the boundary is time-transgressive across China (An et al., 2000; He et al., 2004). This evidence indicates that previous conclusions about the timing and nature of climatic and environmental changes during the Pleistocene/Holocene transition inferred from Chinese loess records need to be reassessed.

Given the discontinuous nature of loess sedimentation, it has been suggested that independent dating is the only means of obtaining an effective chronology for determining the history of depositions and for climatic reconstruction (Lu et al., 2006; Stevens et al., 2006). However, the occurrence of post-depositional diagenesis of loess indicates that the use of climatic proxies that are relatively unaffected by the vagaries of sedimentation and pedogenic diagenesis, is equally important. This is because it is difficult to separate the signals of spatially-synchronous pedogenic processes from those of long-term post-depositional diagenesis by absolute dating alone. The coupled radiocarbon dating and analysis of the oxygen isotopic composition of fossil land snails (δ18O) has significant potential for establishing an accurate chronology of loess accumulation and climate history, for two reasons: 1) δ18O is often directly related to the regional precipitation during the interval when the snails are alive (Balakrishnan et al., 2005; Leng et al., 1998; Liu et al., 2006; Zanchetta et al., 2005); and 2) the combined radiocarbon and δ18O analysis of land snails excludes the possibility of a phase difference between the dating material and the climate proxy. However, to the best of our knowledge, no previous study has used this approach to determine the timing of climatic changes during the Pleistocene/Holocene transition on the CLP.

Here, we present the results of the first coupled analysis of the radiocarbon and oxygen isotopic compositions of land snail shells collected from three loess sequences, spanning the main part of the CLP. Our main aims are to characterize and date the changes in the ASM across Pleistocene/Holocene transition, and to provide fresh insights into the nature of climate changes across Chinese Loess Plateau.

2. Materials and methods

The three study sites, Mangshan, Heshui, and Huanxian, are located along a southeast-northwest transect, which spans the steepest climatic gradient across the CLP, as well as significant differences in the distance to the moisture sources of the Asian Summer Monsoon (Fig. 1). The Mangshan section is located on Mangshan Yuan (34.9603 N, 113.2675 E, 255 m), north Henan Province. Mangshan Yuan is a small plateau consisting of eolian loess, which is presumed to have been the most southeastern part of the Loess Plateau before it was separated by the Yellow River (Jiang et al., 2007). The site has a mean annual temperature (MAT) of ~14.0 °C and a mean annual precipitation (MAP) of ~640 mm. The sampled part of the section is composed of the whole of the Black Loams (1.3 m thick) and the upper 3.1 m of the Malan Loess. The Heshui section (35.8618N, 107.8814E, 1171 m asl) is in northwestern Heshui county, Gansu Province. The MAT and MAP of the area are ~10 °C and ~570 mm, respectively. The samples from this section spanned the whole of Black Loam (1.5 m thick) and upper 1.0 m of the Malan Loess. The northernmost section, Huanxian (36.6233°N, 107.2861°E), is situated in the northernmost part of the Loess Plateau near the city of Huanxian in Gansu Province. The MAT of the area is ~8.3 °C and the MAP is ~350 mm. The studied part of the section includes the whole of the Black Loam (1.5 m thick) and the uppermost 1.2 m of Malan Loess.

A total 95 of samples were collected from the 3 sections at a 10-cm interval for MS measurements. At the same time, soil samples of more than 10 kg weight (range: 10–30 kg) were obtained at a 10-cm interval for collecting fossil land snails. After wet sieving, shells of Cuthicus, which can provide accurate radiocarbon ages (Xu et al., 2010, 2011), were picked out for 14C dating and oxygen isotope analysis. To reduce the possible influence of diagenesis, only well-preserved samples (whole shells) were collected. In addition, our previous studies showed that the measured oxygen isotopic composition of snail shells from the Chinese Loess Plateau attains a stable value with the analysis of an increasing number of individual shells (Liu et al., 2006). Therefore, only samples with more than 7 fossil shells were selected for the radiocarbon dating and stable oxygen isotope analysis. At the same time, a total of 15 samples of modern living snails, each of which consisted of more than 5 shells, were collected from the studied areas to establish a regional baseline value of modern δ18O.

The samples of both fossil and living snail shells were sonicated repeatedly until the water was clean. The shells were then viewed under an optical microscope to remove any remaining macroscopic contamination. Then, the samples were reacted with 6% NaOCl for 48–72 h to remove organic matter, washed repeatedly, and sonicated for 5–10 min to remove any remaining adhering material. The cleaned samples were then washed with 0.01 HCl to remove adhering carbonates, rinsed with distilled water, and dried in a vacuum oven overnight at 70 °C. Finally, the dried samples were ground into 150-mesh powder. To further assess the possible impact of post-depositional diagenesis, a portion of the fossil samples was used for mineral phase analysis by X-ray diffraction (XRD). Only samples without aragonite-calcite transformation, which indicates either no or negligible diagenetic effect (Xuefen et al., 2005), were used for radiocarbon dating and oxygen isotopic analysis. A total of 20 fossil samples were analyzed from the three sites (9 from Mangshan, 6 from Heshui, 5 from Huanxian).

Each powdered fossil snail sample was split into two portions. One portion was converted to CO2 by combustion in vacuum and reduced to graphite at a temperature of 525 °C (Getachew et al., 2006), and then sent to the Accelerator Mass Spectrometry (AMS) facility of Peking University for 14C analysis. The second portion of the fossil samples, as well as the powder of modern snail shells, was converted to CO2 using 100% phosphoric acid in a vacuum and the oxygen isotopic composition measured using a MAT 252 mass spectrometer. The isotopic data are reported in the conventional notation as per mil (%e) deviations relative to the PDB standard with an uncertainty (1σ) of 0.02 (%e). The MS of the three sections was measured in the laboratory on air dried samples using a Bartington Instruments MS2 susceptibility meter following the procedures of Liu (1985) and Kulka et al. (1988).

3. Results

The land snails that graze or aestivate on limestone substrates may incorporate older carbon into their shells by ingestion or
absorption, causing ‘dead carbon anomalies’ (Goodfriend, 1987; Pigati et al., 2010; Romaniello et al., 2008). Therefore, it is necessary to correct for the effect of dead carbon on the radiocarbon age of land snail shells. According to previous studies (Xu et al., 2010, 2011), the contribution of dead carbon in the shells of Cathaica is consistent across the CLP with an age anomaly of −1130 yr. Therefore, the fossil shells of Cathaica can provide reliable radiocarbon ages after the application of an appropriate correction factor. Age versus depth curves for three studied sites, with radiocarbon ages corrected by the method of Xu et al. (2011), are illustrated in Fig. 2. The results show that the radiocarbon ages increase with depth at all three sites, and they further demonstrate the validity of the age correction approach for Cathaica shells on the CLP.

In all three sections, the radiocarbon ages of the Cathaica shells are mainly derived from the loess sequences before the significant shifts of the MS (Fig. 2). Only in the Mangshan section are Cathaica shells well preserved in the upper part of the Black Loam. This pattern is the result of the intense weathering and pedogenesis of the Black Loam. Although there are no well-preserved Cathaica shells in the Black Loam, the radiocarbon ages provide a reliable chronology for the intervals from 16.08 to 8.55 ka BP for the Heshui section, and from 16.49 to 7.13 ka BP for the Huanxian section (Fig. 2), which is adequate for dating the climatic changes spanning the Pleistocene/Holocene transition.

The oxygen isotopic composition of modern living snails ($\delta^{18}O_{ms}$) from the study region is shown in Fig. 2. For the Mangshan section, the values range from −6.26‰ to 7.01‰ ($\bar{x} = 6.60‰$, $\sigma = 0.22‰$). For the Heshui and Huanxian sections, the values are concentrated within the range of −5.30‰ to 4.74‰ (with $\sigma = 0.31‰$ and $\sigma = 0.29‰$ for Heshui and Huanxian, respectively). The relatively low standard deviations are consistent with previous finding that the $\delta^{18}O_{ms}$ values in the CLP are concentrated around a stable value as increasing numbers of shells are analyzed (Liu et al., 2006). The most significant feature of the data set is that the $\delta^{18}O_{ms}$ values exhibit a clearly increasing trend from southeast to northwest across the plateau.

The variation of $\delta^{18}O$ along the studied loess sequences is also plotted in Fig. 2. A prominent feature is the significant $^{18}O$-depletion during the Pleistocene compared to the modern counterparts (Fig. 2). At all three sections, there was little change in $\delta^{18}O$ during the Pleistocene, however, there was an increasing trend in the values from southeast to northwest across the CLP, as is also shown by the modern values. This evidence, together with the small deviations in $\delta^{18}O_{ms}$ values, indicates that the microenvironment had only a limited impact on the oxygen isotopic composition of individual snails, and thus that the $\delta^{18}O$ values reflect changes in large-scale, dominant factors that control the oxygen isotopic composition of snail shells in the CLP. Another prominent feature is that the shift in $\delta^{18}O$ values to negative around the Pleistocene/Holocene transition occurred at almost the same time (~12.0 ka BP) in the three studied sections within the dating uncertainty, and approached the values of the modern counterparts in the Holocene (Fig. 2).

4. Discussion

To constrain the climatic implications of $\delta^{18}O$ values, many studies have been carried out on modern living snails. Most of the studies include modern observations and flux models, and they document a good correlation between $\delta^{18}O$ and the amount and isotopic composition of precipitations, especially during the seasons when land snails are active and grow (Balakrishnan and Yapp, 2004; Balakrishnan et al., 2005; Colonese et al., 2013, 2014; Lécôle, 1985; Liu et al., 2006; Prendergast et al., 2015; Yanes et al., 2008, 2009; Zanchetta et al., 2005). However, the relationship between modern $\delta^{18}O$ and climate is not always clear. Other factors, such as evaporation, carbonates and plants ingested by snails, and the temperature at which the shells are precipitated, may differ significantly depending on locality and species, and can influence the $\delta^{18}O$ of shells (Colonese et al., 2013, 2014; Prendergast et al., 2015; Yanes et al., 2008, 2009). Therefore, it is important to carefully assess the climatic implications of the $\delta^{18}O$ values in the CLP.
before addressing the main aims of the study.

We have previously made a systematic study of the climatic significance of δ18O values in the CLP. Samples from more than 300 living individuals were collected from 18 sites covering the whole CLP (Liu et al., 2006). The results show that δ18O exhibits a strong linear relationship with mean annual precipitation, particularly with average July precipitations when the ASM prevails (Fig. 3). Most importantly, the δ18O values of the modern snails collected in the present study also exhibit this linear relationship (Fig. 3). This characteristic is consistent with the ecological characteristics of the studied snails, which are active and grow in the warm and humid season when the ASM prevails.

Our δ18O data are also consistent with the isotopic composition of precipitations (δ18Oprecip) in the CLP. Based on the Global Network of Isotopes in Precipitation (GNIP) data set (IAEA and WMO, 2016), the δ18O of the precipitations during the season when the ASM prevails increases from southeast to northwest across the CLP. The average value of δ18Oprecip from June to September increases from −7.36‰ (Zhengzhou station, 6–8 yrs) in the southeastern CLP to −3.64‰ (Zhangye station, 10–11 yrs) in the...
northwestern CLP. Corresponding to the changes in $\delta^{18}$O$_{\text{pre}}$, the $\delta^{18}$O of both the modern living snail and fossil shells exhibits an increasing trend from the southeastern to the northwestern CLP (Fig. 2). In addition, we estimated the $\delta^{18}$O values of the Mangshan site using a steady state, evaporative flux balance model (Balakrishnan and Yapp, 2004; Balakrishnan et al., 2005), since this site is close to the Zhengzhou station of GNP. This flux model emphasizes relative humidity at the time of snail activity as an important factor in determining the $\delta^{18}$O value of aragonite shells. In the CLP, humidity is closely related to the strength of the ASM. The results show that all of the measured values of modern snails (~7.01 to ~6.26‰) fall within the range of the estimated values (~8.4 to ~6.16‰) within the temperature range of 15–25 °C favorable for snail growth (Cowie, 1984; Thompson and Cheny, 1996). This provides another confirmation of the dominant role of summer monsoon in determining $\delta^{18}$O across the CLP.

In all three sections, the $\delta^{18}$O shifts to negative during the Pleistocene/Holocene transition, indicating an increase in the intensity of the ASM (Fig. 2). Although there are no chronological and isotopic data for the upper Holocene strata of the Heshui and Huanxian sections, the low $\delta^{18}$O values of the fossil shells collected from the Holocene Black Loam ($S_0$) in the Mangshan section indicate a strengthening of the monsoon during the Holocene compared to the Pleistocene. This pattern is consistent with the nature of the MS records of sites elsewhere on the CLP, which consistently exhibit high values during the Holocene (An, 2000; An et al., 2000).

In the Chinese Loess Plateau, the Holocene Black Loam, which developed under a strong summer monsoon, is characterized by a dramatically higher MS values compared to the upper part of the underlying loess unit $L_1$. Therefore, most studies define the Pleistocene/Holocene boundary by the shift in MS values. Recent OSL dating of the MS shift shows that it becomes progressively younger from southeast to northwest across the CLP (Dong et al., 2015). The age difference is as much as 4.0 ka between the Weinan section in the southeast and the Huanxian sections in the northwest, and it is interpreted as the result of time-transgression of the ASM across the CLP (Dong et al., 2015). Our results also show that the age of MS shift becomes younger from the southeast to northwest across the CLP (Fig. 2). However, there is no significant difference in the age of the negative $\delta^{18}$O shift around the Pleistocene/Holocene transition across the CLP within the uncertainties of the chronology (Fig. 2). Thus, this finding contradicts the assumption of the time-transgressive character of changes in ASM intensity across the CLP.

Although the records from the Heshui and Huanxian sections are of relatively low resolution and cannot precisely constrain the age of onset of the ASM shift, our data clearly indicate that the age discrepancy between the shifts in the ASM during the Pleistocene/Holocene transition is relatively small across the CLP (significantly less than 4.0 ka). Our radiocarbon-dated $\delta^{18}$O records show that the $\delta^{18}$O values shifted to negative from 13.56 to 10.43 ka BP in the Mangshan section, from 11.96 to 10.05 ka BP in the Heshui section, and from 12.39 to 10.32 ka BP in the Huanxian section. Although the amplitudes of the $\delta^{18}$O shifts are low in the Heshui and Huanxian sections (~1‰), the $\delta^{18}$O values consistently shifted towards modern values in a similar pattern across the Pleistocene/Holocene transition in all studied sections (Fig. 2). This evidence, together with the minor variation of the $\delta^{18}$O across CLP during the Pleistocene, suggests that the $\delta^{18}$O shift around the Pleistocene/Holocene transition was the result of the strengthening of the ASM rather than of other factors.

Our records indicate that the timing of the ASM strengthening falls within the interval from 12.0 to 10.0 ka BP (Fig. 2), and that the summer monsoon intensity reached a relatively high level at 10.0 ka BP. This conclusion is further supported by the consistency of our results with the stalagmite $\delta^{18}$O records from Dongge and Hulu Cave in central and southern China (Wang et al., 2001, 2005), which indicate that the summer monsoon began to increase around 12.0 ka BP and reached a relatively high level around 10.0 ka BP. In addition, well-constrained lacustrine $\delta^{18}$O records (from Lake Ahung, Xingyin, and Qiu from south China; Lake Selin Co, Koucha, Donggi Cona, and Qinghai from the Tibetan Plateau; and Lake Hamaerai and Hetongchahan Nur from central-northern China), also indicate that the AMS was no time-transgressive, and that it reached a relatively high level at around 10.0 ka BP across China (Zhang et al., 2011).

The age discrepancy between the $\delta^{18}$O, and MS records provides critical evidence for understanding the nature of climatic changes during the Pleistocene/Holocene transition. It is well known that soil formation is closely related to precipitation, temperature, sedimentation rate, and duration of pedogenesis. The summer monsoon precipitation is regarded as the major forcing of MS changes within the eolian sequences of the CLP. Therefore, previous studies have suggested that the out-of-phase shifts in MS between the southeastern and northwestern CLP were the result of the time-transgressive nature of changes in ASM intensity (An et al., 2000; Dong et al., 2015). It has been suggested that the northern front of the summer monsoon first reached the southern margin of the CLP at ~15 ka, resulting in heavy rainfall and thus strong soil development, but that it did not reach the northwestern CLP until ~8 ka (Dong et al., 2015). However, our results indicate that the summer monsoon strength increased consistently before 10.0 ka BP across the CLP, and therefore that other factors rather than a time-transgressive summer monsoon intensity were responsible for the out-of-phase in the MS.

We suggest that the low amplitude increase in summer monsoon intensity during the Pleistocene/Holocene transition was the main cause of the out-of-phase shifts in the MS in the CLP. The modern gradient in ASM precipitations indicates a decrease from southeast to northwest across China. Consequently, the absolute amount of precipitation would be significantly less in the northwestern CLP, compared to the southeastern part, given an increase in the intensity of the summer monsoon of the same amplitude. In other words, the precipitation amount would reach the threshold for pedogenesis and neo-formation of secondary magnetic minerals (which control the MS record) in the southeastern CLP with only a small amplitude increase in summer monsoon intensity. In contrast, it would need an increase in ASM intensity of much greater amplitude to reach the threshold for soil development in the northwestern CLP. In the early part of the Pleistocene/Holocene transition, the summer monsoon strengthened simultaneously across the CLP, but the amplitude of the increase was small. This small amplitude change was sufficient to exceed the threshold needed for soil formation in the southeastern CLP, but it was significantly less than that needed to initiate a comparable pedogenic response in northwestern part. Therefore, soil development commenced first in the southeastern margin of the CLP, and with the strengthening of summer monsoon in the Holocene, the climatic threshold required for soil formation moved gradually from southeast to northwest, causing a time-transgressive shifts in the MS records across the CLP. The age difference between the $\delta^{18}$O and the MS shifts provides direct evidence for the gradual progression of the climatic threshold required for soil formation across the CLP. The age difference in the Mangshan section is around 1.2 ka, while it increases to ~1.5 ka and ~3.0 ka in the Heshui and Huanxian sections, respectively (Fig. 2). This inference is consistent with the gradual increase in the ASM intensity in the early Holocene, prior to the Holocene Optimum, detected in various palaeoclimatic records across China (Peng et al., 2005; Wang et al., 2001, 2008; Zhao and Yu, 2012; Zhou et al., 2004).

Our results also have some implications for the use of MS record
5. Conclusion

We have presented the results of the first coupled analysis of the radiocarbon and oxygen isotopic composition of fossil land snail shells ($\delta^{18}O_{\text{P}}$) in the deposits of the Chinese Loess Plateau (CLP). Our data reveal that shift in magnetic susceptibility (MS) associated with the Pleistocene/Holocene transition were time-transgressive from southeast to northwest across the CLP. In contrast, the $\delta^{18}O_{\text{P}}$ values, which are related to the summer monsoon precipitation, shifted to negative almost simultaneously during the Pleistocene/Holocene transition, indicating that the intensity of the Asian summer monsoon began to increase at around 12.0 ka BP and reached a relatively high level at around 10.0 ka BP. The time-transgressive nature of the MS shifts may be associated with the ASM precipitation gradient and the low amplitude strengthening of the ASM during the Pleistocene/Holocene transition. The age difference between the $\delta^{18}O_{\text{P}}$ and MS shifts suggests that the MS record of the CLP is relatively insensitivity to low amplitude climatic changes.

Acknowledgements

We thank Prof. Thomas Higham and the anonymous reviewer for their valuable and constructive advice. Special thanks are owed to Prof. Ding Xingfang for help with the AMS radiocarbon measurements, to Prof. Jan Bloemendal for his invaluable suggestion and language polishing, and to Prof. Zhang Yanhui for the stable isotope analysis. This study was financially supported by the National Natural Science Foundation (grants 41172324, 41472152, 41272201, and 41672181).

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