



Luminescence Dating of Relics in Ancient Cities Provides Absolute Dates for Understanding Human-Land Relationships in Qinghai Lake Basin, Northeastern Tibetan Plateau

Manping Sun^{1,2}, Yongjuan Sun^{1,2,3*}, Haicheng Wei⁴, Guangliang Hou^{1,2,3}, Ji Xianba^{1,2}, Liqian Xie^{1,2}, Yongxin Zeng^{1,2} and Chongyi E^{1,2,3*}

¹Qinghai Provincial Key Laboratory of Physical Geography and Environmental Processes, School of Geographical Science, Qinghai Normal University, Xining, China, ²MOE Key Laboratory of Tibetan Plateau Land Surface Processes and Ecological Conservation, Qinghai Normal University, Xining, China, ³Academy of Plateau Science and Sustainability, People's Government of Qinghai Province and Beijing Normal University, Xining, China, ⁴Key Laboratory of Comprehensive and Highly Efficient Utilization of Salt Lake Resources, Qinghai Institute of Salt Lakes, Chinese Academy of Sciences, Xining, China

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*Correspondence:

Yongjuan Sun
yongjuansun@163.com
Chongyi E
echongyi@163.com

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The study of ancient city sites provides valuable evidence for understanding human-land relationships. Qinghai Lake Basin, on the northeastern Tibetan Plateau, was a key location for economic and cultural exchanges between East and West in ancient China and archaeological surveys have identified the remains of many ancient cities. Although there are relatively good historical records for some ancient cities, their absolute ages are still unclear due to a lack of systematic chronological dating. In this study, OSL dating of ceramic and tile remains from three ancient cities in Qinghai Lake Basin, Xihaijun (XHJGC), Beixiangyang (BXYGC), and Fuxi (FSC), was combined with documentary and paleoclimate evidence to investigate historical human-land relationships. Relics from XHJGC and BXYGC were dated to 0–220 AD, in the Han Dynasty, while tiles from FSC were dated to 120–520 AD, largely corresponding to the Wei Jin Southern and Northern Dynasties. Luminescence ages were generally consistent with dates recorded in historical documents, indicating that the OSL method can be reliably used to date buried tiles in ancient cities on the northeastern Tibetan Plateau. Comparing the dates with paleoclimatic records suggests that the warm and humid climate at c. 2 ka was an important driver of immigration to the region that led to the construction of cities in the Qinghai Lake area during the late Western Han Dynasty. During the Wei Jin Southern and Northern Dynasties (220–589 AD), communication between East and the West flourished, and human activities in the area were strong with the continuation of the war in Central China and Hexi Corridor. Fuxi was largely abandoned in the later Wei Jin Southern and Northern Dynasties, although it was still used intermittently until the Ming Dynasty. Pollen records confirm that humans were extensively engaged in agricultural production in the Qinghai Lake area during the Wei Jin Southern and Northern Dynasties.

Keywords: optical stimulated luminescence dating, human-land relationship, tiles, ancient cities, Qinghai Lake Basin

INTRODUCTION

It has been argued that cities are the product of a certain stage in the development of human society, marking the end of primitive society and the arrival of civilization (Bureau of National Cultural Relics, 1996). The study of ancient cities can reveal the political, military, economic, and cultural conditions of a region, which is fundamental for understanding the historical development and evolution of human-land relationships (Li, 1995). On the Tibetan Plateau, northeastern Qinghai Province was an important boundary area between nomadic tribes and agricultural regimes during historical periods (Dong et al., 2016). Many wars between ancient agricultural and pastoral regimes in China have been related to climate change, with deterioration of the ecological environment during periods of unfavorable climate conditions leading to social instability (Dong et al., 2016). Frequent wars may have stimulated the construction of a large number of ancient cities on the northeastern Qinghai-Tibet Plateau; Dong et al. (2016) argue that most were initiated for military defense purposes. Documentary evidence and radiocarbon dating suggest that most of the cities were built or repaired during the Han Dynasty (202 BC–220 AD), Tang Dynasty (618–907 AD), the Five Dynasties and Ten Kingdoms period (907–960 AD), the Song Dynasty (960–1279 AD), and the Ming Dynasty (1368–1644 AD) (Dong et al., 2016).

Qinghai Lake is a saline lake in the northeastern Tibetan Plateau; it is the largest water body on the plateau and supports a vast inland wetland ecosystem. Historically, Qinghai Lake Basin was important for cultural interaction and influence between the Yellow River and the Central Asian civilizations (Huo, 2017), and it has been a key research area for studies exploring cultural collision and economic exchange between East and West. Most studies of human activities in the Qinghai Lake area have focused on the prehistoric period (Brantingham and Xing, 2006; Rhode et al., 2007; Sun et al., 2012; Chen et al., 2015; ChongYi et al., 2015; Hou et al., 2015; Sun et al., 2018), with few looking at the relationship between human activities and environmental changes in the historical period. Archaeological investigations have found that ancient cities around Qinghai Lake were first built in the late Western Han Dynasty (c. 4 AD) (Li, 1995; Bureau of National Cultural Relics, 1996). Contact between the agricultural regimes of the Central Plains government and the nomadic tribes of the northeastern Tibetan Plateau also became closer from the Western Han Dynasty period, and the Qinghai Lake area was an important military frontier. As the power of the Central Plains government gradually expanded to the plateau, the number of cities increased and agricultural technology and production tools were also introduced to the Qinghai Lake Basin. Some nomadic tribes in the Qinghai Lake Basin also began to build cities and engage in farming activities. The Qinghai Road, one of the trunk roads of the “Overland Silk Road” (Wang and Zhao, 2013; Huo, 2017), also provided a means for economic and cultural exchange in the region. Thus, military, political, and economic developments all contributed to the formation and development of ancient cities on the northeastern Tibetan Plateau; reconstructing the chronological development of these cities furthers understanding of human

activities and provides important evidence relating to the evolution of human-land relationships in the historical period.

The chronological framework for ancient cities in China is mainly based on historical documents, types, and the characteristics of cultural relics, and ^{14}C dating of charcoal and bone. However, the Qinghai Lake area is relatively remote and was under the alternate control of farming and nomadic regimes for a long time, so it lacks historical documents with good records. Moreover, most ancient cities were built with rammed earth, and other buildings had walls of sand, stone, and soil, which are vulnerable to erosion and leave little evidence for the historical record. The development of the thermoluminescence technique in the 1950s offered a means for direct dating ancient pottery (Daniels et al., 1953), and the more recent OSL has widened applications to a range of Quaternary sediments and archaeological materials. Several studies have shown the accuracy and reliability of OSL dating of ceramics from archaeological sites on the Tibetan Plateau (Rhode et al., 2007; Sun, 2019; Sun et al., 2021). Tiles are made in a similar way to ceramics, in which the luminescence signals accumulated by the raw materials over geological periods are completely bleached during firing, so the absolute age can be determined using luminescence dating.

In this study, we applied luminescence dating to tiles and ceramics from three ancient cities in Qinghai Lake Basin, Xihaijun (XHJGC), Beixiangyang (BXYGC), and Fuxi (FSC). The cities were selected due to their good preservation, large area, and suitable dating materials. Historical documents show that the three cities served as royal cities or prefectures in the historical period and played an important role in the ancient Silk Road. Although the ages of the cities can be inferred from historical documents and archaeological studies, their occupation chronology is still controversial due to the relatively long history the lack of direct dating. Dong et al. (2016) determined the ^{14}C age of charred sheep droppings in the cultural layer at Fuxi as the Ming Dynasty. However, the large area of the cities means that they were likely multiphased, so it is necessary to use a variety of methods to systematically date different cultural remains. In this study, the OSL dating of tiles and ceramics gives the direct age of cultural artifacts in the three ancient cities and provides a basis for assessing the age of other ancient cities in the area that have similar relics but lack historical records. Combined with historical documents and paleoclimate records, the dating results indicate the historical evolution of human-land relationships in the Qinghai Lake Basin.

STUDY AREA AND MATERIALS

Study Area

Qinghai Lake is located on the northeastern edge of the Tibetan Plateau and is the largest inland saltwater lake in China. The lake basin is characterized by plateau continental climate and is in the region of interaction between the East Asian monsoon and westerlies, which is sensitive to climate change. Qinghai Lake Basin is a closed inland basin and comprises a flat alluvial plain with multilevel shoreline terraces. Vegetation is mainly alpine meadow and alpine shrub.

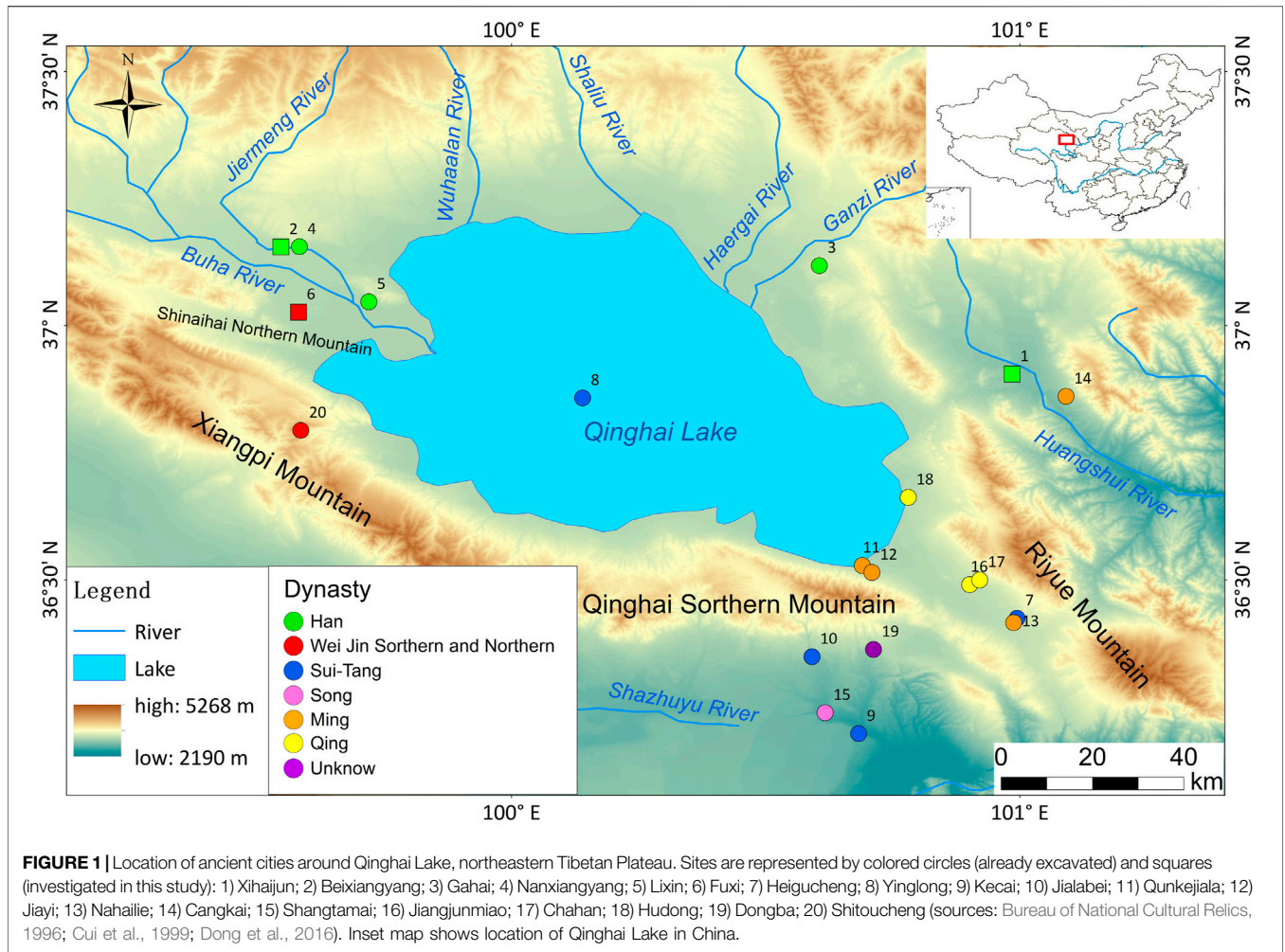


FIGURE 1 | Location of ancient cities around Qinghai Lake, northeastern Tibetan Plateau. Sites are represented by colored circles (already excavated) and squares (investigated in this study): 1) Xihaijun; 2) Beixiangyang; 3) Gahai; 4) Nanxiangyang; 5) Lixin; 6) Fuxi; 7) Heigucheng; 8) Yinglong; 9) Kecai; 10) Jialabei; 11) Qunkejiala; 12) Jiayi; 13) Nahaille; 14) Cangkai; 15) Shangtamai; 16) Jiangjunmiao; 17) Chahan; 18) Hudong; 19) Dongba; 20) Shitoucheng (sources: Bureau of National Cultural Relics, 1996; Cui et al., 1999; Dong et al., 2016). Inset map shows location of Qinghai Lake in China.

TABLE 1 | Area and the dynastic era of ancient cities around Qinghai Lake, northeastern Tibetan Plateau (sources: Bureau of National Cultural Relics, 1996; Li, 1995). See **Figure 1** for locations.

No.	Ancient city	Area (m ²)	Dynasty	No.	Ancient city	Area (m ²)	Dynasty
1	Xihaijun	180,000	Han	11	Jialabei	48,741	Sui-Tang
2	Beixiangyang	120,000	Han	12	Shangtamai	28,500	Song
3	Gahai	189,660	Han	13	Qunkejiala	26,250	Ming
4	Nanxiangyang	10,800	Han	14	Jiayi	-	Ming
5	Lixin	3,000	Han	15	Nahaille	28,224	Ming
6	Fuxi	40,000	Wei Jin Southern and Northern	16	Cangkai	-	Ming
7	Shitou	-	Wei Jin Southern and Northern	17	Jiangjunmiao	27,225	Qing
8	Heigucheng	18,750	Sui-Tang	18	Chahan	123,000	Qing
9	Yinglongcheng	21,210	Sui-Tang	19	Hudong	13,225	Qing
10	Kecaichengzhi	-	Sui-Tang	20	Dongba	49,600	-

Archaeological investigations have shown that the construction of ancient cities in the Qinghai Lake area began in the late Western Han Dynasty (4 AD) (Li, 1995; Bureau of National Cultural Relics, 1996). Many of the ancient cities are not well preserved, due to the length of time since the occupation, strong aeolian activity, and severe trampling by yak and sheep as part of nomadic pastoralism (**Figure 1**). **Table 1** details the area of

ancient cities in the Qinghai Lake region, which range in age from the Western Han Dynasty to the Qing Dynasty. It is difficult to collect representative relics from most cities relative to present day pasture cover. For our study, we selected three cities, XHJGC, BXYGC, and FSC, that are relatively well preserved, are large in area, and have numerous tiles exposed on the surface (**Figure 2**). Details of what is known of the age and occupation of the three

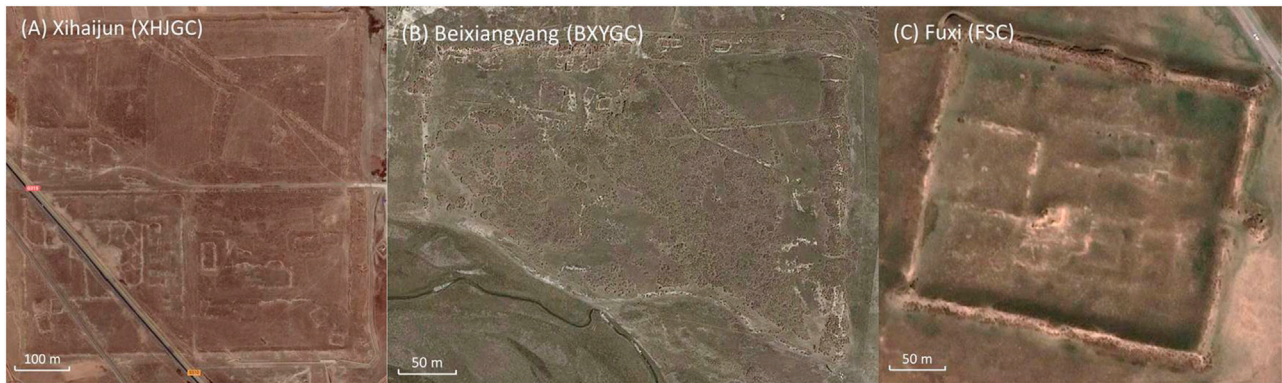


FIGURE 2 | Remote sensing images showing an aerial view of the three ancient cities investigated in this study. **(A)** Xihaijun; **(B)** Beixiangyang; **(C)** Fuxi. See **Figure 1** for locations.

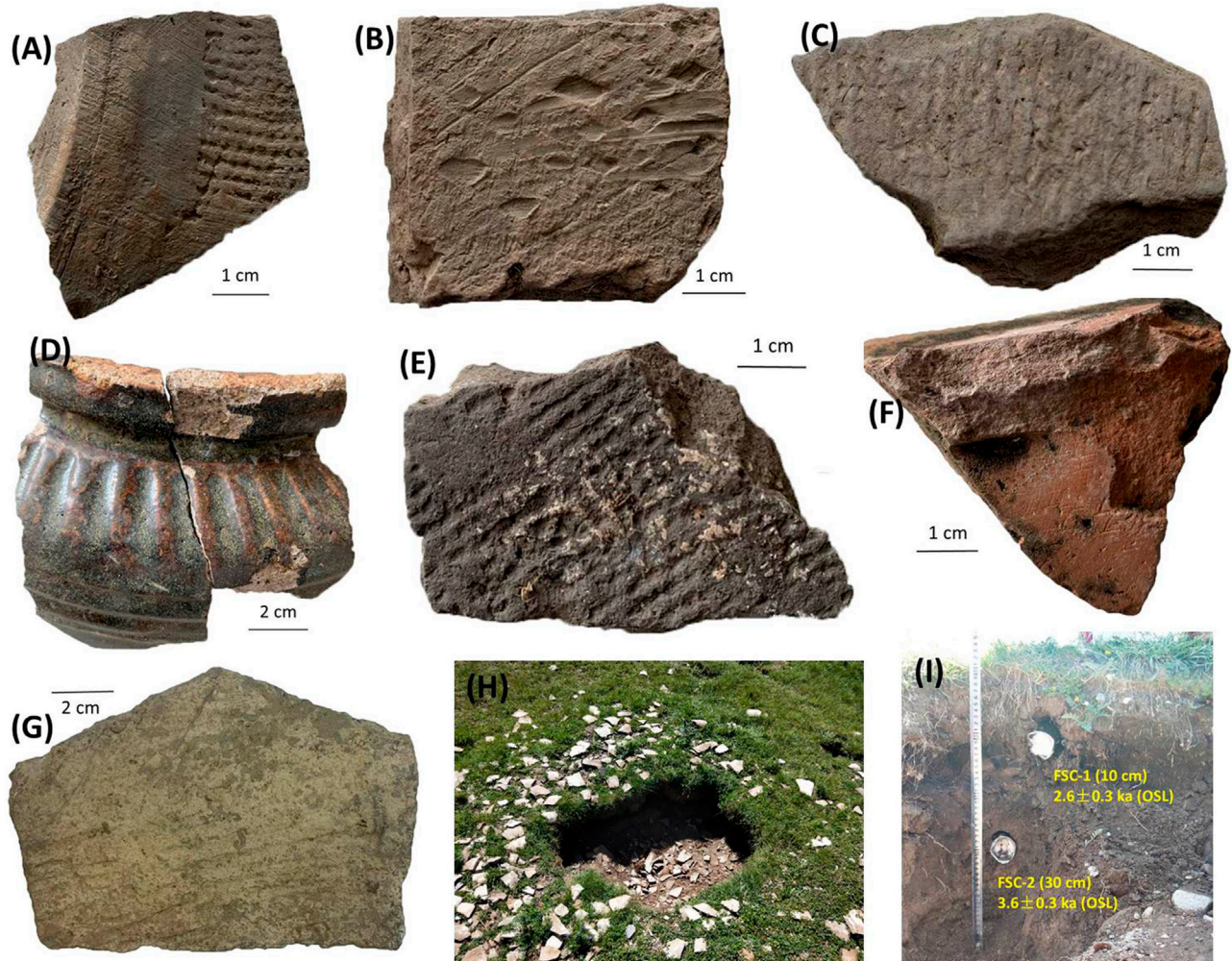


FIGURE 3 | Tiles and ceramics used for OSL dating in this study. **(A–C)** Gray muddy tiles exposed on the ground at Xihaijun. **(D–F)** Tiles and sand-filled red ceramic from Beixiangyang. **(G,H)** Gray-white sand-filled corded tiles found scattered on the ground at Fuxi. **(I)** Subsurface soil samples collected for OSL dating at Fuxi.

cities from the archaeological and documentary record are given below, along with details of samples collected in our fieldwork.

XHJGC (100° 58' 53", 36° 54' 20", 3,022 m) was built by emperor Wang Mang in 4 AD of the Xin Dynasty (9–23 AD), at the end of the Han Dynasty), and was the county capital of Xihai Jun, an administrative district in Chinese history (Cui et al., 1999). The city lies to the east of Qinghai Lake, on the south bank of Huangshui River, and is trapezoidal in shape with sides of 600–650 m. Archaeological finds include a “Xihaijun Tiger Runestone Cabinet,” Kayue cultural sandy coarse pottery, tiles, “Wuzhu coins,” and other currencies of the Western Han Dynasty and Wang Mang periods, a Wadang (traditional embossed roof tile end) with lotus pattern of the Tang Dynasty, and currencies of the Song Dynasty (Li, 1995). For OSL dating, we collected three gray muddy tiles that were exposed on the ground, approximately 1 cm thick, and decorated with rope patterns (Figures 3A–C, samples XHJGC-1, 2, 3).

BXYGC (99° 32' 42", 37° 09' 9", 3,242 m) was one of five county towns of Xihai Jun in the Xin Dynasty, located on the Buha River alluvial plain northwest of Qinghai Lake. The city is rectangular in shape, with east-west walls extending c. 400 m. The southern part of the ancient city was destroyed by a tributary of Buha River, and aeolian deposits have covered much of the area. Remnant walls in the middle and south of the city may be ancient houses, and there is an abandoned modern village on the northwest side. Numerous gray muddy tiles, sand-filled red ceramics, and brown enamel tiles are scattered throughout the city (Figures 3D–F) and probably represent more than one period. Two tiles and one ceramic were selected for OSL dating (samples BXYGC-1, 2, 3).

FSC (99° 34' 52", 37° 01' 32", 3,224 m) was the capital of Tuyuhun, a dynastic kingdom of nomadic peoples. Based on historical documents, Huang and Fang (1962) speculated that the city was founded no earlier than 481–491AD. The city is located about 7.5 km west of Qinghai Lake, with Shinaihai Northern Mountain to the south, on the alluvial fan plain of Cheji River (a tributary of Buha River). The main city is roughly within a 200 m square, with a gate about 10 m wide in the middle of the east wall, and an inner city. An outer fortress, located outside the main city, has suffered severe erosion and the northern part has been washed out by the Cheji River; no remains have been found in the outer city. Numerous gray-white sand-filled corded tiles were found on the north side of the main city in an excavated pit and exposed on the surface (Figures 3G,H). Four tiles were selected for OSL dating (samples FSC-1, 2, 3, 4), along with two soil samples at 10 and 30 cm depth (Figure 3I).

OSL Sample Preparation

Sample preparation was carried out in a dark room under red light. The same pretreatment procedure was used for tiles and ceramics (Sun et al., 2021). The top 2–3 mm was removed from the surface of the sample to eliminate the effect of sunlight exposure. A vice was used to crush the inner part of the sample, which was then gently ground in an agate mortar, and coarse particles of 90–154 μm were screened out. For sediment samples, c. 3 cm was removed from both ends of the sample tube

in case of exposure, and the 63–90 μm fraction of the middle section was screened out using the wet screening method. For all samples, the screened coarse particles were soaked with 10% HCl and 30% H₂O₂ to remove carbonate and organic matter, and then 10% HF was added for etching for 40 min to remove impurities such as heavy minerals. Heavy liquid (2.58 g/cm³) was used to separate feldspar and quartz, and the extracted quartz particles were added to 40% HF for etching for 40 min to remove surface α influence and impurities. Samples were rinsed repeatedly with pure water to neutral after each step. Finally, quartz purity was detected by infrared excitation for 40 s at 125°C followed by blue excitation for 40 s at 125°C. If OSL/IRSL < 95%, HF etching was repeated until OSL/IRSL > 95%.

OSL Measurement Methods and Instruments

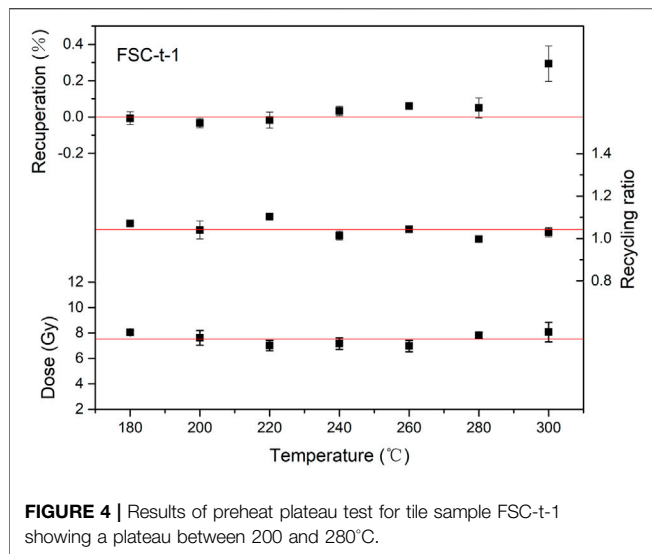
Equivalent doses (D_e) were determined by the single-aliquot regenerative-dose (SAR) protocol (Murray and Wintle, 2000; Wintle and Murray, 2006). For D_e calculations, the initial 0–0.32 s of the OSL signal minus an early background signal of the following 0.32–0.64 s was selected (Ballarini et al., 2007; Cunningham and Wallinga, 2010). We measure 5–18 aliquots for each sample for D_e calculation. The number of aliquots for tile and ceramic samples was relatively low due to the small sample size available.

The dose rate was calculated by measuring the radioactivity of the tile/ceramic and the soil samples. Radioactivity comprises three elements: 1) beta dose from the tile/ceramic; 2) gamma dose from soil surrounding the artifact; and 3) a minor contribution from cosmic rays (Thomas et al., 2008). The concentration of U, Th, and K in tiles and sediments was obtained by inductively coupled plasma mass spectrometry (ICP-MS) and then converted into beta and gamma doses using conversion parameters (Guérin et al., 2012). The range of gamma rays in soil and tile/ceramic is very long (20–40 cm), which is much larger than the thickness of the tile/ceramic, so the internal gamma radiation can be ignored (Wang, 2008). In ceramics, the beta dose provided by the environment is absorbed by 90% at 2 mm depth from the surface; as we removed the top 2–3 mm of our ceramic samples, the beta dose provided by the soil can be ignored (Wang, 1993; 2008). We assumed a homogenous surrounding environment when calculating the dose rate for buried tiles. For those exposed at the surface, we used the gamma dose of the underlying soil to calculate the environmental dose rate. Since the air above the tiles/ceramic does not contribute to the gamma radiation, but we do not know how long the tiles/ceramic is exposed on the surface, half of the gamma dose and the entire gamma dose in the sediment underlying the tiles/ceramic were used to calculate the dose rates, respectively. The cosmic ray dose rate is calculated using the depth, altitude, and geographic coordinates of each sample (Prescott and Hutton, 1994). In this study, a value of 0.95 Gy/ka was used. The depth for estimating the cosmic ray dose rate is the sampling depth (Table 2). The altitude and geographic coordinates are based on those of the ancient city. The water content of the two paleosol samples in FSC was estimated with reference to a previous study

TABLE 2 | Characteristics of OSL samples from ancient cities around Qinghai Lake, northeastern Tibetan Plateau, analyzed in this study.

Sample ID	Depth (cm)	Sample type	Thickness (mm)	Size (cm)	Decoration	U (ppm)	Th (ppm)	K (%)	Beta (Gy/ka)	Gamma (Gy/ka)	Gamma ^a (Gy/ka)	Cosmic ray dose rate (Gy/ka)	Water content (%)	Dose rate (Gy/ka)	Dose rate ^a (Gy/ka)	Disc	D _e (Gy)	Age ^a (ka)	Age (ka)	Dynasty
XHJGC-1	0	Tile	14	3 × 4	Rope	5.00 ± 0.50	15.55 ± 0.80	3.19 ± 0.04	3.40 ± 0.05	1.70 ± 0.005		-	5 ± 5	-	-	-	-	-	-	-
XHJGC-2	0	Tile	15	3.5 × 4.5	Diamond	2.24 ± 0.30	14.59 ± 0.80	3.25 ± 0.04	3.29 ± 0.04	1.69 ± 0.003		-	5 ± 5	-	-	-	-	-	-	-
XHJGC-3	0	Tile	14	3.5 × 5.5	Rope	2.78 ± 0.40	14.51 ± 0.80	2.89 ± 0.04	3.04 ± 0.04	1.60 ± 0.004		-	5 ± 5	4.42 ± 0.18	3.77 ± 0.16	5	9.06 ± 1.22	2.4 ± 0.3	2.1 ± 0.3	Han
XHJGC-DB	0	Sandy loess	-	-	-	3.48 ± 0.40	11.97 ± 0.70	2.15 ± 0.04	2.46 ± 0.04	1.37 ± 0.004	0.69 ± 0.002	0.95	-	-	-	-	-	-	-	-
BXYGC-1	0	Tile	12	3.5 × 5	Brown enamel	4.00 ± 0.50	21.15 ± 1.00	1.92 ± 0.04	2.41 ± 0.05	1.48 ± 0.005		-	5 ± 5	-	-	-	-	-	-	-
BXYGC-2	0	Tile	14	3.5 × 6	Rope	3.00 ± 0.40	16.55 ± 0.90	3.36 ± 0.04	3.44 ± 0.04	1.76 ± 0.004		-	5 ± 5	4.61 ± 0.19	4.06 ± 0.17	6	7.95 ± 0.14	2.0 ± 0.1	1.7 ± 0.1	Han
BXYGC-3	0	Ceramic	17	4 × 5/2	None	2.59 ± 0.40	12.50 ± 0.70	2.45 ± 0.04	2.66 ± 0.04	1.46 ± 0.004		-	5 ± 5	3.93 ± 0.16	3.38 ± 0.14	6	7.41 ± 0.10	2.2 ± 0.1	1.9 ± 0.1	Han
BXYGC-DB	0	Aeolian sand	-	-	-	1.72 ± 0.30	8.85 ± 0.60	1.52 ± 0.04	1.83 ± 0.04	1.16 ± 0.004	0.58 ± 0.002	0.95	-	-	-	-	-	-	-	-
FSC-t-1	0	Tile	18	11 × 9	None	2.91 ± 0.40	14.96 ± 0.80	2.48 ± 0.04	2.55 ± 0.04	1.47 ± 0.004		-	5 ± 5	4.01 ± 0.17	-	18	7.83 ± 1.58	2.3 ± 0.5	2.0 ± 0.4	Wei Jin Southern and Northern
FSC-sed-1	0	Paleosol	-	-	-	1.93 ± 0.30	9.21 ± 0.60	1.69 ± 0.04	1.99 ± 0.04	1.21 ± 0.003	0.60 ± 0.002	0.95	-	-	-	-	-	-	-	-
FSC-t-2	10	Tile	18	12 × 8	None	2.58 ± 0.40	14.08 ± 0.80	2.36 ± 0.04	2.59 ± 0.04	1.46 ± 0.004		-	5 ± 5	3.91 ± 0.16	-	18	4.89 ± 0.54	-	1.3 ± 0.2	Wei Jin Southern and Northern
FSC-t-3	10	Tile	18	11 × 8	None	2.76 ± 0.40	16.00 ± 0.90	2.27 ± 0.04	2.55 ± 0.04	1.46 ± 0.004		-	5 ± 5	3.87 ± 0.16	-	18	7.56 ± 0.31	-	2.0 ± 0.1	Han
FSC-t-4	20	Tile	18	12 × 8	None	2.75 ± 0.40	14.83 ± 0.80	2.28 ± 0.04	2.72 ± 0.04	1.51 ± 0.004		-	5 ± 5	3.82 ± 0.16	-	16	6.53 ± 0.47	-	1.7 ± 0.2	Wei Jin Southern and Northern
FSC-sed-3	10	Paleosol	-	-	-	2.14 ± 0.30	1.41 ± 0.70	1.71 ± 0.04	2.03 ± 0.04	1.25 ± 0.003		0.95	-	-	-	-	-	-	-	-
FSC-1	10	Paleosol	-	-	-	1.85 ± 0.40	10.26 ± 0.70	1.61 ± 0.04	1.92 ± 0.04	1.20 ± 0.006		-	7 ± 5	3.00 ± 0.13	-	18	7.76 ± 0.69	-	2.6 ± 0.3	-
FSC-2	30	Paleosol	-	-	-	1.84 ± 0.40	11.10 ± 0.70	1.69 ± 0.04	1.99 ± 0.04	1.24 ± 0.006		-	7 ± 5	3.07 ± 0.14	-	18	10.93 ± 0.75	-	3.6 ± 0.3	-

^aIndicates the results calculated using half of the gamma dose in the sediments on the surface.



in Qinghai Lake Basin as $7 \pm 5\%$ (Liu et al., 2012), and the tile water content was estimated as $5 \pm 5\%$.

Pretreatment of tiles and stratigraphic samples and determination of D_e were carried out in the Laboratory of Luminescence Chronology at Qinghai Provincial Key Laboratory of Physical Geography and Environmental Processes using a standard Risø TL/OSL DA-20 reader with a $^{90}\text{Sr}/^{90}\text{Y}$ β radiation source. The environmental dose rate was measured at Xi'an Geological Survey Center.

RESULTS

Luminescence Characteristics

A preheat plateau test was carried out on tile sample FSC-t-1 to select the appropriate preheating temperature for D_e measurements (Figure 4). Three aliquots were measured at each temperature. There was a clear plateau between 200 and 280°C, the recycling ratio was between 0.9 and 1.1, and recuperation was stabilized at 0–1%. Based on this, a routine preheat temperature of 260°C and cut-heat temperature of 220°C were employed for all D_e measurements. Typical growth and decay curves for soil, surface exposed tile, and buried tile samples at FSC are shown in Figures 5A–C. The luminescence signal of FSC samples shows characteristics of fast composition, decaying to background levels within 2 s. Decay and growth curves for surface exposed ceramic at BXYGC (Figure 5D) show a strong response, with a bright signal that attained 30,000 counts and a perfect growth curve.

Dating Results

Radionuclide concentration and beta and gamma dose rate for tiles, ceramic, and soil samples are given in Table 2, along with U, Th, and K concentrations and other characteristics. U, Th, and K concentrations in tiles and ceramics are relatively concentrated, ranging from 2.24 to 5.00 ppm, 12.50–21.15 ppm, and 1.92–3.36%, respectively. The dose rate ranges between 3.00 and 4.58 Gy/ka. For the calculation of the dose rate of all tiles/

ceramic, the inner beta dose from tiles/ceramic itself and the outer gamma dose from the sediment surrounding the artifact were used. In XHJGC and BXYGC, the outer gamma doses from XHJGC-DB and BXYGC-DB were used to calculate the dose rate, respectively; in FSC, the gamma dose from FSC-sed-1 was used to calculate the dose rate of FSC-t-1, and the gamma dose from FSC-sed-2 was used to calculate the dose rate of FSC-t-2, 3, and 4. For the sediment samples (FSC-1 and FSC-2), the calculation of dose rate used its own U, Th, and K (Table 2). For the tiles/ceramic on the surface, the ages calculated using half of the gamma dose rates are older, and the ages calculated using the entire gamma doses are closer to the ages of the cities recorded in the historical documents (Table 2). Since it is not clear how long the tiles/ceramic are exposed on the surface, we assume that the tiles/ceramic are not exposed for a long time. Finally, the entire gamma doses were used to determine the age of the tiles/ceramic on the surface.

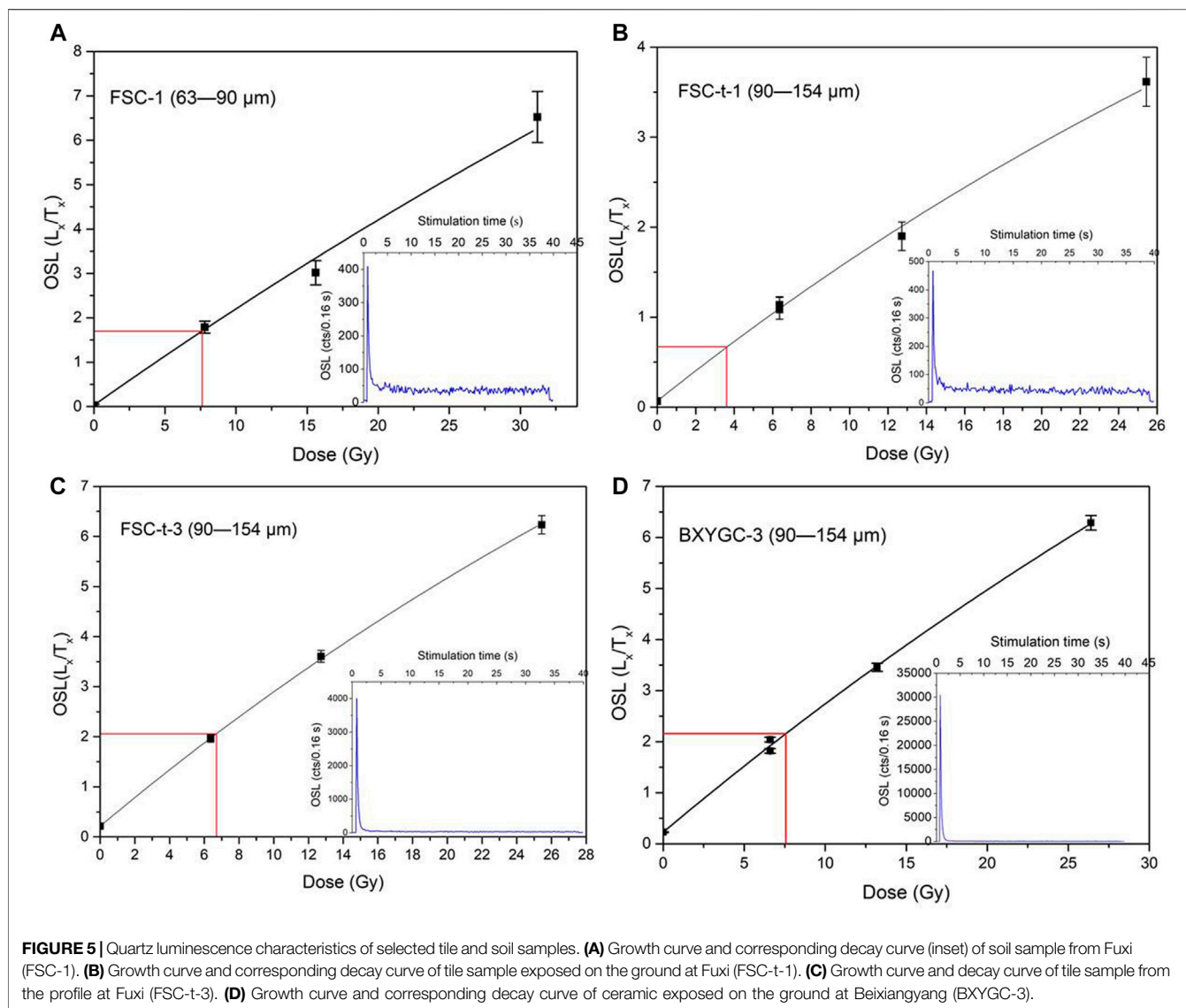
The OSL age results (Table 2) show dates of 0–220 AD for XHJGC and 20–220 AD for BXYGC, both of which are in the Han Dynasty. Tiles at FSC were dated between 120 and 520 AD, mostly in the Wei Jin Southern and Northern Dynasties. The age of the surface exposed tile at FSC (FSC-t-1) is 20–420 AD, which is within the error range of buried tiles; this suggests that surface tiles at Fuxi are less affected by sun bleaching and may not have been exposed at the surface for a long time. The luminescence ages of paleosol samples at FSC are 2.6 ± 0.3 ka at 10 cm depth and 3.6 ± 0.3 ka at 30 cm depth, which is much earlier than the construction of Fuxi suggested by the tile dates. This indicates the city wall was built on the surface, the ground was not rammed, and the use of the city site did not disturb the soil below 10 cm depth.

DISCUSSION

Age of Construction and Occupation of Ancient Cities in Qinghai Lake Basin

The gray muddy corded tiles we collected at XHJGC and BXYGC have similar typological characteristics, so likely originated from the same period, and this is confirmed by the OSL results that show tiles and ceramics from XHJGC and BXYGC date to 0–220 AD. The dates indicate the cities were built in the Xin Dynasty, which coincides with documentary evidence for the establishment of Xihai Jun (Cui et al., 1999). Based on the similarity of construction systems and artifacts, the three ancient cities identified in archaeological surveys of Qinghai Lake Basin, Gahai, BXYGC, and Nanxiangyang, were considered as three of the five county cities of Xihai Jun (Cui et al., 1999). With the collapse of the Wang Mang regime in 23 AD, Xihai Jun was abandoned, the Qinghai Lake area was reoccupied by the Qiang people, and wars of resistance against the Eastern Han regime erupted many times (Cui et al., 1999). Ancient cities built during the Wang Mang period may have been reused for military defense during these wars. Various finds at XHJGC, including Wadang with the writing of the Eastern Han Dynasty, Lotus pattern tiles of the Tang Dynasty, and coins of the Song Dynasty (Li, 1995), indicate the city was restored and reused in these periods.

Tiles found at FSC, gray-white sand-filled corded types with a rough, thick texture, differ from those of the late Western Han



Dynasty found at XHJGC and BXYGC, indicating they belong to a different culture. The luminescence age of the FSC tiles confirms this, dating them to 120–520 AD which mainly corresponds with the Wei Jin Southern and Northern Dynasties. The ancient texts of *Wei Shu*, *Tuyuhun Zhuan* and *Bei Shi*, *Tuyuhun Zhuan*, record the construction of Fuxi city during the Wei Jin Southern and Northern Dynasties, and it became the command center for Tuyuhun occupation of the Qinghai Lake area (Huang and Fang, 1962). No traces of earth ramming have been found at Fuxi; it is likely that the nomadic Tuyuhun set up camps, and that the site was only occupied briefly at that time. However, Fuxi was reoccupied for long periods as an imperial city due to its strategic location on the Silk Road and it played an important role in economic and cultural communications between East and West (Cui et al., 1999). Based on the ^{14}C dating of charcoal sheep dung in the cultural layer (Dong et al., 2016), the city was occupied intermittently through to the Ming Dynasty, 1450–1524 AD.

Qinghai Lake Ancient Cities and Human-Land Relationships

In the late Western Han Dynasty, Wang Mang served as the governor of Xihai Jun and established the earliest city in the Qinghai Lake area in c. 4 AD. Tens of thousands of lawbreakers in the Central Plains were forcibly relocated to Xihai Jun and carried out farming activities there (according to the text of *Han Shu*, *Wangmang Zhuan*). The number and area of cities reflect the high intensity of human activities in the Qinghai Lake area at that time (Table 1). Archaeologists have excavated farming tools in the ancient city of Caolongduo (one of the five counties in Xihai Jun, now submerged by the Longyangxia Reservoir) (Chen, 2005), which confirms that residents of Xihai Jun reclaimed farmland in the Qinghai Lake area in the late Western Han Dynasty. Paleoenvironmental records, including alkenone-based water temperature records from Qinghai Lake sediment core QHL3-

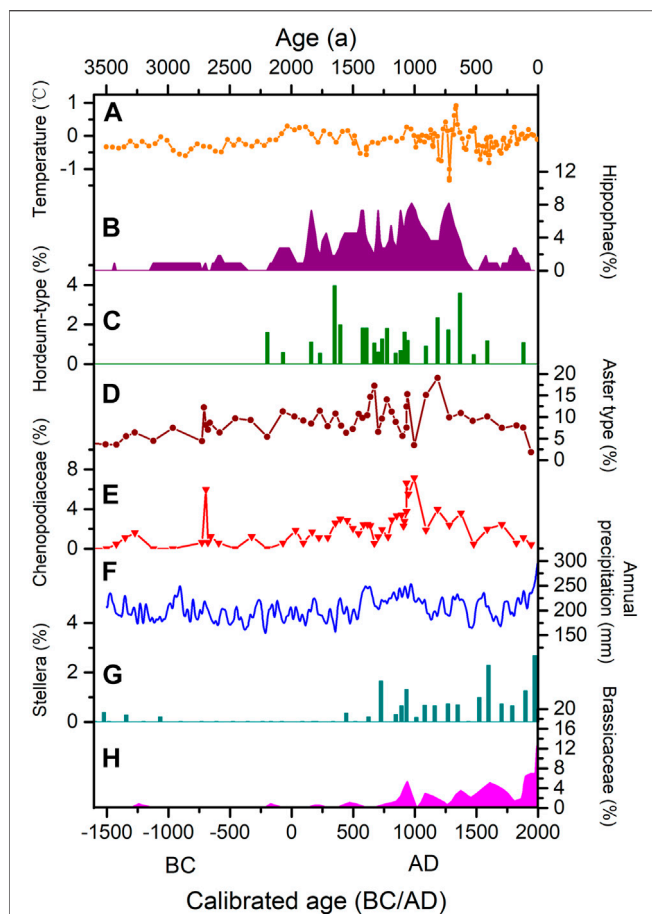


FIGURE 6 | Various paleoenvironmental records for Qinghai Lake area, northeastern Tibetan Plateau. **(A)** Alkenone-based water temperature records from Qinghai Lake (Liu et al., 2006). **(B)** Percentage of *Hippophae* pollen in the LGR section at the southeastern margin of Qinghai Lake Basin (Wei et al., 2020). **(C–E)** Human-related pollen taxa from the LGR section (Wei et al., 2020). **(F)** Annual precipitation reconstruction for the northeastern Tibetan Plateau based on tree rings (Yang et al., 2014). **(G)** *Stellera* pollen percentages from core GGHA from Genggahai Lake in the Gonghe Basin (Huang et al., 2017). **(H)** Brassicaceae pollen percentages from Lake Genggahai (Liu et al., 2016).

1 (Figure 6A) and *Hippophae* pollen percentages in the LGR section in eastern Qinghai Lake Basin (Figure 6B), indicate this was a relatively warm and humid period (Liu et al., 2006; Wei et al., 2020). Thus, hydrothermal conditions in the late Western Han Dynasty were favorable for the development of agriculture. Pollen taxa associated with human activity, such as *Hordeum*-type, *Aster*-type, and *Chenopodiaceae*, first appeared or increased significantly around 2.2 ka (Figures 6C–E), indicating ancient humans were engaged in extensive agricultural production in the Qinghai Lake Basin at that time (Wei et al., 2020). After the fall of the Wang Mang regime in 23 AD, Xihai Jun was abandoned and the Qinghai Lake area came under the control of the nomadic Qiang people, so the intensity of agricultural activities declined.

At the beginning of the 4th century AD, the nomadic Tuyuhun occupied the Qinghai Lake area and after they came into contact with agricultural influences via the Silk Road, they began to

engage in grazing activities and plant barley, beans, and vegetables (according to the text of *Bei Shi, Tuyuhun Zhuan*). Chen et al. (2016) reported a period of strong aeolian activity from c. 1.5 to 0 ka in the northeastern Tibetan Plateau; however, precipitation reconstruction from tree rings contradicts this, with an upward trend between 500 and 600 AD (Figure 6F) (Chen et al., 2016; Yang et al., 2014). This discrepancy suggests increasing aeolian activity after c. 1.5 ka BP may be related to human activity rather than climate change (Chen et al., 2016). A range of proxy indicators indicate an increased intensity in grazing and cultivation activities in Qinghai Lake region at that time. Magnetic susceptibility results for a section in the southwest of Qinghai Lake show paleosol development at 2–1.5 ka (Zhang et al., 2018). Brassicaceae and *Stellera* pollen in a sediment core from Genggahai Lake increased significantly after c. 1.5 ka (Figures 6G,H) (Liu et al., 2016; Huang et al., 2017).

From the time of the Western Han Dynasty (206 BC to 9 AD) to the end of the Qing Dynasty (1911 AD), a period of more than 1,800 years, there were frequent contacts between the nomadic tribes and the Central Plains regime in the Qinghai Lake region, the number of ancient cities increased, and there were repeated shifts between nomadism and farming. Although the total area of cultivation was limited, it was sufficient to damage the natural alpine meadow and shrub vegetation cover of the Qinghai Lake area. Hence, beginning at least 2,000 years ago, human activities might have started to overtake natural climatic variability as the dominant controls of dust storm activity in eastern China (Chen et al., 2020).

CONCLUSION

OSL dating was undertaken on tiles and ceramics from the ancient Qinghai Lake Basin cities of XHJGC, BXYGC, and FSC. The OSL ages of 0–220 AD for XHJGC and BXYGC and 120–520 AD for FSC are consistent with historical documents. The results provide an absolute chronology for tiles in the late Western Han Dynasty and the Tuyuhun period. Paleoclimate records show that the warm and humid climate at c. 2 ka provided good hydrothermal conditions for human activities in the Qinghai Lake area, including farming. During the Wei Jin Southern and Northern Dynasties, Fuxi became the control center for Tuyuhun. As an important transportation node on the Silk Road, the Qinghai Lake area flourished with east-west communication, so the intensity of human activities increased, and the concentration of sporopollenin related to human activities also increased significantly. Since the late Western Han Dynasty, repeated shifts between nomadic pasture and farmland damaged vegetation cover in the Qinghai Lake area and intensification of human activities has been an important influence on aeolian activity.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/Supplementary Material; further inquiries can be directed to the corresponding authors.

AUTHOR CONTRIBUTIONS

YS, CE, and MS contributed to conception and design of the study. MS and HW organized the database. GH provided a typological study of tiles. YZ and LX participated in field sampling. JX performed the statistical analysis. MS wrote the first draft of the manuscript. All authors contributed to manuscript revision and read and approved the submitted version.

REFERENCES

- Ballarini, M., Wallinga, J., Wintle, A. G., and Bos, A. J. J. (2007). A Modified SAR Protocol for Optical Dating of Individual Grains from Young Quartz Samples. *Radiat. Measurements* 42, 360–369. doi:10.1016/j.radmeas.2006.12.016
- Brantingham, P. J., and Xing, G. (2006). Peopling of the Northern Tibetan Plateau. *World Archaeology* 38, 387–414. doi:10.1080/00438240600813301
- Bureau of National Cultural Relics (1996). *Atlas of Chinese Cultural Relics-Fascicule of Qinghai Province*. Beijing: China Cartographic Publishing House Press. (in Chinese).
- Chen, F., Chen, S., Zhang, X., Chen, J., Wang, X., Gowan, E. J., et al. (2020). Asian Dust-Storm Activity Dominated by Chinese Dynasty Changes since 2000 BP. *Nat. Commun.* 11 (1), 992. doi:10.1038/s41467-020-14765-4
- Chen, F. H., Dong, G. H., Zhang, D. J., Liu, X. Y., Jia, X., An, C. B., et al. (2015). Agriculture Facilitated Permanent Human Occupation of the Tibetan Plateau after 3600 B.P. *Science* 347, 248–250. doi:10.1126/science.1259172
- Chen, F., Wu, D., Chen, J., Zhou, A., Yu, J., Shen, J., et al. (2016). Holocene Moisture and East Asian Summer Monsoon Evolution in the Northeastern Tibetan Plateau Recorded by Lake Qinghai and its Environs: A Review of Conflicting Proxies. *Quat. Sci. Rev.* 154, 111–129. doi:10.1016/j.quascirev.2016.10.021
- Chen, X. H. (2005). The Human Activity in the Region of the Qinghai Lake and Environment. *J. Qinghai Nationalities Inst. (Social Sciences)* 31, 38–43. (in Chinese with English abstract). doi:10.3969/j.issn.1674-9227.2005.01.010
- Cui, Y. H., Zhang, D. Z., and Du, C. S. (1999). *The History of Qinghai Province*. Xining: Qinghai People Press. (in Chinese).
- Cunningham, A. C., and Wallinga, J. (2010). Selection of Integration Time Intervals for Quartz OSL Decay Curves. *Quat. Geochronol.* 5, 657–666. doi:10.1016/j.quageo.2010.08.004
- Daniels, F., Boyd, C. A., and Saunders, D. F. (1953). Thermoluminescence as a Research Tool. *Science* 117, 343–349. doi:10.1126/science.117.3040.343
- Dong, G., Liu, H., Yang, Y., Yang, Y., Zhou, A., Wang, Z., et al. (2016). Emergence of Ancient Cities in Relation to Geopolitical Circumstances and Climate Change during Late Holocene in Northeastern Tibetan Plateau, China. *Front. Earth Sci.* 10, 669–682. doi:10.1007/s11707-015-0575-7
- Guérin, G., Mercier, N., Nathan, R., Adamiec, G., and Lefrais, Y. (2012). On the Use of the Infinite Matrix assumption and Associated Concepts: A Critical Review. *Radiat. Measurements* 47, 778–785. doi:10.1016/j.radmeas.2012.04.004
- Hou, G. L., Lai, Z. P., Cao, G. C., E, C. Y., Sun, Y. J., Rhode, D., et al. (2015). The Earliest Prehistoric Pottery in the Qinghai-Tibetan Plateau and its Archaeological Implications. *Quat. Geochronol.* 30, 431–437.
- Huang, S. Z., and Fang, Y. (1962). The Ancient Capital of Tuyuhun-Discovery of Fuxi City. *Archaeology* 8, 436–440. (in Chinese).
- Huang, X.-Z., Liu, S.-S., Dong, G.-H., Qiang, M.-R., Bai, Z.-J., Zhao, Y., et al. (2017). Early Human Impacts on Vegetation on the Northeastern Qinghai-Tibetan Plateau during the Middle to Late Holocene. *Prog. Phys. Geogr. Earth Environ.* 41, 286–301. doi:10.1177/0309133317703035
- Huo, W. (2017). Ancient Qinghai and the Silk Road from Relics and Archaeology Study. *J. Qinghai Nationalities Univ. (Social Sciences)* 43, 10–15. (in Chinese with English abstract). doi:10.3969/j.issn.1674-9227.2017.01.002
- Lai, Z. P., Hou, G. L., Cao, G. C., Sun, Y. J., Wang, Y. X., et al. (2015). Age Determination for a Neolithic Site in Northeastern Qinghai-Tibetan Plateau Using a Combined Luminescence and Radiocarbon Dating. *Quat. Geochronol.* 30, 411–415. doi:10.1016/j.quageo.2015.01.007

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- Li, Z. X. (1995). *The Examination of Ancient Cities in Qinghai Province*. China, Xi'an: Northwestern University Press. (in Chinese).
- Liu, S. S., Huang, X. Z., Qiang, M. R., Lin, X. R., Bai, Z. J., and Peng, W. (2016). Vegetation and Climate Change during the Mid-Late Holocene Reflected by the Pollen Record from Lake Genggahai, Northeastern Tibetan Plateau. *Quat. Sci.* 36, 247–256. doi:10.11928/j.issn.1001-7410.2016.02.01
- Liu, X., Lai, Z., Yu, L., Sun, Y., and Madsen, D. (2012). Luminescence Chronology of Aeolian Deposits from the Qinghai Lake Area in the Northeastern Qinghai-Tibetan Plateau and its Palaeoenvironmental Implications. *Quat. Geochronol.* 10, 37–43. doi:10.1016/j.quageo.2012.01.016
- Liu, Z. H., Henderson, A. C. G., and Huang, Y. S. (2006). Alkenone-Based Reconstruction of Late-Holocene Surface Temperature and Salinity Changes in Lake Qinghai, China. *Geophys. Res. Lett.* 33, 370–386. doi:10.1029/2006gl026947
- Murray, A. S., and Wintle, A. G. (2000). Luminescence Dating of Quartz Using an Improved Single-Aliquot Regenerative-Dose Protocol. *Radiat. Measurements* 32, 57–73. doi:10.1016/s1350-4487(99)00253-x
- Prescott, J. R., and Hutton, J. T. (1994). Cosmic ray Contributions to Dose Rates for Luminescence and ESR Dating: Large Depths and Long-Term Time Variations. *Radiat. Measurements* 23, 497–500. doi:10.1016/1350-4487(94)90086-8
- Rhode, D., Haiying, Z., Madsen, D. B., Xing, G., Jeffrey Brantingham, P., Haizhou, M., et al. (2007). Epipaleolithic/Early Neolithic Settlements at Qinghai Lake, Western China. *J. Archaeological Sci.* 34, 600–612. doi:10.1016/j.jas.2006.06.016
- Sun, M. P. (2019). *Luminescence Dating on Ceramic of Nuomuhong Talitaliha Site and Tianjun Karst Cave Site*. master's thesis. Xining, Qinghai province: Qinghai Normal University.
- Sun, M., Sun, Y., E, C., Hou, G., Zhang, J., and Shi, Y. (2021). Luminescence Dating of Nuomuhong Culture Ceramics at Talitaliha Site on the Northeastern Qinghai-Tibetan Plateau. *Geochronometria*. doi:10.2478/geochr-2020-0034
- Sun, Y., Chongyi, E., Lai, Z., and Hou, G. (2018). Luminescence Dating of Prehistoric Hearths in Northeast Qinghai Lake and its Paleoclimatic Implication. *Archaeol Anthropol. Sci.* 10, 1525–1534. doi:10.1007/s12520-017-0472-y
- Sun, Y., Lai, Z., Madsen, D., and Hou, G. (2012). Luminescence Dating of a Hearth from the Archaeological Site of Jiangxigou in the Qinghai Lake Area of the Northeastern Qinghai-Tibetan Plateau. *Quat. Geochronol.* 12, 107–110. doi:10.1016/j.quageo.2012.01.010
- Thomas, P. J., Nagabhusanam, P., and Reddy, D. V. (2008). Optically Stimulated Luminescence Dating of Heated Materials Using Single-Aliquot Regenerative-Dose Procedure: A Feasibility Study Using Archaeological Artefacts from India. *J. Archaeological Sci.* 35, 781–790. doi:10.1016/j.jas.2007.06.015
- Wang, S., and Zhao, X. (2013). Re-Evaluating the Silk Road's Qinghai Route Using Dendrochronology. *Dendrochronologia* 31, 34–40. doi:10.1016/j.dendro.2012.05.001
- Wang, W. D. (2008). The Correct Estimation of Palaeodose in Porcelain Thermoluminescence Dating. *Sci. China Press* 38, 460–468. doi:10.1088/0953-8984/20/42/425208
- Wang, W. D. (1993). Thickness Response of β Dose Rate in Pottery Fragments. *Nucl. Tech.* 16, 228–231.
- Wei, H., E, C., Zhang, J., Sun, Y., Li, Q., Hou, G., et al. (2020). Climate Change and Anthropogenic Activities in Qinghai Lake basin over the Last 8500 Years Derived from Pollen and Charcoal Records in an Aeolian Section. *Catena* 193, 104616. doi:10.1016/j.catena.2020.104616

- Wintle, A. G., and Murray, A. S. (2006). A Review of Quartz Optically Stimulated Luminescence Characteristics and Their Relevance in Single-Aliquot Regeneration Dating Protocols. *Radiat. Measurements* 41, 369–391. doi:10.1016/j.radmeas.2005.11.001
- Yang, B., Qin, C., Wang, J., He, M., Melvin, T. M., Osborn, T. J., et al. (2014). A 3,500-Year Tree-Ring Record of Annual Precipitation on the Northeastern Tibetan Plateau. *Proc. Natl. Acad. Sci.* 111, 2903–2908. doi:10.1073/pnas.1319238111
- Zhang, J., E, C. Y., and Zhao, Y. J. (2018). A High-Density Optical Stimulated Luminescence (OSL) Dating at Heima He Loess Section in Qinghai Lake Area. *J. Earth Environ.* 9, 557–568. (in Chinese with English abstract).

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