



# Luminescence-based chronologies for Palaeolithic sites in the Nihewan Basin, northern China: First tests using newly developed optical dating procedures for potassium feldspar grains



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## ABSTRACT

The Nihewan Basin in northern China is a key region in East Asia for the study of early human evolution, owing to the abundance of Palaeolithic sites with ages spanning the entire Pleistocene. However, most of the sites assigned to the Middle to Late Pleistocene have not been dated or are poorly dated, due to the lack of suitable numerical dating techniques. Optically stimulated luminescence (OSL) dating of quartz grains is commonly restricted to deposits younger than ~200 ka, but recent developments using the infrared stimulated luminescence (IRSL) emissions from grains of potassium-rich feldspar (K-feldspar) offer the potential to date Middle Pleistocene deposits using post-infrared IRSL (pIRIR) signals that do not suffer from 'anomalous fading'. In this paper, we report the first archaeological applications of the recently developed pre-dose multiple elevated temperature pIRIR (pMET-pIRIR) procedures for K-feldspar, which we applied to the sedimentary deposits at one Lower Palaeolithic site (Donggutuo) and one putatively Middle Palaeolithic site (Motianling) in the Nihewan Basin. Equivalent dose ( $D_e$ ) values were measured, and non-fading signals were identified, using single-aliquot (SAR) and multiple-aliquot (MAR) regenerative-dose pMET-pIRIR procedures. For a sample from Donggutuo expected to be in field saturation, the natural pMET-pIRIR signals were consistent with, or close to, the laboratory saturation levels only when the MAR procedure was used. For the samples from Motianling, however, both the SAR and MAR procedures could be applied and these yielded indistinguishable  $D_e$  estimates. Our study shows that  $D_e$  values of up to 1500 Gy (or possibly 2000 Gy) can be measured using pMET-pIRIR procedures, corresponding to ages of up to 500 ka (or 650 ka) for deposits with environmental dose rates of ~3 Gy/ka, as is typical for this region. Our results also indicate that samples from a single study area (the Nihewan Basin) can respond differently to the same measurement conditions. As regards the archaeology of the Nihewan Basin, we date the upper part of the cultural layer at the Motianling site to  $322 \pm 33$  ka and the underlying culturally sterile deposits to  $370 \pm 50$  ka. These ages challenge the stratigraphic correlation of the stone artefacts to the Middle Palaeolithic and suggest that they should, instead, be assigned to the Lower Palaeolithic. Given this revised chronology, there is clearly a need to reassess the antiquity of other sites in the Nihewan Basin that have similarly been assigned previously to the Middle Palaeolithic. The pMET-pIRIR procedures tested in this paper show great promise as suitable chronometers for this task, and should be able to provide a timeline for human evolution and activities extending over the last half-million years in this key region of East Asia.

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## 1. Introduction

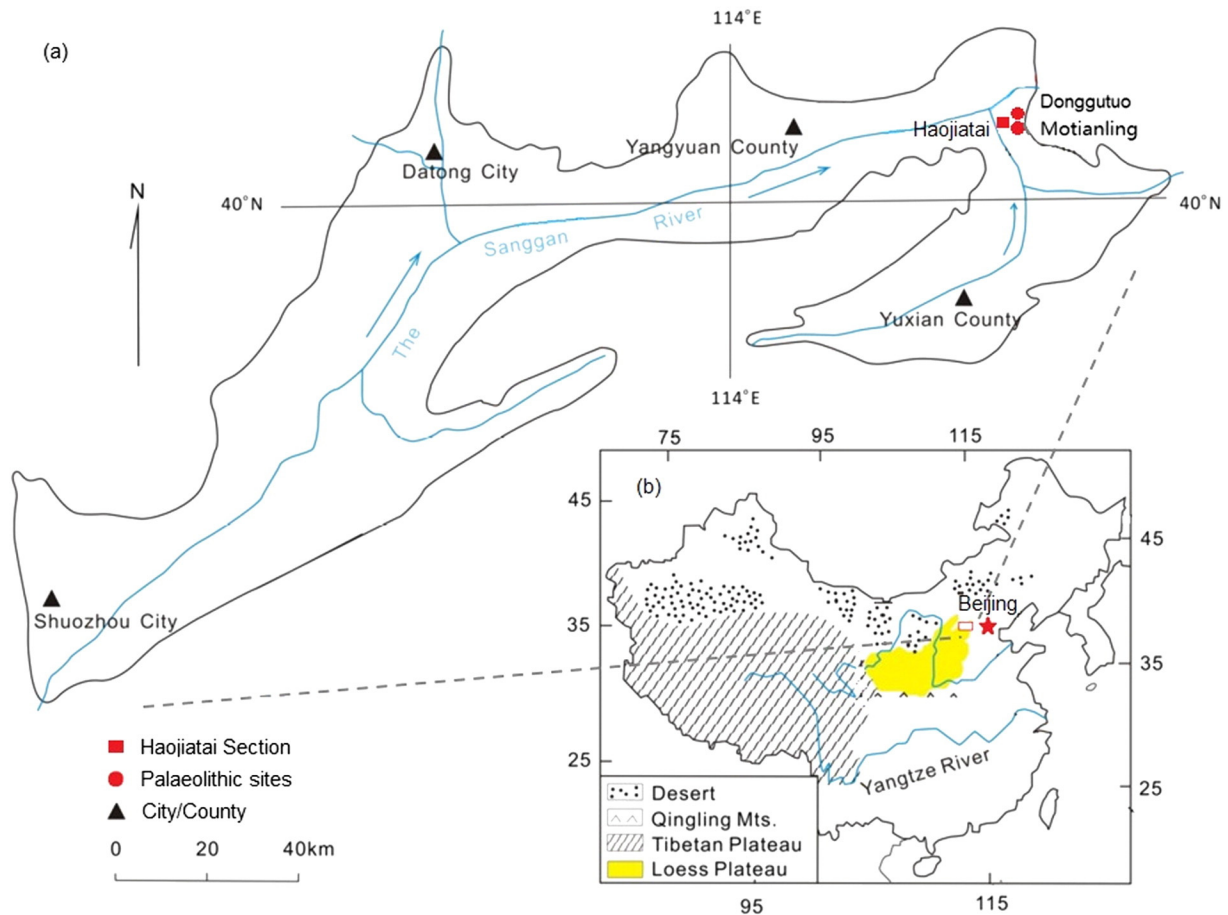
The Nihewan Basin is located at the northeastern edge of the Chinese Loess Plateau, ~150 km west of Beijing, in Hebei and Shanxi Provinces (Fig. 1). It covers an area of ~9000 km<sup>2</sup> and contains thick lacustrine and fluvial sediments of the so-called Nihewan Formation (Yuan et al., 1996; Zhu et al., 2007). The Nihewan Formation and the overlying loess contain abundant mammalian fossils and lithic artefacts, making it one of the most important regions to study the palaeoenvironment,

palaeontology and Palaeolithic archaeology of East Asia (Xie et al., 2006; Dennell, 2009, 2013). Over 100 Palaeolithic sites have been recorded in the Basin, and these have been assigned ages that span the entire Pleistocene, encompassing cultural phases extending from the Lower Palaeolithic to the Upper Palaeolithic (Xie et al., 2006).

Extensive chronological work has been conducted previously to constrain the ages of Early Pleistocene sites in the Basin, based on magnetostratigraphic dating (e.g., Zhu et al., 2004; Wang et al., 2005; Ao et al., 2013). Little work, however, has been done to date younger Pleistocene sites, mainly due to the lack of suitable dating techniques for these sites. Establishing a solid chronological framework for these sites is crucial not only for understanding the evolution of Palaeolithic

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**Fig. 1.** Maps showing the Nihewan Basin and the locations of the two study sites (Donggutuo and Motianling). The Haojiatai section studied by Zhao et al. (2010) is also shown. Modified from Wei (2004) and Ao et al. (2010).

cultures in East Asia, but also to help resolve long-standing debates about the presence or absence of the Middle Palaeolithic in East Asia (Gao, 1999, 2013; Gao and Norton, 2002; Norton et al., 2009; Yee, 2012; Li, 2014). At present, most of the 'Middle' and 'Late' Pleistocene sites in the Nihewan Basin have been classified as such based on the typology of the stone tools and on the stratigraphy of the cultural layers (Xie et al., 2006; Yuan et al., 2011), which are usually imprecise and sometimes controversial.

Radiocarbon ( $^{14}\text{C}$ ) dating is applicable to plant and animal remains up to ~50 ka in age, but suitable organic materials are not always present at archaeological sites, even within this time range. Optical dating can be used to determine when mineral grains, such as quartz and potassium-rich feldspar (K-feldspar), were last exposed to sunlight (Huntley et al., 1985; Aitken, 1998; Roberts and Lian, 2015). The optically stimulated luminescence (OSL) emissions from quartz grains have been widely used to date the time of deposition of sediments at archaeological sites younger than ~200 ka (Lian and Roberts, 2006; Jacobs and Roberts, 2007; Wintle, 2008; Roberts et al., 2015), but there have been few applications to sediments in the Nihewan Basin using the quartz OSL signal (Shitaoka and Nagatomo, 2013; Nian et al., 2014). Zhao et al. (2010) investigated the potential of extending the age range beyond 200 ka using the recuperated OSL (ReOSL) signal from quartz grains extracted from the Nihewan Formation and overlying loess at the nearby (non-archaeological) Haojiatai section (Fig. 1). Their preliminary ages indicated that the Nihewan Formation was deposited before ~270 ka, followed by a hiatus before the loess began to accumulate at ~130 ka.

The infrared stimulated luminescence (IRSL) emissions from K-feldspar saturate at much higher radiation doses than does the conventional quartz OSL signal, so IRSL dating should be applicable to much

older deposits if the problem of age underestimation associated with 'anomalous fading' (Wintle, 1973) can be corrected for appropriately (Huntley and Lamothe, 2001) or avoided altogether. Progress towards the latter goal has been achieved only recently with the development of post-infrared IRSL (pIRIR) procedures. These involve infrared stimulation as a two-step process (Thomsen et al., 2008) or at multiple elevated temperatures (Li and Li, 2011). The latter MET-pIRIR procedure enables non-fading signals to be isolated for dating, and Li and Li (2012) have shown that equivalent dose ( $D_e$ ) values of up to 1000 Gy can be measured reliably, corresponding to maximum ages of 300–500 ka at environmental dose rates of 2–3 Gy/ka. Li et al. (2013b, 2014a) extended this age range by more than half using a novel approach based on their observation that the sensitivity of the MET-pIRIR signal is dependent on the radiation dose received by the sample since burial.  $D_e$  values of up to ~1600 Gy could be measured using this newly developed 'pre-dose' MET-pIRIR (or pMET-pIRIR) procedure, thus bringing the entire Middle Pleistocene – and possibly the final stages of the Early Pleistocene – within dating range.

Although pIRIR and MET-pIRIR methods have been successfully tested on, and applied to, deposits throughout northern China (e.g., Li and Li, 2011, 2012; Buylaert et al., 2012; Fu et al., 2012; Gong et al., 2014), there have been no systematic investigations of the applicability of these procedures to sediments in the Nihewan Basin. Given the fact that the performance of pIRIR procedures is known to be sample dependent and can be affected by various measurement conditions (see recent review by Li et al., 2014b), it is important to test the suitability of these procedures for the samples of interest, and to characterise the luminescence behaviour of the dated K-feldspar grains, before attempting to determine the  $D_e$  values for age calculation.

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