

# Deciphering the chronology of Tepe Sialk (South) “ziggurat”, North Central Iranian Plateau, through Optically Stimulated Luminescence (OSL) dating<sup>i</sup>

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## Abstract

Tepe Sialk of Kashan in the north central Iranian Plateau, is acknowledged as one of the five most important archaeological sites in Iran. It consists of two prominent mounds, Sialk North and Sialk South with cultural layers spanning from the beginning of the 6<sup>th</sup> millennium BCE until the Achaemenid period. Sialk South is crowned by a massive, ziggurat-like, mudbrick platform as one of the major phenomena of the Iron Age of Iran. Considering that Sialk South lacks the plentiful of ages formerly generated for Sialk North, the application of optically stimulated luminescence (OSL) dating was considered necessary to revise the later occupation of Sialk. This project generated a chronological framework for the construction of Sialk South and focus on the large mudbrick platform by dating through OSL. Our results ranged from the Early Chalcolithic period to the Iron Age period of Sialk South. The existence of layers related to Iron Age right underneath the mudbrick platform at Sialk South attracts great interest and seems that Tepe Sialk

could be a key-location in deciphering the chronology of the advancement of the Achaemenid Empire in the central Iran.

**Key-Words:** *Tepe Sialk, Iran, Ziggurat, Neolithic, Chalcolithic, Chalcolithic, Proto-Elamite period, OSL dating*

## 1. Introduction

Tepe Sialk of Kashan located in the north central Iranian Plateau, is considered as one of the most important archaeological sites in Iran (Fig.1). Tepe Sialk of Kashan occupies an extensive cultural span from the beginning of the sixth millennium BCE until the Achaemenid period (Fazeli Nashli et al 2013, Pollard et al 2013). Tepe Sialk consists of two prominent mounds 500 meters apart, namely Sialk North and Sialk South. The most prominent mound is Sialk South crowned by a massive, ziggurat-like, mudbrick platform. Chronologically, Sialk South captures the main periods of Early Chalcolithic, Middle Chalcolithic, Late Chalcolithic, Proto-Elamite to the Iron Age (Nokhandeh, 2010).

A number of  $^{14}\text{C}$  ages generated for both sites (Pollard et al., 2013) under a new investigation project in 2008-2009 run by Prof. Hassan Fazeli Nashli from the University of Tehran, indicate that the beginning of occupation at Tepe Sialk North took place before 5841–5679 cal BCE, but not earlier than the beginning of the 6th millennium BCE. The radiocarbon dates revised previous ideas about the chronology of the site proposed by the first excavators in 1930's which dated Tepe Sialk to the Chalcolithic-Early Bronze Age period. Additionally, the existence of layers related to the proto-Elamite period right underneath the mudbrick platform at Sialk South attracts great interest, as the proto-Elamite period is essential, not only because it bears the earliest evidence for a writing system but also because it is a widespread cultural horizon across Iran (Matthews and Fazeli Nashli 2022).

Given the fact that Sialk South lacks the plentiful of dating formerly generated for Sialk North and charred material suitable for  $^{14}\text{C}$  dating from archaeological layers is less frequent, the application of optically stimulated luminescence (OSL) dating was

considered necessary. This project generated a chronological framework for the construction of Sialk South by dating through OSL mudbricks from the platform and sediments which is very useful for the cultural hiatus of site and also the interpretation of environmental crisis during the Bronze and Iron Age as well.



**Fig. 1** Location of Tepe Sialk in Iran

## 2. Archaeological setting

Scholars have identified six phases of habitation in Tepe Sialk. The first three groups progressed from living in simple huts during the Late Neolithic I to irregularly-shaped mud brick homes and later to rectangular-shaped mud brick homes with arched rooftops, thus illustrating some of the earliest examples of Iranian architecture during the Transitional Chalcolithic/Sialk II period. The fourth wave of residence between 4100 and 2900 BC was marked by significant advancement in pottery and tools, which indicated signs of trade during the Chalcolithic period, the proto-Elamite inscription, and an ability to make bronze. The Proto-Elamite period, also known as Susa III, was dated from c. 3200 BC to 2900/2800 BCE (Fazeli and Matthews 2022). After a long gap the two last phases characterized with the Late Bronze and Iron Ages material culture. The construction of the ziggurat was further proof of the socio-cultural development and the rise of political power during the first half of the first millennium BCE in Iran. The beginning of Iron Age took place at Sialk South around 1200 BCE with the construction of a religious or even political center in Sialk South, representing the significance of the region before the rising of the Achaemenid Empire.

The lack of secure stratigraphic sequences and radiocarbon dating led archaeologists to re-excavate some old sites over the past 20 years, as well as some new ones, in order to propose a new model for cultural changes. Excavation was resumed for several seasons between 1999 and 2004 by a team from Iran's Cultural Heritage Organization led by Sadeq Malek Shahmirzadi called the "Sialk Reconsideration Project" (Shahmirzadi, 2002). Since 2008 an Iranian team led by Hassan Fazeli Nashli and supported by Robin Coningham of the University of Durham have worked at the northern mound finding six Late Neolithic burials, indicating that the beginning of occupation at Tepe Sialk took place before 5841 BCE and ends ca. 4900 cal BCE, but not earlier than the beginning of the 6th millennium BCE (Pollard et al 2013, Sołtysiak et al., 2010).

The total duration of the dated Neolithic and Transitional Chalcolithic occupation of Sialk North is about 1000-900 years which is chronologically comparable with the sites of Ebrahim Abad and Chahar Bone in the Qazvin plain and also numbers of

sites such as Cheshmeh-Ali in the Tehran plain (Fazeli Nashli et al. 2009; Pollard et al. 2012). The beginning of site occupation is unlikely to be earlier than 6000 BCE and the end of dated occupation is modeled at 4900 cal BCE, meaning that the site was abandoned during the earlier phases of the transitional Chalcolithic period. After a hiatus of 800 years of abandonment, occupation resumes at Sialk South 4100 cal BCE (Nokandeh 2010; Pollard et al., 2013). That gap between the occupation of Sialk North and Sialk South has been attributed to cataclysmic environmental events that had an impact on local habitation and led to abandonment (Kourampas and Simpson, 2013, Vidale et al 2018).

Sialk South involves a massive, ziggurat-like, mudbrick platform (Fig.2). Chronologically, the South mound covers the main periods of Early Chalcolithic, Middle Chalcolithic, Late Chalcolithic, proto-Elamite and Iron Age (Shahmirzadi, 2002). The radiocarbon dates from Sialk South show continuity between Early Chalcolithic, Middle Chalcolithic, Late Chalcolithic and the start of the Early Bronze I, but an apparent gap of around 1500 years within the Early Bronze I sequence.



**Fig. 2** View of Tepe the southern Sialk

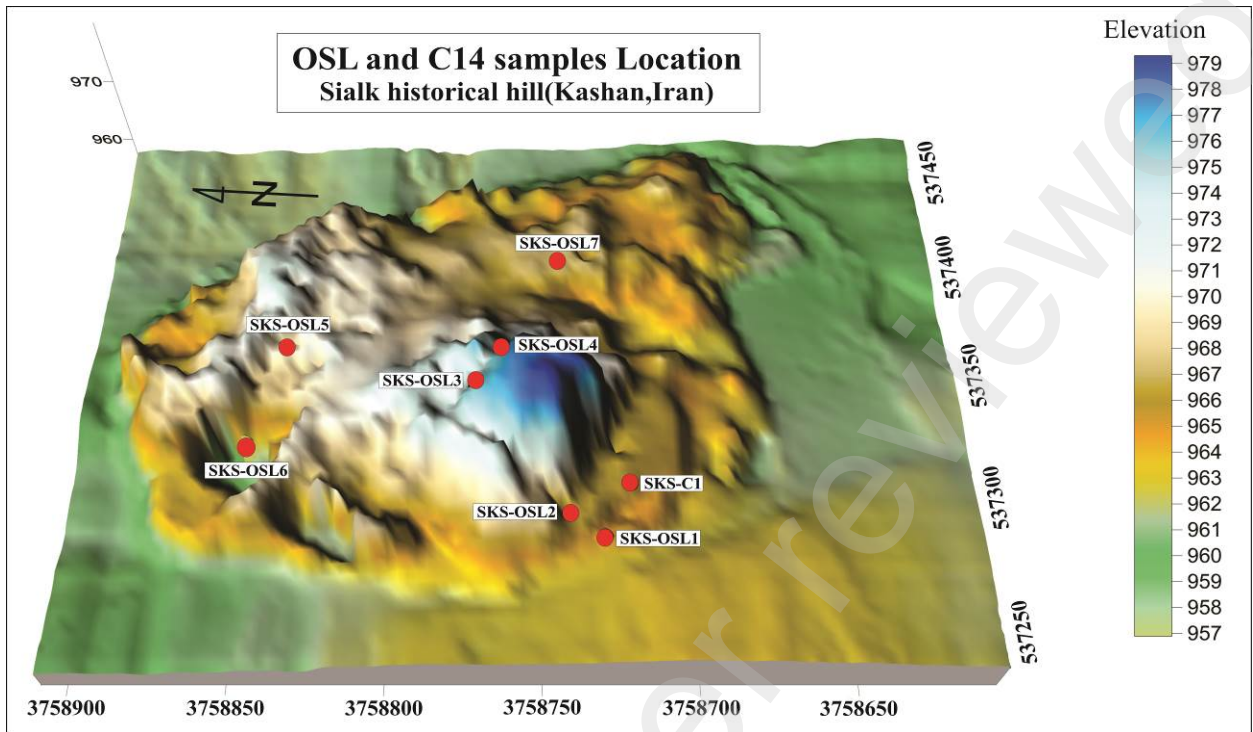
### 3. Material- Methods

Considering to the  $C^{14}$  chronology of Sialk South, we knew that the cultural contexts of the Bronze and Iron Ages was lack of secure absolute chronology in compare with the Chalcolithic periods, therefore it was necessary to do a comprehensive study on comparative chronology of Sialk South, especially of the mudbrick structure, Chalcolithic and Proto-Elamite layers, which was done in this study by using the application of optically stimulated luminescence (OSL) dating.

OSL dates the last exposure to natural light (or heat) of minerals contained in archaeological sediments (Duller, 2008). OSL can also date ancient clay such as mudbricks, baked bricks and pottery (Aitken, 1985), since it expected that the clay's sediment grains were exposed to natural light during manufacturing. The major advantage of OSL is that the optical signal from quartz mineral grains constitutes a natural 'chronometer' when is reset by direct exposure to natural light (or heat). This necessity is met for the majority of sediments and clay artifacts (Athanasas et al. 2014), and thus for the mud bricks of Sialk South and the proto-Elamite layers underneath the mudbrick structure respectively.

Fieldwork and sampling on Sialk South mudbrick platform started with sample collection and in situ measurements. Fig. 3 presents the sampling sites for OSL dating.





**Fig. 3** The sampling sites for OSL dating

Sample collection for OSL dating included extraction of a few grams of clay from the mudbricks, either by drilling into them or by extracting a piece from the brick(s). Samples were also collected from underlying proto-Elamite archaeological layer sequences. Sampling was carried out by introducing light-tight (aluminum) tubes into the excavated soil profile underneath the platform and in case of crumbling sediments, samples were collected at night to avoid the risk of exposing the sediment to sunlight, by delving into the soil profile with a spade and then sealing them in opaque casing (Fig. 4, 5, 6).

We took seven samples of mud bricks and loose sediments with aluminum tubes underneath the mudbrick platform at Sialk South for OSL dating. The sample SKS-OSL1-2018, a mudbrick, was collected under the sunshade, about 40 cm upper the current floor; SKS-OSL2-2018 (mudbrick) sample was collected from the complete tunnel, up side of western entrance of tunnel, at about 100cm upper the current floor; SKS-OSL3-2018 sample was collected from the northern part of a mudbrick structure where painted mudbricks were found- about 80cm upper the current floor; SKS-OSL4-2018 (sediments) sample was collected by an aluminum tube, upper the SKS-OSL3-2018

mudbricks; SKS-OSL5-2018 sample (sediments) was collected by an aluminum tube from the central part of the Ziggurat; SKS-OSL6-2018 sample (sediments) was collected by an aluminum tube from the beginning of the Ziggurat and finally SKS-OSL7-2018 (mudbrick) from the eastern part of the site.

### 3.1 Field sampling

#### *Mudbrick samples*

The first sample was a mudbrick from the wall obtained from the trench of Ghirshman (1937), which was taken from the height of 40 cm of the current floor under the sunshade installed (for the purpose of protection) on the brick wall and coded as SKS-OSL1-2018. After the analysis, the age of this sample is estimated around  $900 \pm 300$  BC. About 11 meters and 70 centimeters east of this sample a charcoal sample taken from the same wall. Considering that this sample and the analyzed charcoal are located in a wall, the date of this wall can be considered around 800 to 900 BCE (Fig.4, 5).



**Fig. 4** OSL sample code SKS-OSL1-2018





**Fig. 5** OSL sample code SKS-OSL1-2018, after sampling

The next mudbrick sample was taken from the roof of the western gate of the complete Ghirshman tunnel in the west of the Silk South ziggurat structure and was recorded with the code SKS-OSL2-2018 (Fig.6). The height of this sample from the tunnel floor was about 100 cm.



**Fig. 6** OSL sample code SKS-OSL2-2018

Another mudbrick sample was taken from the northern part of the Silk South mudbrick structure. This sample was located where the painted mudbricks were obtained. The sample was selected from the original part that kept without thatch cover at the time of conservation, whose height is about 80 cm from the current floor. This sample was registered in the project database with the code SKS-OSL3-2018 (Fig.7).



**Fig. 7** Location of OSL sample code SKS-OSL3-2018

Another sample of mudbrick was selected from the Hamid Fahimi trench in Silk South, whose mudbrick wall is about 20 cm high from the current floor. This sample is from a wall, which according to the excavator belongs to the Iron Age, was registered in the collection with the code SKS-OSL7-2018 (Fig.8).



**Fig. 8** OSL sample code SKS-OSL7-2018



### *Sediment Samples*

During the fieldwork the team found some natural layers either between the archaeological context of the Sialk South or even in the top soil of the Sialk South. Therefore, we have decided to date such sediments in order to know more about the cause of the abandonment date of Sialk South and also to understand the interval gap during the Bronze Age. The first sample in Silk South was selected from the layered sediments formed above the wall from which the mudbrick sample with the code SKS-OSL3-2018 was taken, which is located about 14 meters east of the mudbrick sample. These sediments include 1 to 3 and rarely 5 mm calcites. In this way, the pipe was placed horizontally in the sediments and after packing it was sent to the laboratory. This sample was registered with the code SKS-OSL4-2018 (Fig. 9,10).



**Fig. 9** OSL sample code SKS-OSL4-2018



**Fig. 10** OSL sample code SKS-OSL4-2018

The second sample was sediment formed in the central part of Silk South, 155 cm below the current level of sediments. This sample was registered with the code SKS-OSL5-2018 (Fig.11).



**Fig. 11** OSL sample code SKS-OSL5-2018



The next sample was selected at the depth of the trench of Ghirshman, which indicates the beginning of the occupation in the Silk South. This sample was taken from the northernmost point of the eastern wall of the trench, close to the floor (considering the sediments formed during these years, we tried to choose the lowest point) of the trench and was registered with the code SKS-OSL6-2018 (Fig.12).

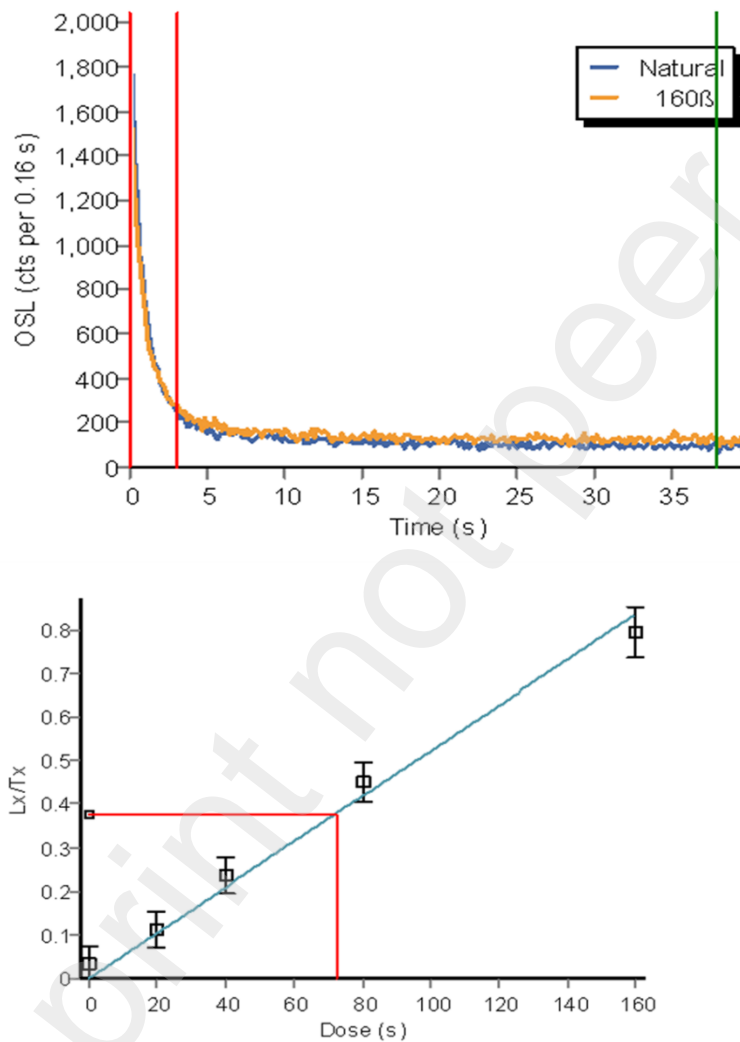


**Fig. 12** OSL sample code SKS-OSL6-2018

### **3.2 Laboratory techniques**

The sample preparation was carried out at the Department of Geology and Geoenvironment, University of Athens, and aimed at eliminate quartz grains from the sampled colluvial. Pure quartz grains were separated according to standard preparation procedures (e.g., Athanassas 2011) that involve treatment with (i) 10% hydrochloric acid to extract calcite minerals; (ii) 20% hydrogen peroxide (to remove any amount of organic matter); (iii) sieving to isolate grains of sizes ranging between 125 and 200  $\mu\text{m}$ ; (iv) density separation using sodium polytungstate (with densities of 2.769 and 2.62  $\text{g cm}^{-3}$ ) to separate quartz grains from the feldspathic content) hydrofluoric acid (40%) etching to remove the alpha-particle influenced outer rim of the quartz grains and (vi) a final rinsing in 10% HCl to remove any residual soluble fluoride salts.

OSL measurements were carried out at the OSL dating laboratory at the Archaeometry Center, University of Ioannina, Greece. Equivalent doses were estimated using the standard single aliquot re-generated (SAR) dose protocol by Murray and Wintle (2000), while for the estimation of the dose rate, measurements of U, Th and K were conducted by ICP-MS and then converted to dose rate units (Gy/ka) using conversion factors by Liritzis et al. (2013). A typical natural OSL signal (decay curve) of one of the samples measured here and its associated growth curve is shown in Fig. 13. Table 1 summarizes the OSL dating results.



**Fig. 13** Typical decay and growth curves of SKS-OSL6 sample

#### 4. Results- Conclusions

The results derived by the application of OSL dating ranged from 6.08 ka- 2.39 ka, thus from the Late Chalcolithic period to Proto-Elamite and Iron Age (Table 1). Our findings agree with the archaeological record, giving evidence for the use of the Sialk South since then. Although it is known that the proto-Elamite period commenced in south-western Iran, little is known about the time-line during the first Millennium BCE with the re-appearing of monumental architecture.

Sample code	Type	Grain size ( $\mu\text{m}$ )	OSL age (ka)
SKS-OSL1-2018	Mudbrick	125-200	2.9 ka $\pm$ 0.34 ka
SKS-OSL2-2018	Mudbrick	125-200	4.44 ka $\pm$ 0.53 ka
SKS-OSL3-2018	Mudbrick	125-200	2.68 ka $\pm$ 0.32 ka
SKS-OSL4-2018	Tube (sediments)	125-200	2.39 ka $\pm$ 0.27 ka
SKS-OSL5-2018	Tube (sediments)	125-200	3.92 ka $\pm$ 0.47 ka
SKS-OSL6-2018	Tube (sediments)	125-200	6.08 ka $\pm$ 0.73 ka
SKS-OSL7-2018	Mudbrick	125-200	5.8 ka $\pm$ 0.7 ka

**Table 1** Summary of the OSL dating results

Our results were also correlated with the available radiocarbon dates (Pollard et al., 2015). The data from Tepe Sialk South consisted of 12 radiocarbon dates, 10 of which come from the stratified sequence of Trench E1. Two are Early Chalcolithic, three are Middle Chalcolithic and five are Early Bronze I. The modelled dates showed continuity during the Chalcolithic period and an apparent gap of around 1000 years within the Early Bronze I.

Specifically, our SKS-OSL1-2018 sample, collected at about 11.70 m to the left of SKS-C1-2018 (sample for radiocarbon dating) had a good correlation with radiocarbon date around 960 BCE. About 11.70 meters east of this sample a charcoal sample was taken from the same wall. Considering that this sample and the analyzed charcoal are located in a wall, the date of this wall can be considered around 800 to 900 BCE.

The chronology of SKS-OSL2-2018 sample above the mudbrick structure at  $2440 \pm 500$  BCE, does not correlate easily with archaeological context. Probably this mismatch is due to the sampling location and the soil required for these mudbricks could come from older deposits.

SKS-OSL3-2018 was dated around  $680 \pm 300$  BC. This result could indicate the possibility of restructure or completing the building in different periods. The sediments from SKS-OSL4-2018 were dated around  $390 \pm 270$  BCE, which probably indicates the period when Sialk south was abandoned permanently.

The dating of SKS-OSL5-2018 was calculated around  $1920 \pm 470$  BC. We are not sure whether such sediments are artificial or natural. Further research is needed to clarify such accumulation of gravels but if the date is correct, it is probably back to the Middle Bronze Age when the central plateau went under a deep climate crisis during the third millennium BCE.

SKS-OSL6-2018 sample was estimated to be around  $4080 \pm 700$  BC, which probably indicates the date of the first occupation there. SKS-OSL7-2018 was dated around  $3600 \pm 700$  BC. The result of this sample unfortunately does not match with the archaeological evidence and the discrepancy will be further explored.

It is also very important to mention that Shahmirzadi (2002) claimed that the mudbrick structure could be dated back to the Proto-Elamite layers but the radiocarbon chronology of the site represent three  $c^{14}$  dates back to 2123, 2071, 1965 BCE (Pollard et al 2013, p. 37). It is also to note that some archaeologists supposed the mudbrick structure back to the first millennium BC (Fahimi 2013, Malekzadeh and Nasery 2013) and therefore the best way of this contradiction was that to date Sialk south for the later period, using other chronological methods, such as OSL dating. Our results from the application of OSL dating reject the use of the mudbrick structure back to the Proto-Elamite period, as supposed by Shahmirzadi (2002).

Additionally, it is worth mentioning the gap of around 1000 years within the Early Bronze I, as testified by radiocarbon dating, which is characterized as a period of abandonment (Pollard et al., 2013). This period belongs to the same horizon of Uruk III in Mesopotamia and similar evidences of abandonment were discovered also at sites such as Hissar, Sofalin, Pardis, Chaltasian, Maral Tepe (Uzbeki) etc in the north central

plateau. This trend of abandonment documented in the centre of the Iranian Plateau continued for about 500-1500 years. Paleoclimate research in western Asia have shown that around 3200 BCE and 2700 BCE a climate change caused a severe drought (Staubwasser and Weiss, 2006: 372, Lawrence et al. 2022). The occurrence of this climate event (called 5.2 ka event) is demonstrated by data obtained from the Soreq Cave (Bar-Matthews et al. 1997) and Kilimanjaro (Thompson et al. 2006). These climate changes threatened life more at the center of the Iranian Plateau than in Mesopotamia and Khuzestan, probably forcing the migration of people from the center of the Iranian plateau to more favourable areas (Shaikh et al., 2016).

It would be worthwhile in a future dating investigation to confirm this habitation gap by the OSL dating method, as it could give us very important information for the precise determination of the chronological framework of this palaeo-environmental event which affected the Near East. The three sediment dates (SKS-OSL4-2018, SKS-OSL5-2018 and SKS-OSL6-2018) of this project is very significant which not only support the climate events (4.2. ka events) but also address when the Sialk South was abandoned around 400 BCE during the Achaemenid period. The current evidence not only encourage to study carefully the sediments accumulated in the upper parts of Sialk in order to verify the environmental crisis during the third and second millennium BCE, but also, it is very significant to date the collapse of Sialk during the Iron Age IV. SKS-OSL6-2018 date supports the earlier dating of Sialk (Pollard et al 2013) at the beginning of Sialk occupation, dating back to the late fourth millennium BCE.

In conclusion, this project aimed to generate a chronological framework for the construction of Sialk South mudbrick structure ziggurat through OSL mud bricks back to the first millennium BCE. It is the first attempt to date the site using the OSL dating, apart from other methodologies, such as radiocarbon dating. More OSL dating results are needed, as it seems that Tepe Sialk could be a key-location in deciphering the chronology of the advancement of the Achaemenid chiefdoms in the central Iran.



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