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Article

Geology

Component and simulation of the 4,000-year-old noodles excavated from the archaeological site of Lajia in Qinghai, China

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Abstract Noodles are a global food, but the periods when and places where they were made and originated, as well as their ingredients and the cooking methods used to manufacture them, have remained contentious. In the 2005 edition of the journal *Nature*, we wrote a summary of the millet noodle specimens found in the Late Neolithic Qijia cultural stratum of the Lajia archaeological site in Qinghai Province, China. However, how the ancient people made millet noodles remains controversial. This paper provides a systematic analysis of the remains of noodles found within an earthenware bowl at the Lajia "noodle house" in terms of their plant composition including phytoliths, starch and biomarkers. It provides evidence of how people used millet 4,000 years ago and, most specifically, of the principal methods used for producing millet-based noodles. Further, we show how we used traditional *hele* tools to make *hele* millet noodles, with especial reference to the gelatinized hydrogel-forming method, to simulate morphology consistent with the composition and form of the unearthed millet noodles. The results of this study provide new evidence and new insights into the cultural characteristics of the prehistoric human diet.

Keywords Lajia site · Phytolith · Starch · Biomarkers · Origin of noodles · Millet noodles

1 Introduction

Modern noodles can be subdivided into two main types [1–3], according to the ingredients used in their production. The first type uses the crop gluten viscosity, with the gluten coming principally from hard wheat (*Triticum durum* Desf.), common wheat (*Triticum aestivum* L.), rye (*Secale cereal* L.) and barley (*Hordeum vulgare* L.). There are many types of wheat noodles produced in Asia, with approximately 40 % of all wheat produced being used in the manufacture of noodles [4].

The second category of noodle is which uses crop-produced viscous "starch gels" to produce starchy noodles [2, 5]. These include bean starch noodles which use ingredients such as mung beans [*Vigna radiata* (L.) Wilczek], red beans (*Phaseolus radiatus* var. *Aurea*) [6], kidney beans (*Phaseolus vulgaris*), broad beans (*Vicia faba* L.), peas (*Pisum sativum* L.) [5]; tuber starch noodles which use

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ingredients such as cassava (*Manihot esculenta* Crantz) [7], sweet potato [*Ipomoea batatas* (L.) Lam], potato (*Solanum tuberosum* L.) [8] and the like; and cereal starch noodles which use corn (*Zea mays* L.) [9], rice (*Oryza sativa* L.) [10, 11], sorghum (*Sorghum vulgare* Pers) [12, 13], foxtail millet [*Setaria italica* (L.) Beauv.] [14], broomcorn millet, also known as common millet (*Panicum miliaceum* L.) [15, 16], buckwheat (*Fagopyrum esculentum* Moench) [2] and the like. An early production method for starchy noodles was recorded in his agricultural encyclopaedia entitled "*Qi Min Yao Shu*" (essential skills for benefiting the people) by the writer Jia Sixie in the Northern Wei Dynasty [5, 17]. This starch noodle production method does not need to join the wheat flour gluten protein. Different plant starches produce different noodle forms, greatly enriching the species of noodles.

Different noodle varieties are symptomatic not only of the pre-eminence of noodles as a global food type but also because they reflect the different cultures found around the world. Nonetheless, when it comes to the origins of noodles, this has proven a somewhat controversial topic, with China, Italy, Arabia and other nations and regions all claiming their rights over the invention of noodles [1, 18]. Of the literature of these countries concerning noodles, Chinese records are the earliest and can be predated to the Eastern Han Dynasty about 2,000 a BP [19]. However, in prehistory, before there were any written records, what were the materials and manufacturing processes used when ancient peoples began to make noodles? There is a genuine lack of primary research material available to enthusiasts [1, 3, 18], most importantly because noodles are rarely preserved for long periods.

At the Qinghai Lajia excavation site, there is evidence that the Qijia culture was affected by natural disasters such as earthquakes, flooding and mudslides [20, 21]. Archaeological excavations have unearthed human bones, a large number of pottery pieces, jade, animal bones and the like underneath the detritus of these major natural catastrophes. There was a rapid accumulation of sediment created by these disasters which buried, sealed and preserved a pottery bowl filled with noodles at the Lajia excavation site. In 2002, when excavations unearthed this bowl of noodles, the discovery not only provided the earliest physical evidence of noodles in the world, but it also allowed recently developed research methods for phytoliths [22–25], starchy grain [26, 27] and molecular marker [28] to be used for a compositional analysis. This find provided a rare opportunity for the study of ingredients used in ancient noodles and provided an opportunity to simulate the traditional methods used to make ancient noodles.

2 Materials and methods

2.1 The stratigraphy of the unearthed noodles at the excavation site

The Lajia site is located on the secondary terracing of the northern banks of the Yellow River in the Guanting Basin, Minhe County, Qinghai Province, China, at a height of 1,700–1,800 m a.s.l. (Fig. 1). The lower strata of the valley terrace margins are of fluvial gravel which, towards their tops, are covered by a fine-grained alluvial (floodplain) sediment as well as the sedimentary remains of late Pleistocene *Malan* loess and Holocene paleosol deposits. The principal crops grown in the Guanting Basin today include barley, highland barley (*H. vulgare* L.var. *nudum* Hook), wheat, broomcorn millet, foxtail millet, sorghum, corn and potatoes, while in the environs of the excavation site fruits such as apples (*Malus domestica* Borkh), pears (*Pyrus communis* L.) and peaches (*Juglans regia* L.) are grown.

From September to December 2002, in the southeastern corner of the V courtyard area of the excavation site, a room earmarked as F20 was excavated [29]. Its area was 5 m × 5 m. The upper section of the cross section of the site's sidewalls revealed a yellow–brown ploughed stratum with a thickness of 0.3–0.5 m, and below this covering at a depth of about 3 m there was a fluvial–alluvial sedimentary layer (Fig. 1c). The area below this fluvial–alluvial layer included a Neolithic cultural level with prehistoric noodles and ancient flooring indicative of prehistoric human domestic activity.

The ancient flooring in F20 was scattered with a great quantity of pottery, stone tools and many other remnants. The pottery bowl with noodles was found in the northeastern part of the room (Fig. 2a) [29, 30]. The pottery bowl was concealed within a brownish yellow–grey silty clay layer; after the clay layer covering the bowls had been cleaned off, the bowl appeared to be upside down upon the floor. The base of the bowl pointing upward was approximately 5.5 cm in diameter. The diameter of the open "mouth" end of the bowl was of approximately 14 cm, and the depth of the bowl was roughly 6–6.3 cm (Fig. 2b, c).

When the bowl was uncovered, the bowl and the clay sediment within it had already separated naturally and the sedimentary frustrum took the shape of the inside of the bowl upturned on the floor. Within the sculpted filling, we discovered matter resembling noodles, though at the time the staff at the site was not at all certain that they were noodles and were also doubtful in case they turned out to be fungal substances (Fig. 2c, d) [30]. The archaeologists immediately photographed the materiel from all angles for the record.

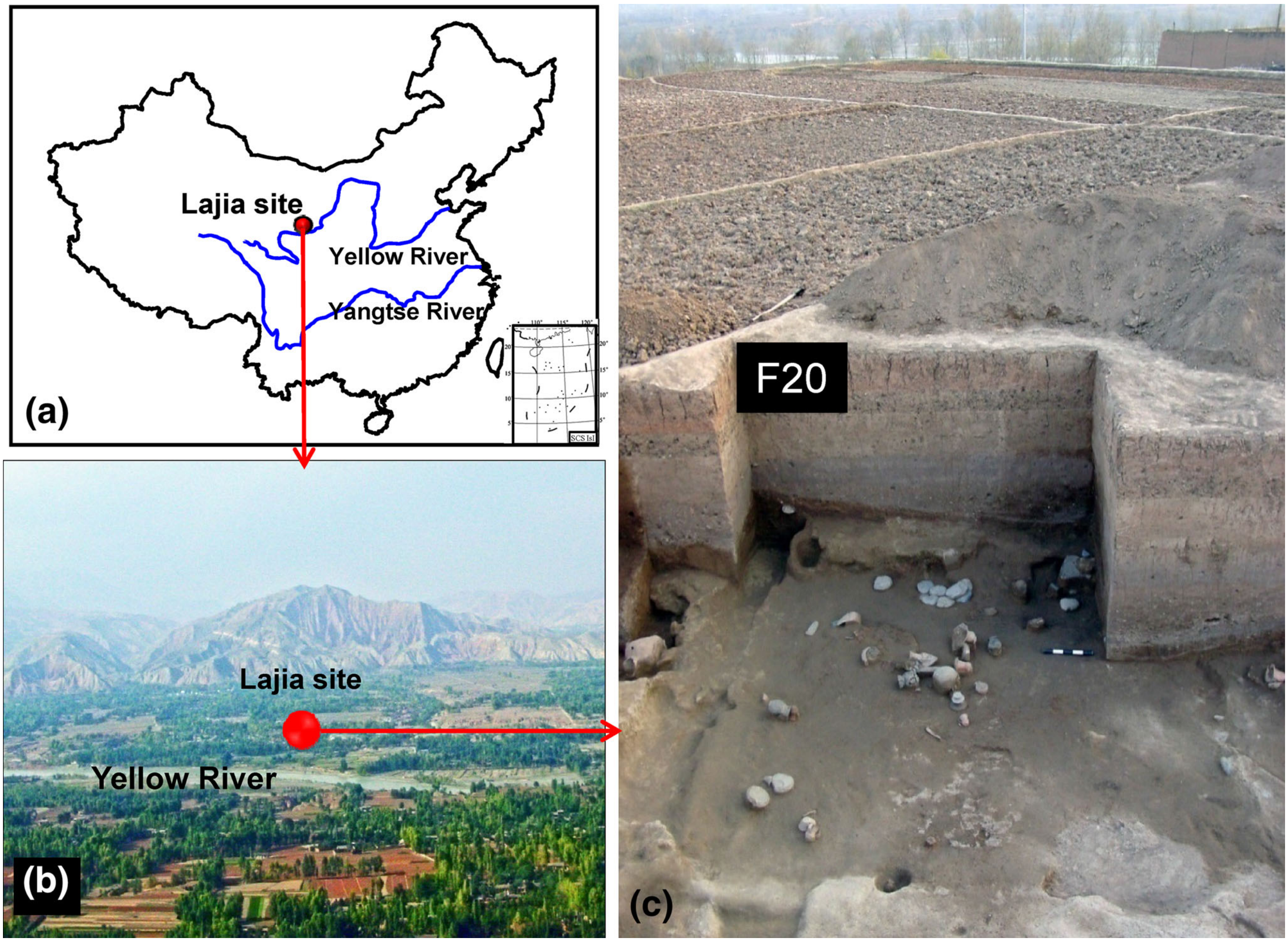

Fig. 1 Location of the research site. **a** General map of the location of the excavated Lajia Noodle House. The Yellow River and the Yangtze River are the principal water systems of northern and southern China, **b** the Lajia Noodle House excavation site on the secondary terrace of the northern bank of the Yellow River, **c** the F20 excavated room at the Lajia Noodle House excavation site

The sediment filling within the pottery bowl was of approximately 6 cm in thickness and could be divided into two layers by colour and composition. The filler below the shaped sediment was approximately 3 cm in thickness and was composed of relatively pure brown–grey silty clay. The clay layer covering the outside of the pottery bowl was of a fundamentally similar composition. The filler of its upper layer of about 3 cm thickness was taupe in hue, displaying pale grey sediment in its somewhat loosely structured upper layer. There were a good number of exposed curved pale yellow noodles as well as some within the sedimentary strata. At the top of the rounded filler were curled remains of pale yellow noodles of uniform thickness and a diameter of about 0.3–0.4 cm. These had been weathered in the middle, thus presenting a rounded cross section. The noodle tips were visible in the middle section on the sedimentary frustrum, and from their wound up appearance, it was estimated that their length from tip to tip was approximately 50 cm (Fig. 2d) [30].

2.2 Sample collection

2.2.1 *Archaeological sample collection*

In order to analyse the composition of the noodles within the pottery bowl, in 2004, we removed samples of the sediment filling, the noodle residue within the bowl, and collected the sediment from the excavation site.

The noodles uncovered from the shaped sediment fill were either shattered or weathered. Nonetheless, the noodles within the upper 3 cm still evinced noodle remains with approximate lengths of 1–3 cm, with multiple noodle fragments of <1 cm (Fig. 3a–d). We took samples from both the noodle lengths and noodle fragments. We discovered that the much smaller noodle fragments, starch and other forms had already been scattered throughout the noodle-ridden sedimentary clay layer during the shattering and weathering process. We decided to take samples every 1 cm from the top to the bottom of the sediment filling up to a total of six

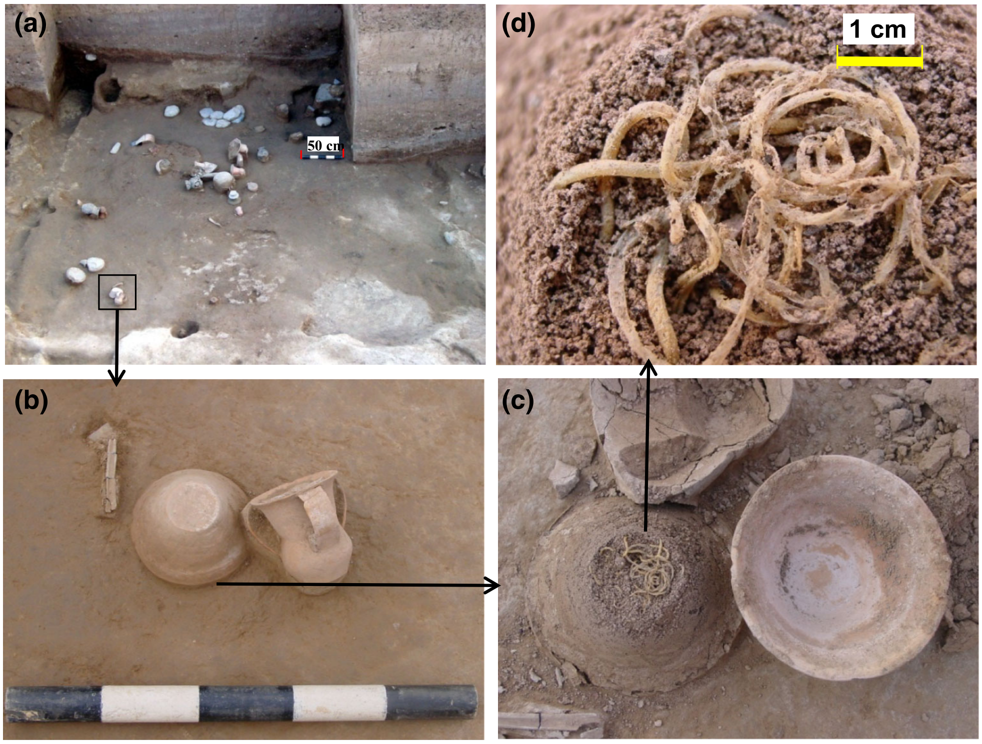

Fig. 2 Unearthed bowl and noodles as found in the F20 room at the Lajia Noodle House excavation site. **a** The bowl in which the noodles were found in the northeastern corner of the room at the site, **b** the bowl *in situ* prior to turning, **c** the prehistoric noodles in the position in which they were found within the sediment filling (frustrum) after turning over the bowl, **d** the sediment filling (frustrum) within which the noodles were found

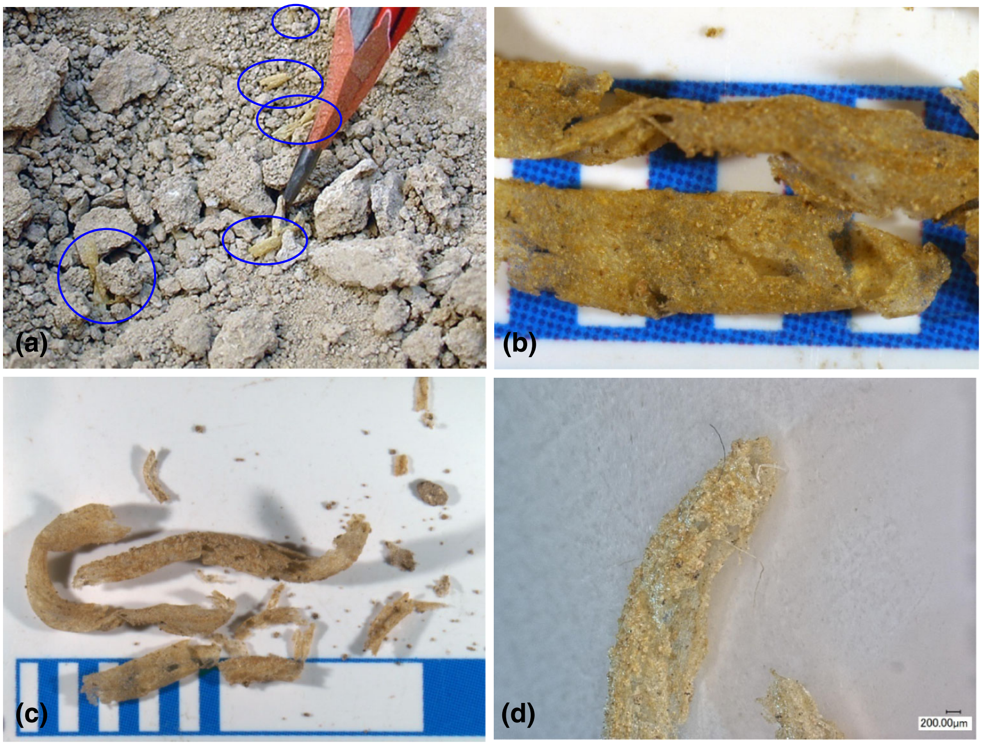

Fig. 3 Noodle remnant samples from within the pottery bowl from the F20 room at the Lajia site. **a** Close-up photographs of part remains of noodles from the upper parts of sediment cones from within the pottery bowl, showing bluish, curled noodle lengths and fragments (**b**–**d**)

samples, which were then labelled ①–⑥. The samples within the upper 3 cm of the noodle-containing sedimentary layer were taken as samples ①, ② and ③. Here, fragments or incomplete pieces of fine-grained noodles were occasionally seen. Loess samples were also taken from strata surrounding the F20 excavation site for comparative analysis.

2.2.2 *Modern sample collection*

So as to verify phytolith, starch and biomarker analytical methods used in the identification of the composition of ancient noodles, we collected modern plant and crop samples for analysis and identification and for clear identification markers.

Plants and crops commonly grown in northwestern China were collected and included barley, highland barley, wheat, sorghum, rice (*O. sativa*), foxtail millet, green foxtail grass [*Setaria viridis* (L.) P. Beauv.], broomcorn millet, corn, potato and the like. In addition, we used pre-published phytolith, starch and biomarker data for identification purposes [22, 24, 28, 31, 32].

2.3 Analytical methods

2.3.1 *Phytolith analysis*

For the archaeological sediment analysis of phytoliths, we used the methods identified by Piperno [33] and Lu et al. [34] with the following modifications: first, we dispersed sodium pyrophosphate ($Na_4P_2O_7$) throughout the sediment samples; then, we used a 30 % hydrogen peroxide (H_2O_2) concentration for the removal of organic matter; following this, we employed a 15 % hydrochloric acid (HCl) concentration to remove any calcium cement; and, finally, we used a $ZnBr_2$ heavy liquid (of specific gravity 2.35 g/cm^3) for phytolith flotation, and mounting on a microslide with Canada Balsam. In addition, we employed a biological phase contrast microscope Leica lens of 400× for identification of the phytoliths.

We focused on an analysis of selected inflorescence bracts. We followed the method used by Lu et al. [22]. (1) After each part of the plant spikelets has been separated, it is washed with distilled water; (2) all samples are placed in 20 mL of concentrated nitric acid and soaked for up to 12 h to aid the complete oxidation of organic matter; (3) the solution is placed in a centrifuge set at speed 2,000 r/min for 10 min, the supernatant liquid decanted, the residue then washed twice with distilled water, then with 95 % ethanol, and the supernatant cleaned out; (4) the washed phytoliths are transferred to a clean test tube, a few drops are extracted evenly and placed on a microslide, pasted with a covering of Canadian Balsam and coverslip for microscopic observation and measurement; (5) light photomicrography (phase contrast and microscopic interferometer) at 400× magnification was used to determine their anatomy and silicon structure patterns in the glumes, lemmas and palea; and (6) image analysis software is used to measure the morphological parameters of the phytoliths.

2.3.2 *Starch analysis*

Starch analysis of archaeological sediment samples: we followed the method used by Piperno [35] and Yang et al. [36] with some slight modifications. We weighed out a 0.5-g sample into a 50-mL centrifugal test tube, added 20 mL of a 6 % H_2O_2 solution, and let it stand for 12 h. We discarded 4/5 of the supernatant and added 40 mL of sodium hexametaphosphate (calgon), oscillating the admixture for 10 min, washing it until the pH was neutral. The centrifuged supernatant was then discarded, and a heavy caesium chloride solution (specific gravity 1.8 g/cm^3) was used for flotation; a few drops of starch were placed on a microslide with 10 % glycerol medium. A 400× Leica polarizing microscope was then used for observations, identifications and statistics. The starch's granular microscopy was recorded using a digital camera.

The noodle debris from the pottery bowl was analysed by using the method of modern plant starch analysis.

We used the method described in [22, 31] with some minor modifications. After we immersed the plants' fruit (or food) in distilled water, we crushed them using a rubbing process, loaded them with 10 % glycerol. We used a 400× biological microscope with the differential interference contrast (DIC) and the polarization. For statistical accuracy, we conducted at least 100 random observations of particles for each sample, recording and measuring the special morphology of the starch particles.

2.3.3 *Biomarker analysis*

Due to the small biomass of the noodle debris, the test requirements could not be met. The main noodle ingredients had already been dispersed within the sedimentary noodle strata. We analysed samples ① and ② (contained noodle fragments), and then, for comparison, analysed selected Holocene loess and atmospheric dust samples. We also selected and analysed modern samples of living broomcorn millet, foxtail millet and wheat from northern China.

The samples were first air-dried and then 1 g of each sample selected. Each of these was mixed using an acetone and pentane 1:1 solution and then was ultrasonic extracted three times. After the extract was filtered, each was separated by column chromatography into saturated hydrocarbon, aromatic hydrocarbon and non-hydrocarbon fractions, with hexane, dichloromethane and methanol as eluent. The aromatic fraction was fed by gas chromatography–mass spectrometry (GC–MS).

GC–MS was conducted by using an Agilent 6890N GC-5973N MSD mass selective detector system (EI 70 eV; ion source temperature, 230 °C) fitted with a fused silica chemically bonded capillary column (J&W DB-5; 0.25 mm in diameter, 30-m long, 0.25-μm film thickness). Each sample was injected onto the column at 280 °C in the splitless mode. After a 1-min isothermal hold at 50 °C, the column temperature was increased by 30 °C/min to 120 °C; and then 3 °C/min to 290 °C, successively, with a 30-min isothermal hold at 290 °C. The flow rate of the helium carrier gas was 1.2 mL/min (40 cm/s) [24].

By measuring the total peak area for the ionized millet hormone (miliacin), the relative content of this millet hormone could be calculated. It was noted that the relative content of millet hormone yielded slightly different results versus different gas chromatography–mass spectrometry settings. Such minor differences may have resulted from differences in the concentrations of the ion sources.

2.3.4 *Age dating*

After retrieving 12 carbon debris samples and five bone samples, from the ash pits, tombs and a total of 17 rooms were buried by a catastrophic event at the Lajia site, and these were sent to the Radiocarbon Dating Laboratory of the Archaeology Research Institute of the Chinese Academy of Social Sciences (ZK) and the Accelerator Mass Spectrometry (AMS) Radiocarbon Dating Laboratory at Beijing University for age dating.

3 Results

3.1 Results of phytolith analysis

We investigated six samples extracted from the sediment found within the inverted earthenware bowls (Fig. 4; serial numbers ①–⑥) and compared these with a soil sample taken from a cross section at the excavation site (Fig. 4). Many phytolith types were found in all the samples, with Poaceae of subfamily level, such as *Panicoid* type, *Chloridoid* type and *Festucoid* type, identified amongst these (Fig. 4). Remarkably, close to the bottom of a bowl (in the upper sediment frustum) of three samples (Nos. ①, ② and ③), three types of husk phytolith were found. Based upon their shapes and patterns, and comparing them with modern crops phytolithic husks (Fig. 5) and referencing them to previously published research findings from investigations of phytoliths found in crops and wild plants [22, 23, 37, 38], we discovered three types of phytolith: Ω-shape (Setaria-Ω), the epidermal long cell walls are Ω-undulated (undulations rounded, wider towards the apex and narrower at the base) from the foxtail millet (*S. italica*); π-shape (*Setaria-π*), heavy silicon epidermal cells and jointed cuticles presenting ridgy line sculpture from the foxtail millet (*S. italica*); η-shape (*Panicum-η*), the epidermal long cell walls are η-undulated from the broomcorn millet (*P. miliaceum*) [22].

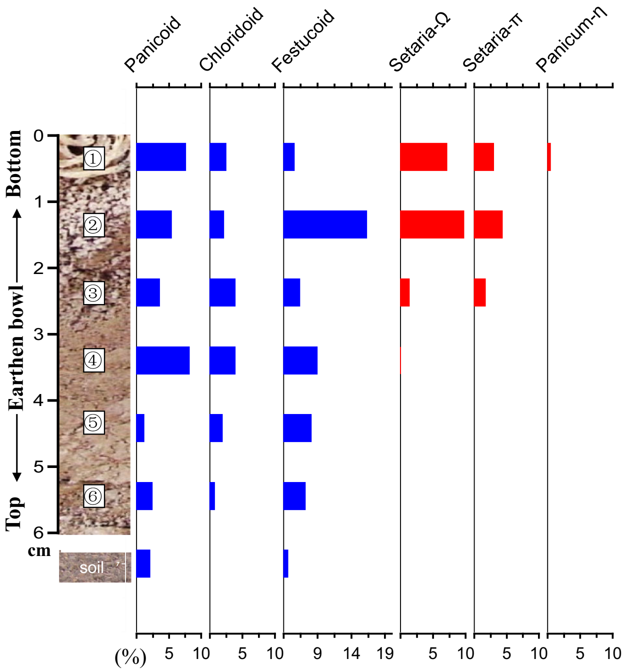

Fig. 4 Variation in phytolith types within the sedimentary material found within the inverted earthenware bowls compared with soil phytoliths from the excavation site

The η-shaped phytoliths were found in sample ① from the earthenware bowl bottom and constituted only approximately 0.5 % of the total. Ω- and π-shaped phytoliths found in the three samples from the earthenware bowl bottom (in the upper sediment frustum) containing noodle strata constituted upward of 10 %–15 % of the total. Two Ω-shaped phytoliths were found in the noodle fragment samples, though none of the three types of millet (Ω-, π- and η-shaped) nor hulled millet phytoliths were found in the three sediment samples (Nos. ④, ⑤ and ⑥) from the earthenware bowl mouths or the soil samples from the excavation site. When the overall numbers of foxtail millet and broomcorn millet phytoliths found were calculated, foxtail millet phytoliths were seen to be completely dominant, constituting 95 % or more of the total.

3.2 Results of starch analysis

A wealth of starch grains was found in the prehistoric noodle fragments (Fig. 6b–e) and, furthermore, a considerable number of starch grains were extracted from the three samples (serial numbers ①, ② and ③) taken from the

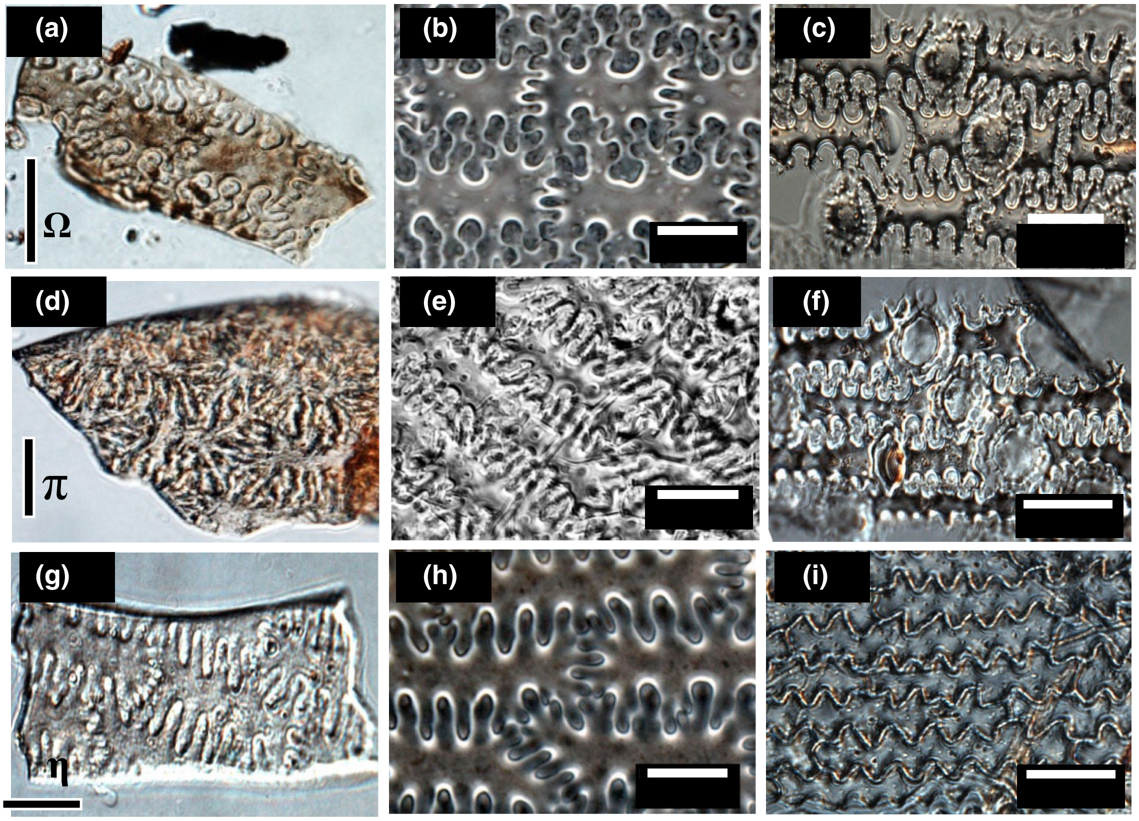

Fig. 5 (Color online) Husk phytoliths in the noodle sediment strata alongside modern plant husk phytoliths. **a**, **d**, **g** Separated, are the Ω-, π- and η-shaped phytoliths found in noodle fragments or in noodle strata; **b**, **e** separated, are Ω- and π-shaped phytoliths in modern foxtail millet; **h** is η-shaped phytoliths from modern broomcorn millet; **c**, **f**, **i** separated, are husk phytoliths from modern wheat, barley and sorghum. *Scale bar* = 20 μm

lower strata within the earthenware bowls (Fig. 6a). No starch grains were extracted from samples taken from other strata.

Starch grains found in the noodle sediment strata and within noodle fragments all evince a definite pasted character, partly heavily gelatinized, and displaying a gradually tapering and fading cruciform birefringence shape (Fig. 6d); once the starch forms have been partly gelatinized, these cruciform structures become not obvious (Fig. 6e). On some of the noodle fragments, however, a great number of rounded or quasi-rounded edges, with umbilical points in the centre, can still be found on the starch granules, and the cruciform birefringence structure remains relatively apparent (Fig. 6b).

The starch grains extracted from the noodle sediment strata are typical of a relatively large average grain size. The weights of the complete starch granules found in the sedimentary strata and in noodle fragments were measured and found to be, on average, 8.2 ± 2.8 μm. Comparing this with the size and morphology (Fig. 7) of starch granules taken from modern plants grown in northwest China (Table S1), the archaeological noodle samples had starch granules which, in size, appeared smaller than those of barley (18.4 ± 8.7 μm), highland barley (16.8 ± 5.9 μm) or wheat (15.7 ± 6.1 μm), but close in morphology and size to those of foxtail millet (8.6 ± 2.0 μm) and broomcorn millet (7.8 ± 2.1 μm).

After gelatinization, the morphology of many starch grains began to change and/or fade, and it became very difficult to identify accurately the origins of the plant species. Looking at the starch grains found in the noodle fragments and noodle sediment strata currently in question, the material used to make noodles certainly came from plant starch and, judging from the size of the starch grains, these had characteristics closer to the starches found in foxtail millet and broomcorn millet.

3.3 Results of biomarker analysis

Research has already clarified that millet hormone (miliacin) is a specific component of broomcorn millet and foxtail millet [24, 28] and, although the miliacin content in broomcorn millet and foxtail millet is not the same, their emergent derivatives are dissimilar [24], allowing broomcorn millet and foxtail millet to be differentiated.

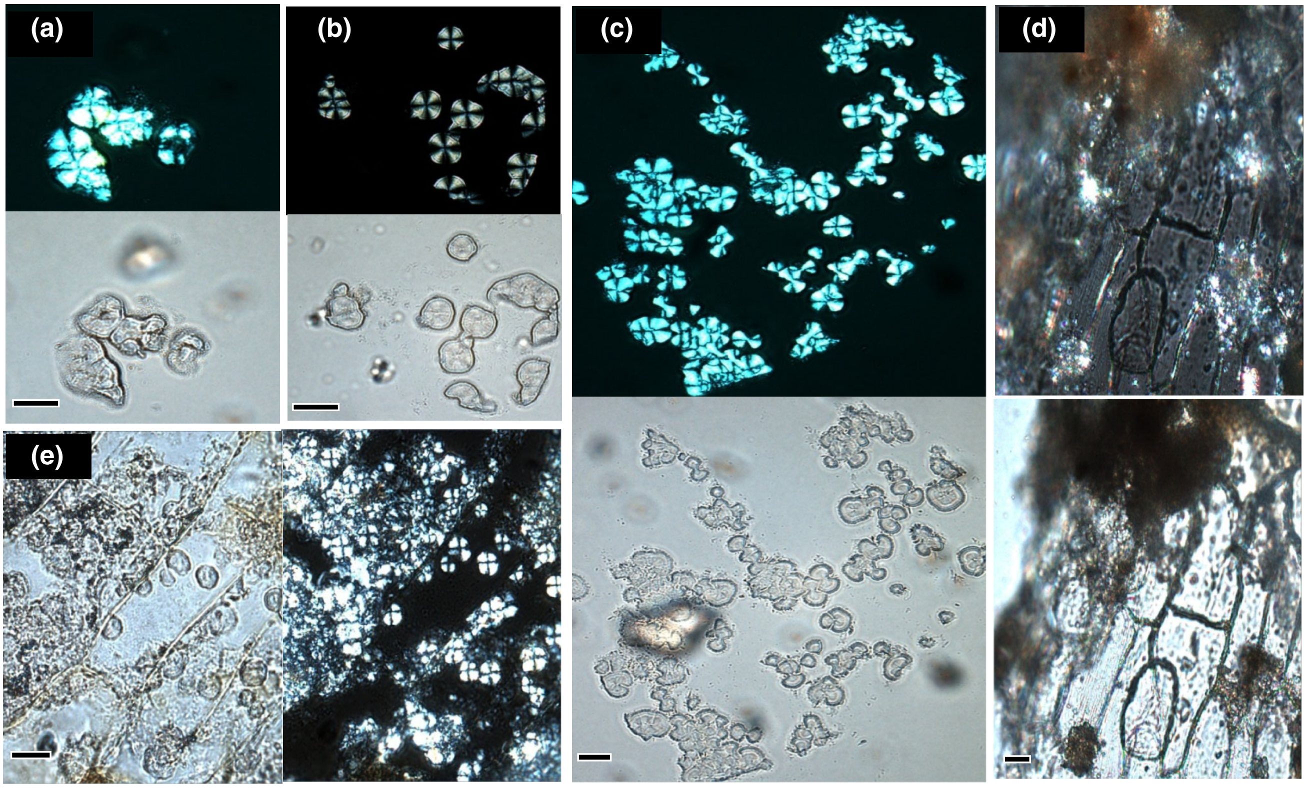

Fig. 6 (Color online) Starchy granules discovered in the noodle sediment strata and in incomplete noodle pieces. **a**–**d** The upper half of each photograph is an orthogonal polarization image; **e** the right half of the photograph is an orthogonal polarization image. *Scale bar* = 20 μm

Figure 8a, b shows a portfolio of five types of miliacin derivatives contained within broomcorn millet and foxtail millet. These five compounds are as follows: peak 1, olean-12-en-3β-ol ME: β-amyrin ME, β-amyrin ether; peak 2, olean-18-en-3b-ol ME: miliacin, broomcorn millet hormone; peak 4,urs-12-en-3β-ol ME; α-amyrin ME, α-amyrin ether; peaks 3 and 5, as yet structurally undetermined PTMEs (pentacyclic triterpenoid methyl ethers).

Figure 8a, b separately shows the continuous distribution of the aromatic hydrocarbon fraction of broomcorn millet and foxtail millet (including broomcorn millet [miliacin] hormone derivatives) between 54 and 62 min. Of the principal millet hormone (miliacin) compounds (compound 2) (Fig. 8d) present in broomcorn millet and foxtail millet, we measured a significant difference in the soluble organic matter content between the two plants, which accounted for 89.0 % ± 1.64 % (broomcorn millet) and 33.8 % ± 22.2 % (foxtail millet), respectively. More important was the presence of compounds 1, 4 and 5 in broomcorn millet, whereas only compound 3 was present in foxtail millet. It was possible to identify and differentiate between broomcorn and common millet on the basis of the content and composition of these compounds.

Figure 8c shows the analytical results of investigations into the molecular markers of the archaeological samples taken from the noodle strata at the earthenware bowl, with a compound 2 peak evincing a morphological phase consistent with that of modern foxtail millet, and a lack of combinations of extant broomcorn millet compounds 1, 4 and 5. This shows that the samples' molecular markers clearly come from foxtail millet and are consistent with the results of analysis of phytoliths and starch.

We analysed a sample of modern wheat in the same way, but did not detect any millet hormone (miliacin) components (Fig. 9a). We measured other sample types including Holocene and Pleistocene loess samples, and modern dust samples, but did not detect any miliacin components (Fig. 9b). We thus ruled out the possibility that, during the burial and excavation process, the samples could have been contaminated. We also comprehensively analysed the results of phytoliths, starch grains and biomarker, these need further clarification, that principal ingredient of the noodles was foxtail millet, possibly mixed with a small quantity of broomcorn millet.

3.4 Age-dating results

Table S2 shows the age-dating results for 17 plant remains and bone samples. The ^{14}C dating range for the carbonized plant remains and bones found within the cultural settlement strata is 4,260–3,840 a BP, with the principal dates focused on 4,070 a BP, consistent in age with the Qijia culture.

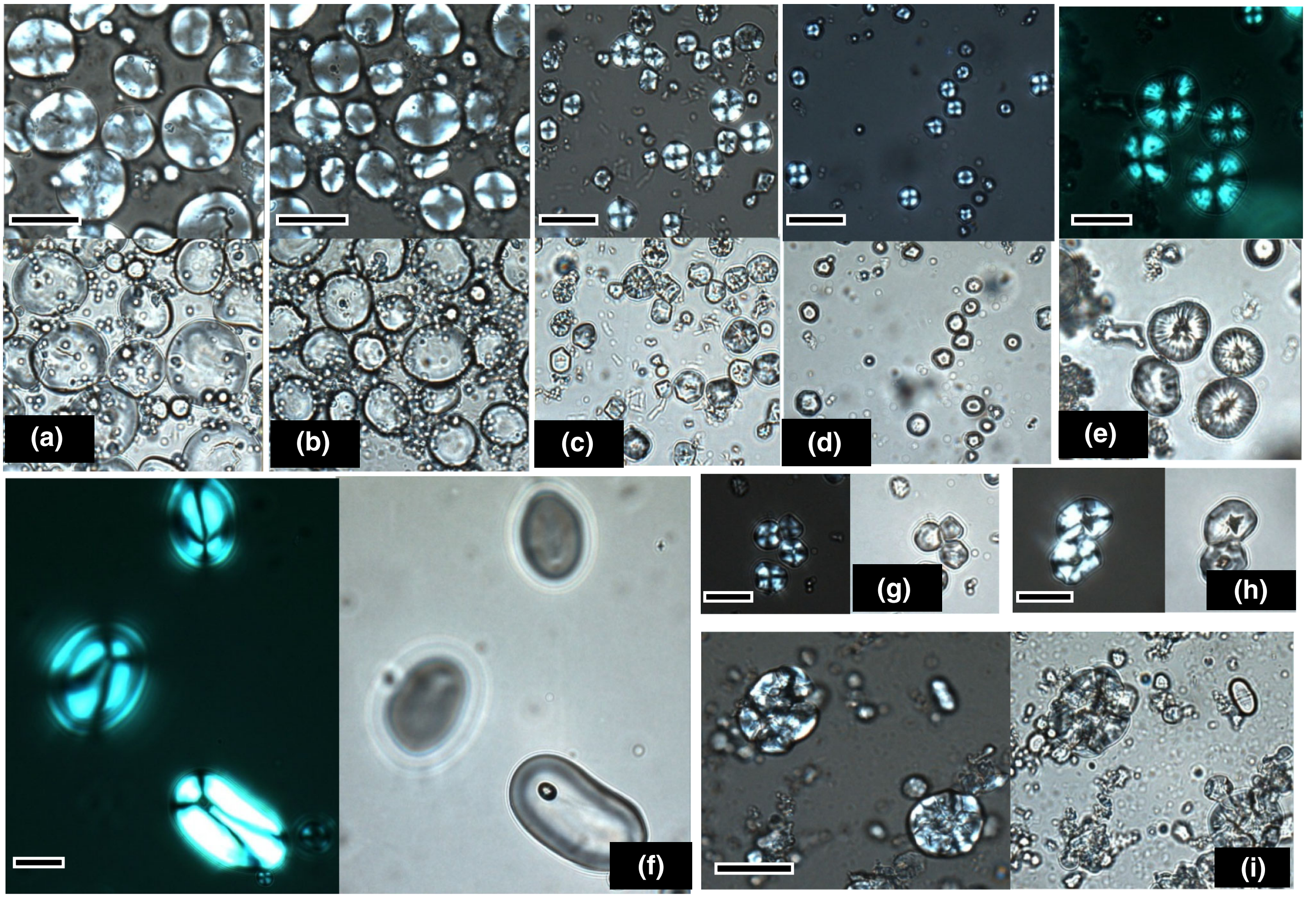

Fig. 7 (Color online) Polarized and optimal micrographs of starchy grains extracted from parts of modern cultivated plants. **a** Barley, **b** highland barley, **c** foxtail millet, **d** green foxtail, **e** sorghum, **f** potato, **g** broomcorn millet, **h** corn, **i** wheat. **a–e** The upper half of each photograph is the polarized image; **f–i** the left half of each photograph is the polarized image. *Scale bar* = 20 μm

4 Simulated testing

4.1 Aim

To take the results from analyses of noodles with an age of 4 ka BP unearthed at the Lajia site and, exploring the simple methods, make the uniform thickness of noodles in average length >30 cm, thermoforming millet noodles in water.

4.2 Materials and tools

Foxtail millet flour, shelling foxtail millet (both purchased from Xifeng in Gansu Province), Foxtail millet (still shelled and collected from Hebei, Liaoning and Shanxi provinces and from the Chinese Academy of Agricultural Sciences, Beijing), water, mortars, steamers, *hele* noodle pressure machine (big and small types, purchased from Xifeng in Gansu Province) and an electromagnetic cooker.

4.3 Simulation method

The manufacturing process of the viscous matter for starch noodles (gel) is different from that for wheat noodles (glutinous protein). The gel is made through heating and gelatinizing the starch, and so, the simpler methods involved in crushing, smashing, grinding and rolling methods, as wheat noodle making process, cannot be used to make long and slender starch noodles.

This test referenced: traditional starch noodle production methods (from Shouyang and Ying counties, Shanxi Province and from Xun County, Henan Province); literature concerning the production methodology for ancient starch noodles [17]; and modern starch noodle production methods [18]. All these methods involve increasing the viscosity of the starch. The three major ways in which the viscosity of the starch can be increased can be summarized as follows: (1) ferment, let it sit for a while; (2) repeated pounding; and (3) moderate heating. Therefore, the comparative tests for these three methods were designed thus:

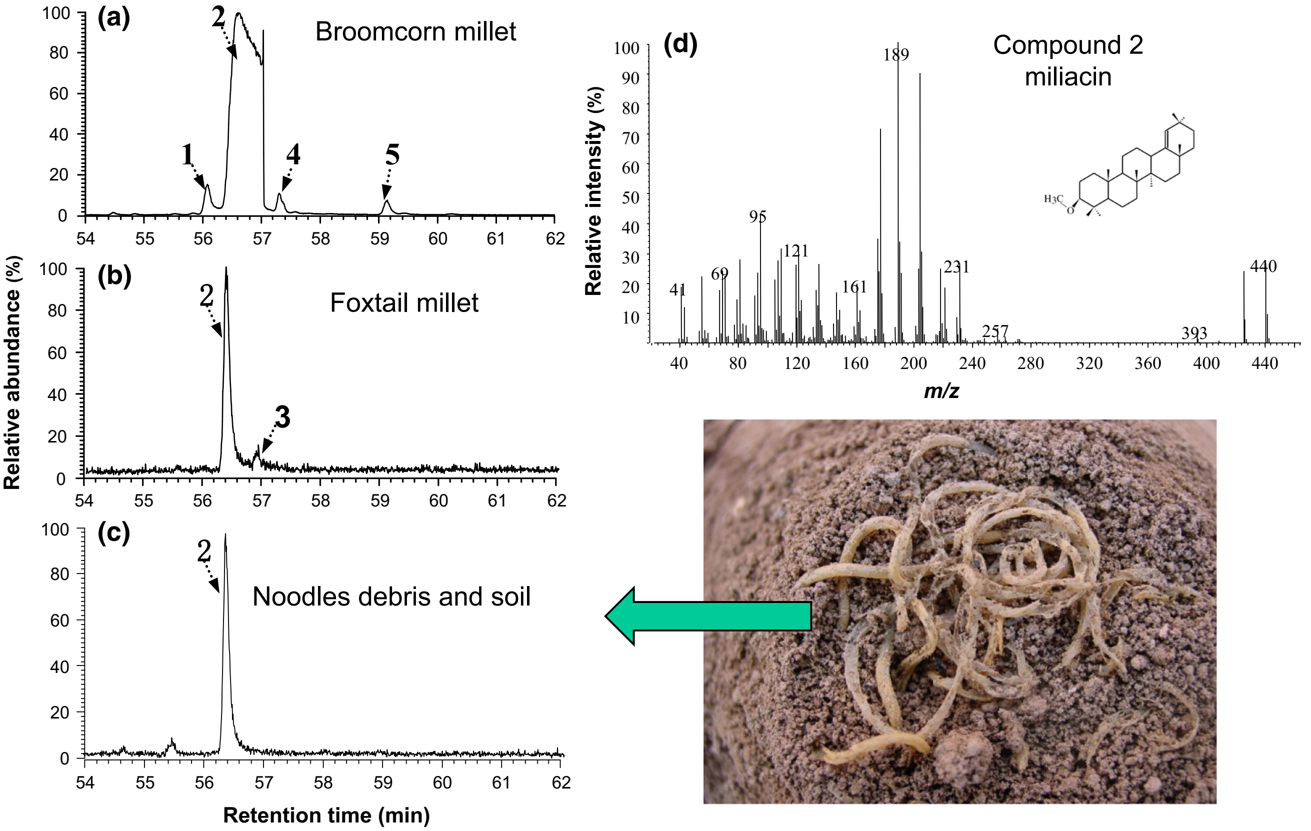

Fig. 8 (Color online) Aromatic ion chromatogram of broomcorn millet (**a**), foxtail millet (**b**) and a noodle debris and sediment sample from the Lajia site (**c**). Peaks 1, 2, 3, 4 and 5 labelled, respectively, as PTME-1[M^+440, *m/z*, 425, 393, 257, 218, 204, 189, 161, 135, 109 (100 %), 95, 69]; PTME-2 [miliacin,olean-18-en-3β-ol ME, M^+ 440, *m/z*, 425, 393, 257, 218, 204, 189 (100 %), 177, 161, 135, 109, 95, 69]; PTME-3 [M^+ 440, *m/z*, 425, 397 (100 %), 365, 261, 229, 218,204, 189, 175, 161, 135]; PTME-4 [α-amyrin ME, urs-12-en-3β-ol ME, M^+ 440, *m/z*, 393, 259, 218 (100 %), 203, 189, 161, 135, 109, 95] and PTME-5[M^+ 440, *m/z*, 425, 408, 393, 257, 221, 203, 189(100 %), 147, 135, 121, 109, 95]. **d** Showing the mass spectrometry of PTME-2 [miliacin, olean-18-en-3β-ol ME]

(1) Waiting for the ferment, let it sit for a while

Method 1: water (30 %–40 %) was added to the millet flour, dough was made by kneading by hand, and a *hele* noodle press was then used (as below) to extrude the noodles;

Method 2: water (30 %–40 %) was added to the millet flour, dough was made by kneading by hand, the dough surface was covered with a damp cloth (at 25 °C for 4 h), and a *hele* noodle press was then used to extrude the noodles;

(2) Repeated pounding

Method 3: water was added to the millet flour, dough was made by kneading, the material was pounded in a mortar for 20 min, and a *hele* noodle press was then used to extrude the noodles;

(3) Heating

Method 4: after the shelling millet had been cooked, they were pounded to form a thick paste, and once cooled, a *hele* noodle press was used to extrude the noodles.

Method 5: warm water (30–40 °C) was used to knead the millet dough, and a *hele* noodle press was used to extrude the noodles;

Method 6: once the millet dough had been kneaded, and after it had been steamed (dough of 8 cm diameter was steamed separately for 15 and 60 min), a *hele* noodle press was immediately used to extrude the noodles.

Table S3 shows the results of employing the *hele* noodle press to make noodles using the different millet dough methodologies. Both the production of a ferment and the repeated pounding were able to increase the viscosity of the starch and the shearing force of the dough, thereby increasing the length of the *hele* noodles, although there was a limit to the possible length increase and, after cooking, the noodles easily fractured, meaning that the length could not practically exceed 15–20 cm. Once the millet had been cooked, it was repeatedly pounded to form

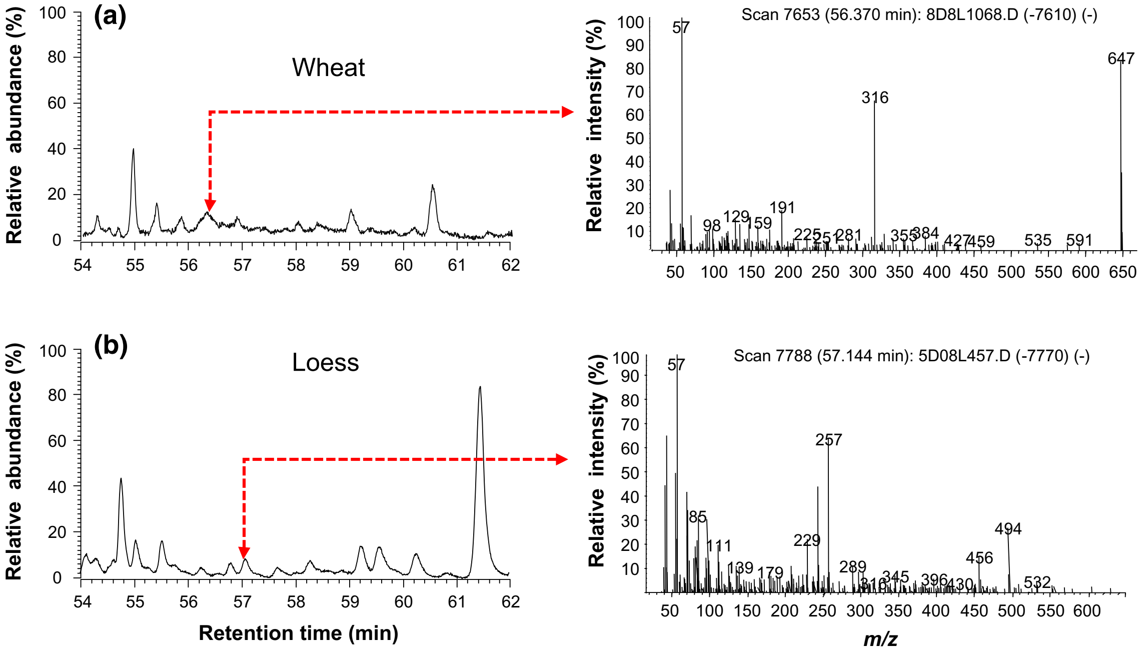

Fig. 9 (Color online) Aromatic ion chromatogram of modern wheat (**a**), loess sample (**b**). Mass spectra (right) showing the aromatic fraction is completely different with miliacin

a thick paste, once cooled, or after warm water had been added for kneading the millet dough, and the *hele* noodle press had been used to extrude the noodles, then the length of the noodles could also be increased, though not beyond 15–20 cm.

After the kneaded millet dough was steamed, a *hele* noodle press was used immediately to make the noodles. This method was able to increase significantly the length of the noodles; it becoming possible to squeeze noodles 30–50 cm long out of the press. However, if the dough was steamed for too long, achievable lengths declined.

4.4 The final simulation method

A comprehensive multi-step method for increasing starch viscosity was chosen for the manufacture of millet noodles, as follows: (1) 500 g of shell-bearing millet was taken, pounded in a mortar and shelled, about 20 %–30 % of husk residues; (2) after being soaked at room temperature for 12 h, it was pounded in a mortar to produce dough and left at room temperature for one hour for the ferment; (3) the dough was pounded for 2–3 h, then placed in a steamer and heated for approximately 15 min, and then, the dough was pounded again; and (4) the heat was used to extrude some noodles using a *hele* noodle press, and these were then placed in boiling water. Noodle length reached up to 120 cm (Fig. 10). Different millet types used yielded the same results.

In June 2011, China Central Television recorded this whole manufacturing process of noodle simulation for their programme "Forward Science" in the Palaeoecology Laboratory of the Institute of Geology and Geophysics, Chinese Academy of Sciences. We used a large-scale *hele* noodle press to extrude the noodles, producing noodles 520 cm in length. The link for the programme is: http://kejiao.cntv.cn/science/zoujinkexue/classpage/video/20110610/100115.shtml.

5 Discussion

5.1 The burial and survival of the noodles

When the pottery bowl, unearthed in room F20 at the Lajia site, was excavated in accordance with strict archaeological procedures [30], they were found to be buried facing downwards, with the open end of the bowls tilting down. This means that a discrete space could have existed in between the upper surface of the sediment frustrum and the bowl's base. This sediment within the bowl could have further contracted due to long-term sealing and oxygen loss, allowing the upper noodle surface to maintain its shape for 4,000 years. Both the photographic record of the noodles excavated at the site and the noodle fragments extracted from within the bowl in the room present a clear record of the existence of such noodles. Although the

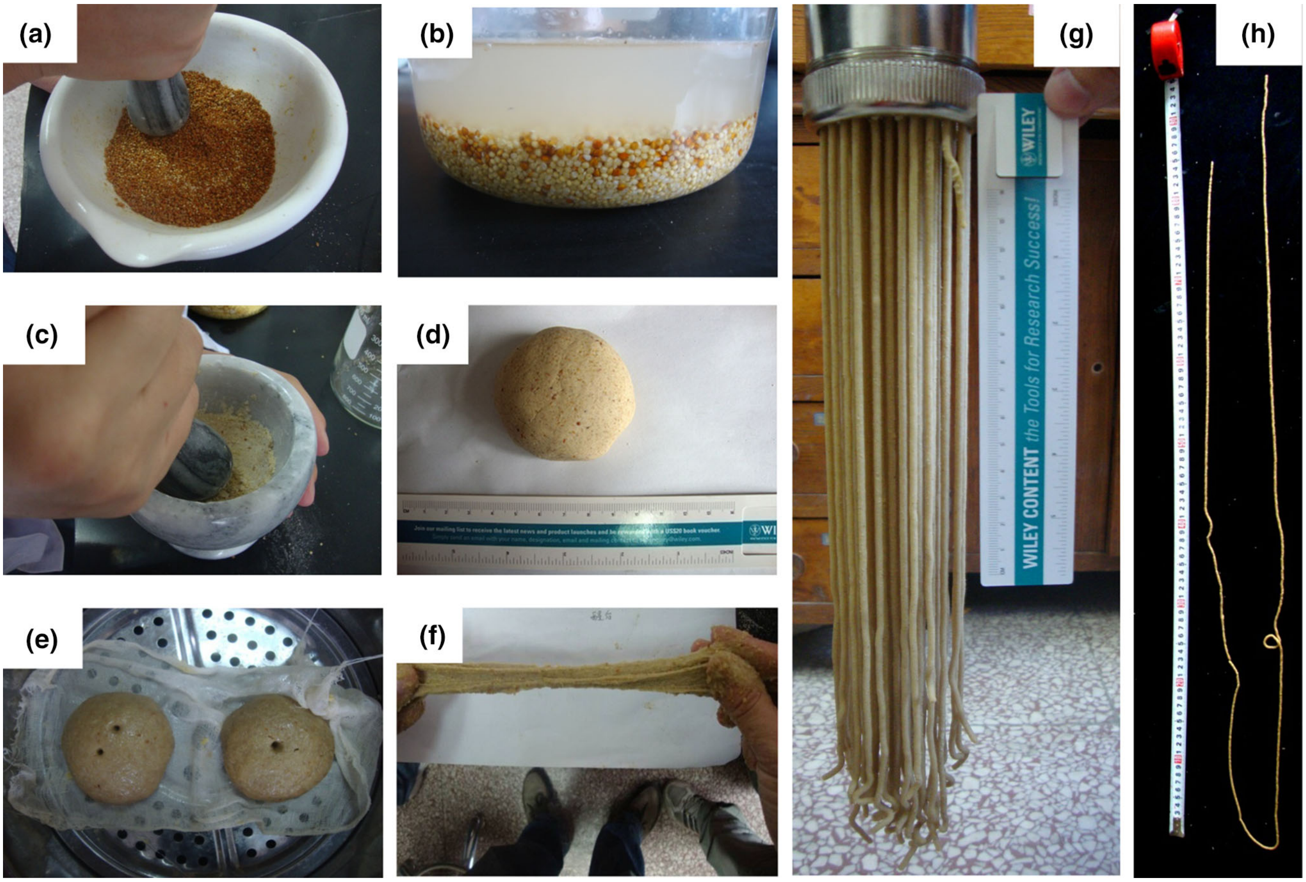

Fig. 10 Final noodle simulation method, with results. **a**–**f** Noodle simulation procedure; **g**, **h** noodle simulation results

noodles excavated from the upper surface of the sediment frustrum within the bowl appeared weathered and fragmented, there was also a great quantity of noodle fragments and ends within the noodle sediment strata at the bottom of the bowl; fine particles of noodle ingredients were also scattered throughout. These all provided material for analysis and research. They constitute an extremely rarely seen example of soil-sealed and extant noodle material following post-earthquake flooding [20] as well as a very occasionally discovered prehistoric noodle archaeological find.

Analysis of samples excavated from the site and examined in the laboratory showed that the soil that filled the mouth of the bowl and that buried outside the bowl have the same composition, i.e. they are relatively pure light brown–grey silty clay. However, they are not consistent with the composition of red clay layer, which is often layered or patchy distribution in the Lajia site. The earthenware bowls containing the noodles are very likely to have been in a major earthquake and then been successively filled by soil, flipped upside down, then buried by red clay and alluvial material. This situation appears the same in the many rooms excavated at the Lajia site. In some of the rooms, the excavated artefacts or the human remains appear to be covered, first of all, by a soil type similar in its composition to loess, then by red clay from sudden landslides [29, 39].

Analysis revealed that millet husk phytoliths, starch and millet hormone molecular markers were found only within the three sediment samples taken from the base of the bowl and that the same phytoliths and starch can also be found in the noodle fragment samples. However, these components were not found in other three soil samples from the mouth of the bowl or in those comparative soil samples taken from the excavation site cross section and from loess. It was clear from the analysed foxtail millet and broomcorn millet husk phytoliths, starch and millet hormone molecular markers that they were not from a filling that was imported from outside but from the noodles which were there at the time.

5.2 The gelatinization and extinction of starch

A wealth of starch grains was discovered in all the noodle fragment samples analysed. Some of the starch grains in the noodle fragments had been shattered or gelatinized, and their extinction features and ornamentation already blurred by gelatinization. Nonetheless, the residual starch grains still possessed clear outlines. The morphology of some of the starch grains within the noodle fragments was apparent, and some of the starch grains extracted from the noodle sediment strata had relatively clear cruciform extensions. Millet starch surficial radiation patterns could also be seen often on the surface of some starch grains (Fig. 6c).

In addition to using a polarizing microscope to observe and identify these starches, Congo Red dyeing, fluorescence microscopy and many other simple iodine dyeing methods can be employed [40–42]. These allow the starch grains within the noodle fragments, as opposed to other mineral grains, to be easily determined. There is no need to use any analytical methods [such as scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS)] to infer whether or not grains are minerals. Some scholars consider that both “gypsum sphaerocrystal”and “fungal spores” have cruciform extinction [43]; in fact, gypsum crystals are monoclinic and cannot form sphaerocrystal. Out of all the minerals found naturally, these can be grouped into three major crystal categories, seven major crystal systems and 32 crystal types; none of these can form sphaerocrystal. Even isometric crystal minerals, there was no comprehensive crossextinction. The so-called gypsum sphaerocrystal exists nowhere in the natural world. The sphaerocrystal that is found either in starch or in polymers (e.g. polyvinyl chloride polymers) is crystalline in form, so, of course, PVC molecular particles will not respond to gelatinization or iodine staining. Fungal spores display clear spore walls, decoration, colour, hypha and transparency; starch does not have features like this, such as external walls. It is therefore very easy to differentiate starch grains from spores under an ordinary optical microscope, although this is an essential skill for those who work on identifying microfossils under the microscope. As yet, only a small quantity of fresh <3-μm spores with cruciform extinction has been found (these may have radial inclusions), and once these spores have decayed and died, they do not extend outward. Further, starch grains that can be identified are all 3 μm and above in size; the starch grains discovered in our noodle residue were, on average, above 8.2 μm.

Looking at the profile of noodle starch extenuation structures and any damage to the edges of the starch, the noodles must have experienced a heating process. Part of the starch retains its cruciform extinction; the most reasonable and logical explanation thus has to be that the starch in the noodles had still not completely gelatinized. The reasons for this lack of total gelatinization could be manifold or solitary.

Gelatinization occurs where there is sufficient moisture. When it is heated to a certain temperature, swelling and cracking occur to form a uniform gelatin paste. Starch gelatinization usually requires three stages from start to finish (the reversible absorption, irreversible absorption and particle dissolution stages). The temperature range from the start of gelatinization to its conclusion is usually 5–20 °C. For example, the temperature range for corn gelatinization is 62–84 °C. Gelatinization commences at 62 °C and, once 84 °C has been reached, 98 % of the colour interference of the starch grains has disappeared (this 98 % is therefore used as a marker for the conclusion of the gelatinization process). Since the starch grains are not completely gelatinized between 62 and 84 °C, they retain some of their colour interference. This, along with the intensity in brightness of the starch’s cruciform extinction, can logically be used in any discussion of the extent of starch gelatinization.

There may be many reasons why the starch has not been completely gelatinized when it has been in contact with moisture, salt or an alkaline solution, whether or not fat and/or protein substances are present [44]. (1) Starch moisture content. When starch moisture content falls below 50 %–60 %, it is difficult for heating to cause total gelatinization [45]. Under extreme conditions, when there is no water involved, the starch can be heated to 200 °C without any change occurring [46]. Testing by Jang and Pyun [47] showed that when wheat starch was heated to 212 °C, its colour changed and, at 257 °C, its cruciform extinction had completely disappeared. Recently, Raviele [48] heated food waste to 232 °C and was still able to identify natural starch granules. These phenomena could be attributable to the lack of sufficient water molecules, making it almost impossible for the starch to be completely gelatinized. (2) The presence of salt and alkaline solutions and/or fat and proteins can all significantly increase the temperature at which starch gelatinizes. Salts can breakdown the aqueous membrane on the surface of the starch, reducing any moisture’s ability to act, thus causing the gelatinization temperature to rise. Experiments have shown that, by adding a 7 %–9 % cooking salt solution, this can facilitate the starch gelatinization temperature to increase by approximately 18 °C [49]. By adding a 1 % calcium hydroxide (hydrated lime) solution, the starch gelatinization can usually be raised by approximately 10–15 °C [46]. Amylose can form compounds with stearate, meaning that gelatinization does not occur when it is heated to at least 100 °C.

Despite there being at present, no clarity over the addition of salt and/or electrolytes to the noodles found at the Lajia site; however, the analysis of the noodle samples uncovered a great quantity of fats and vitamins (to be published). Although, currently, there are no methods for verifying whether these ingredients have been added during the kneading process, explaining the composition of the noodles and/or their solutions is not at all simple. The existence of these complex ingredients can increase the gelatinization temperature of the noodle starch. In addition, considering the influence of the comparative height above sea level (a.s.l.) of the Lajia site, and the opportunities for regrowth of starch that a history of 4,000 years has provided, namely supporting starch retrogradation through starch chain recrystallization [50], both the factors are able to retain and/or restore part of the cruciform structure.

We tested for the survival of starch and phytoliths after the steaming of the simulated noodles (Fig. S2) when we had added no salt, alkaline solution or fatty solvents whatsoever. We took samples for analysis 3–5 min after we had heated the boiling water and, as Figure S2 shows, the great majority of noodle starch granules had lost their cruciform structure (red point solenoid). We also discovered that some of the starch had reunited in tapered and radial cruciform structures. This was possibly because the pressure had brought lumps of starch together, creating its gelatinization temperature to rise, unlike situations when starch can easily obtain moisture from within a solution. Approximately 20 %–30 % of modern foxtail millet and broomcorn millet starch granules appear to have a round (or spherical) or nearly round profile, and 70 %–80 % are irregular, polygonal spheres. Gelatinization testing [42] has revealed that when the gelatinization temperature is between 60 and 90 °C, the edges of the starch grains gelatinize gradually into a rounded shape, and within 5–20 min of heat being applied, the starch has gradually lost its cruciform characteristics.

In fact, more often than not, no gelatinized starch granules were discovered at archaeological sites which used pottery for steaming [46] or where ancient foodstuffs were steamed [51]. As Crowther [46] has pointed out, “Starch gelatinization can arise from the heating applied during cooking, but heating applied during cooking does not necessarily lead to starch gelatinization”. Thus, some of the starch in the excavated noodles had a regular, cruciform extinction structure; however, we have no way of verifying that this bowl of noodles had definitely been thoroughly cooked and that the noodles had been completely gelatinized. Some archaeologists are even inclined to regard this bowl of noodles as an offering; the shape and aesthetic appearance of an offering would have been vitally important, or perhaps, it did not need to be cooked so thoroughly.

Starch is a semi-crystalline compound granule formed from amylose and amylopectin. Crystallized and non-crystallized areas surrounded an umbilical point in alternate permutations; the crystallized areas with a double-helix amylopectin formation, forming inseparably tight starch granular strata, and the non-crystallized areas formed thinly spread starch granular strata. Both of these, arranged alternately, formed a ring-like structure. The theory states that a high-resolution microscope is able to obtain clearer images of those starch granules which have a ring-like composition. However, owing to different starch having different double-helix configurations, their ring-strata compositions will appear dissimilar. In general, the incidence of ring-strata in wheat starch appears relatively high, but other starch, once they have undergone physical or chemical change, can also exhibit clear ring-strata compositions. Sometimes only hot water, an extensive period of immersion in cold water, or a rarified acid solution or other electrolyte solution applied slowly, need be used to bring out such ring-strata compositions.

Until now, basic research into the evaluation of starch granule morphology has been extremely limited. In particular, following gelatinization or semi-gelatinization, the polygonal features of millet starches are often broken up, and the outlines of the edges of the starch granules appear more rounded, semi-rounded or irregular. Such starch granules have been unable to accurately identify the plant species. Although semi-gelatinized starch granules in size at the Lajia noodle, smaller than wheat or barley starch, and similar to the millet starch, however, we cannot in any way take these as sufficient evidence in our appraisal of the noodle ingredients. Nevertheless, they do provide direct material evidence of noodle starch and, together with the mutually linked evidence provided by the phytoliths and molecular markers, they prove that foxtail millet was the principal ingredient of the noodles with a minor broomcorn millet.

5.3 Millet noodles can be made

In our earliest report, we introduced this noodles as resembling “La-Mian” (“hand-pulled”) noodles in shape [1], but we did not assert that they were *la-mian* noodles because the millet did not have a viscous glutinous protein. Naturally, there is no way of making noodles employing a *la-mian* noodle approach. Using the wrong method to test the veracity of existing results can often lead to incorrect understanding. Our simulation experiment was based upon knowledge of how people enhanced viscosity through pounding flour in a mortar then heating it up in hot water. It made use of *hele* noodle equipment and employed, as a reference, the method of shaping noodles by extruding gelatinized flour gel [52–54]. In conditions where no extra viscosity was added, millet noodles were manufactured, proving that noodles could have been made from millet.

In fact, there were many types of sticky foxtail millet grown in ancient China but, because their production volumes were not high, there are now small amount of planting in northern China and the surrounding mountainous regions. Figure S3a shows sticky foxtail millet harvested in the hilly area of Xunzhong County in Liaoning

Province from which it is much easier to make *hele* millet noodles (Fig. S3). In line with the National Standard GB8232-87 of the People's Republic of China, the two major foxtail millet types grown are non-glutinous foxtail millet and glutinous foxtail millet (also called sticky millet). Currently, close to 30 types of sticky foxtail millet is being grown in the testing plot of the Chinese Academy of Agricultural Sciences in the suburbs of Beijing (Fig. S3). Although we still do not understand whether the foxtail millet grown by the Qijia culture at the Lajia site was sticky millet or not, these traditional crops still exist and so have provided us with plentiful opportunities for simulation and interpretation.

Archaeological investigations in Subeixi Cemetery in Xinjiang have discovered 2,400-year-old complete noodles made using a millet-type substance (broomcorn millet) [16]. New archaeological evidence further suggests to us that noodles could well have been made from a sticky millet-type material.

Folklore holds that, in the villages in Henan, Hebei and Shanxi provinces involved in our investigation, there remain even today very many places which make *hele* millet noodles using a wooden "bedding" rack above the stove, whereby the dough is squeezed into the holes in the bottom part of the *hele* noodle rack. Operators then apply pressure to the *hele* noodle rack's wooden handle, and the *hele* millet noodles are produced (Figs. S4a, 4b). Figure S4 shows both traditional millet *hele* noodle manufacturing devices and the manufacturing process. The shape of the manufactured millet *hele* noodles is very similar to those noodles excavated at the Lajia site (Fig. S4c). The local television station in Hebi City in Henan Province has produced a special report on the famous Xun County snack—foxtail millet *hele* noodles which can be viewed on the link http://www.hebitv.com/a/hebixinwen/2012/0204/6452.html.

In fact, when using wheat flour to make *la-mian* noodles, it is usually necessary to add a small quantity of Peng Barilla ash to produce the viscosity required for extrusion. At present, there is no evidence to suggest that the viscosity-enhancing ingredients were added to the noodles from the Lajia site. The comprehensive evidence from folklore, archaeology and the results of our simulation would all suggest that, regardless of whether or not glutinous ingredients are added, foxtail millet can be used to make noodles. Even though, as in the simulation, ancient peoples were unable completely to shell the cereal (20 %–30 % of the millet remained unshelled), it is now possible with the latest simulation methods to produce noodles longer than 5 m. In reality, in our investigations into folk practices, these husks are also called grain chaff and are very rich in nutrients, being an important source of food in periods of difficulty in the countryside. Before the 1970s, the rural population of Ying County in Shanxi Province often crushed unshelled cereals to make different foodstuffs.

Using millet starch or other starches which lack the viscosity of glutinous wheat protein to make noodles has become as important in the modern food industry as the industrial production of corn starch noodles [9], sorghum starch noodles [12, 13], broomcorn millet starch noodles [15, 16] and foxtail millet starch noodles [14]. There has already been a good deal of research into this subject, ranging from theory to methodology. The method for producing extruded gelatinized noodle gel [54] is already being used as standard in the production of starchy noodles. These grain starches include an abundance of nutrients and are also suitable for the "gluten intolerant" (coeliac disease) sector of the population [55], becoming a widely consumed foodstuff. There are grounds for believing that millet can be made not only into simple noodles, but also into a diverse variety of noodles.

5.4 Noodles as representative of Chinese culinary culture

The noodles found at the Lajia site provide by far the oldest known physical evidence of noodles. Although people customarily place the source where the earliest site is found, it is perfectly possible that noodles were around at an even earlier date. So where is the source of the earliest noodles? Some scholars have attempted to determine the geographical origins of noodles by tracing the crops used in the manufacture of noodles. For example, Dr. Li Zhengxu, in the so-called documentary "Noodle Road", broadcast by the Korean Broadcasting Service (KBS), ignored the 4 ka evidence provided by the noodles found at the Lajia site as well as the circa 2.4 ka-old broomcorn millet noodles unearthed in the Subeixi Cemetery in Xinjiang [16]; it was maintained, incorrectly, that they were wheat noodles and that the wheat had originated in the Middle East, concluding that noodles originated in the Middle East.

Ancient Chinese people learnt, at a very early stage, how to cultivate drought-resistant foxtail millet and broomcorn millet crops. Systematic material evidence for sources of foxtail millet and broomcorn millet in China already exists [24, 27]. The Qijia culture evident at the Lajia site was part of the Yellow River civilization, and the millet noodles excavated there have undoubtedly increased the likelihood that noodles originated in China. There is a multiplicity of varieties of noodle in China, a history which stretches back into the dim and distant past. Specific *hele* noodle production methods are similar to those described by Jia Sixie in the "compacting powder method" of "Pastry Method No. 82" from his "*Qi Min Yao Shu*" (533–544 CE) [17]. The many types of noodle from about 4,000 a BP, the Lajia Noodle House to the present day, the

Chinese have shown that they are good at adapting foodstuffs to yield innovative cooking and, as a result of these innovations, noodles have become not only the standard bearer of Chinese cuisine, but they have also made a unique contribution to global culinary culture.

Of course, the question as to which people invented noodles can be considered unimportant; it may also be considered to be a discovery symptomatic of an historical convergence of developments in human culinary practices. Regardless of whether noodles originated in China or elsewhere, their emergence was most probably even earlier than previously thought, and only a serendipitous twist of fate may unearth their remains, although such a probability is likely to be very small.

6 Conclusion

This paper documents the process of excavation of earthenware noodle bowls at the Lajia archaeological site, as well as the gathering of samples of earthenware bowls filled with noodles and sediment and the subsequent analytical methods. It describes the comprehensive analysis of the phytoliths, starch, molecular markers and other ingredients in unearthed residues and noodle fragment, thus proving that ancient humans at the Lajia site used millet as their principal ingredients in the manufacture of noodles. We further to use the extrusion gelatinization moulding method, reproducing millet noodles similar in composition and shape to the excavated noodles. The noodles from the Lajia site bring to life a rare find of ancient food remains and make an important contribution towards the study of the eating and drinking habits of the ancients. It should be noted that fat and other ingredients have already been extracted from the earthenware noodle bowls and still need further to study.

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Conflict of interest The authors declare that they have no conflict of interest.

References

1. Lu H, Yang X, Ye M et al (2005) Culinary archaeology: millet noodles in Late Neolithic China. Nature 437:967–968
2. Fu BX (2008) Asian noodles: History, classification, raw materials, and processing. Food Res Int 41:888–902
3. Hou G (2001) Oriental noodles. Adv Food Nut Res 43:141–193
4. Liu JJ, He ZH, Zhao ZD et al (2001) Review of noodle industrial quality of wheat. J Triticeae Crops 21:81–84 (in Chinese)
5. Tan HZ, Li ZG, Tan B (2009) Starch noodles: history, classification, materials, processing, structure, nutrition, quality evaluating and improving. Food Res Int 42:551–576
6. Lii CY, Chang SM (1981) Characterization of red bean (*Phaseolus radiatus* var. Aurea) starch and its noodle quality. J Food Sci 46:78–81
7. Nwabueze T, Anoruoh G (2011) Evaluation of flour and extruded noodles from eight cassava mosaic disease (CMD)-resistant varieties. Food Bioprocess Technol 4:80–91
8. Kim YS, Wiesenborn DP, Lorenzen JH et al (1996) Suitability of edible bean and potato starches for starch noodles. Cereal Chem 73:302–308
9. Yuan ML, Lu ZH, Cheng YQ et al (2008) Effect of spontaneous fermentation on the physical properties of corn starch and rheological characteristics of corn starch noodle. J Food Eng 85:12–17
10. Bhattacharya M, Zee SY, Corke H (1999) Physicochemical properties related to quality of rice noodles. Cereal Chem J 76:861–867
11. Han JA, Seo TR, Lim ST et al (2011) Utilization of rice starch with gums in Asian starch noodle preparation as substitute for sweet potato starch. Food Sci Biotechnol 20:1173–1178
12. Suhendro EL, Kunetz CF, McDonough CM et al (2000) Cooking characteristics and quality of noodles from food sorghum. Cereal Chem J 77:96–100
13. Liu L, Herald TJ, Wang D et al (2012) Characterization of sorghum grain and evaluation of sorghum flour in a Chinese egg noodle system. J Cereal Sci 55:31–36
14. Huang SJ, Wu YW (2010) A study and produce on millet noodle. Food Ind 1:71–72 (in Chinese)
15. Taylor JRN, Schober TJ, Bean SR (2006) Novel food and non-food uses for sorghum and millets. J Cereal Sci 44:252–271
16. Gong Y, Yang Y, Ferguson DK et al (2011) Investigation of ancient noodles, cakes, and millet at the Subeixi Site, Xinjiang, China. J Archaeol Sci 38:470–479
17. Jia SX (1982) 6th century: Qimin Yaoshu. China Agriculture Press, Beijing (in Chinese)
18. Hou GG (2011) Asian noodles: science, technology, and processing. Wiley, New York
19. Zhang QH (1998) The ancient noodles. Cook Knowl 9:32–33 (in Chinese)
20. Xia ZK, Yang XY, Ye ML (2003) Pre-historical disasters at Lajia site in Qinghai. Chin Sci Bull 48:1200–1204 (in Chinese)
21. Ye ML (2002) The excavation of the prehistorical Lajia site in Minhe County, Qinghai Province. Archaeology 7:3–5 (in Chinese)
22. Lu H, Zhang J, Wu N et al (2009) Phytoliths analysis for the discrimination of foxtail millet (*Setaria italica*) and common millet (*Panicum miliaceum*). PLoS One 4:e4448
23. Zhang J, Lu H, Wu N et al (2011) Phytolith analysis for differentiating between foxtail millet (*Setaria italica*) and green foxtail (*Setaria viridis*). PLoS One 6:e19726
24. Lu H, Zhang J, Liu KB et al (2009) Earliest domestication of common millet (*Panicum miliaceum*) in East Asia extended to 10000 years ago. Proc Natl Acad Sci USA 106:7367–7372
25. Piperno DR (1988) Phytolith analysis: an archaeological and geological perspective. Academic Press, San Diego
26. Piperno DR, Ranere AJ, Holst I et al (2000) Starch grains reveal early root crop horticulture in the Panamanian tropical forest. Nature 407:894–897

27. Yang X, Wan Z, Perry L et al (2012) Early millet use in northern China. Proc Natl Acad Sci USA 109:3726–3730
28. Jacob J, Disnar JR, Arnaud F et al (2008) Millet cultivation history in the French Alps as evidenced by a sedimentary molecule. J Archaeol Sci 35:814–820
29. Ye ML, Ren XY, Wang GD et al (2004) The discovery of the earthen altar and the pile-dwelling of the Qijia culture in Lajia site, Minhe County, Qinghai Province. Archaeology 6:3–6 (in Chinese)
30. Ye ML (2006) Decoding the secret of the thousand-year-old noodles. Encycl Knowl 4:46–49 (in Chinese)
31. Yang X, Zhang J, Perry L et al (2012) From the modern to the archaeological: starch grains from millets and their wild relatives in China. J Archaeol Sci 39:247–254
32. Piperno DR (2009) Identifying crop plants with phytoliths (and starch grains) in Central and South America: a review and an update of the evidence. Quat Int 193:146–159
33. Piperno D (2006) Phytoliths: a comprehensive guide for archaeologists and paleoecologists. AltaMira, New York
34. Lu HY, Wu NQ, Yang XD et al (2006) Phytoliths as quantitative indicators for the reconstruction of past environmental conditions in China I: phytolith-based transfer functions. Quat Sci Rev 25:945–959
35. Piperno DR, Ranere AJ, Holst I et al (2009) Starch grain and phytolith evidence for early ninth millennium BP maize from the Central Balsas River Valley, Mexico. Proc Natl Acad Sci USA 106:5019–5024
36. Yang X, Yu J, Lu H et al (2009) Starch grain analysis reveals function of grinding stone tools at Shangzhai site, Beijing. Sci China Ser D Earth Sci 52:1164–1171
37. Ball TB, Gardner JS, Anderson N (1999) Identifying inflorescence phytoliths from selected species of wheat (*Triticum monococcum*, *T. dicoccon*, *T. dicoccoides*, and *T. aestivum*) and barley (*Hordeum vulgare* and *H. spontaneum*) (Gramineae). Am J Bot 86:1615–1623
38. Berlin AM, Ball T, Thompson R et al (2003) Ptolemaic agriculture, “Syrian Wheat”, and “*Triticum aestivum*”. J Archaeol Sci 30:115–121
39. Yin ZQ, Qin XG, Zhao WJ et al (2013) Reexploration on the formation mechanism of the red clay layer in the Guanting basin of the upper reaches of Yellow River. Quat Sci 33:995–1004 (in Chinese)
40. Lamb J, Loy T (2005) Seeing red: the use of Congo Red dye to identify cooked and damaged starch grains in archaeological residues. J Archaeol Sci 32:1433–1440
41. Bailey J, Whelan W (1961) Physical properties of starch I. Relationship between iodine stain and chain length. J Biol Chem 236:969–973
42. Zheng Q, Wang C (2013) Starch evidence of glutinous plant content as the additive to Ming Great Wall mortar at Gubeikou, Beijing. Quat Sci 33:575–581 (in Chinese)
43. Ge W, Liu L, Chen XC et al (2010) Can noodles be made from millet? An experimental investigation of noodle manufacture together with starch grain analyses. Cult Relics South Chin 4:26–32 (in Chinese)
44. Haslam M (2004) The decomposition of starch grains in soils: implications for archaeological residue analyses. J Archaeol Sci 31:1715–1734
45. Hu Q, Meng YC (2004) A review on the gelatinization of rice starch and aging. Food Res Dev 25:63–66 (in Chinese)
46. Crowther A (2012) The differential survival of native starch during cooking and implications for archaeological analyses: a review. Archaeol Anthropol Sci 4:221–235
47. Jang J, Pyun Y (1996) Effect of moisture content on the melting of wheat starch. Starch Stärke 48:48–51
48. Raviele ME (2011) Experimental assessment of maize phytolith and starch taphonomy in carbonized cooking residues. J Archaeol Sci 38:2708–2713
49. Wootton M, Bamunuarachchi A (1980) Application of differential scanning calorimetry to starch gelatinization. III. Effect of sucrose and sodium chloride. Starch Stärke 32:126–129
50. Yang YC (2009) The process and influence factor of the gelatinization in starch. Agric Process 2:18–19 (in Chinese)
51. Samuel D (1996) Investigation of ancient Egyptian baking and brewing methods by correlative microscopy. Science 273:488–490
52. Meng Y, Liu X, Chen J (2011) Relationship between rice components, stress-relaxation and instant rice noodles quality. Trans Chin Soc Agric Eng 27:375–382
53. Jin ZY (2005) Extrusion and post extrusion coating in feed manufacture (1). Feed Chin 23:31–34 (in Chinese)
54. Wei YF (2009) Research progress of extrusion technology. Mech Rural Pastoral Areas 6:15–16 (in Chinese)
55. Li YG, Xing RR, Li SP (2012) Cloning and sequence analysis of new α-gliadin genes from Triticum monococcum. J Triticeae Crops 32:387–392 (in Chinese)

Supplementary Materials for

Component and simulation of the 4000-year-old noodles excavated from the archaeological site of Lajia in Qinghai, China

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This supplementary file includes:
Table S1 to S3
Figs. S1 to S4

Table S1 Starch particle sizes of modern cultivated plants and prehistoric noodles

	Average length (±s.d.) (μm)	Range (μm)*	Count
(a) Modern cultivated plant starch from the research area			
Corn (*Zea mays* L.)	15.7±4.5	5-30	130
Potato (*Solanum tuberosum* L.)	38.5±11.1	15-100	130
Barley (*Hordeum vulgare* L.)	18.4±8.7	3-38	150
Highland barley (*Hordeum vulgare* L.*var.nudum* Hook.)	16.8±5.9	2-34	150
Wheat (*Triticum aestivum* L.)	15.7±6.1	2-33	150
Sorghum (*Sorghum bicolor* (L.) Moench)	14.8±3.9	5-25	110
Rice (*Oryza sativa*)	5 ±?	<2-10	120
Foxtail millet (*Setaria italica* (L.) Beauv.)	8.6±2.0	5-18	110
Green foxtail (*Setaria viridis* (L.) P. Beauv.)	5.5±2.9	2-14	120
Broomcorn millet (*Panicum miliaceum* L.)	7.8±2.1	6-16	110
(b) Prehistoric noodle starch			
Noodle fragment and sedimentary material starch	8.2±2.8	4-16	150

s.d. standard deviation starch grain length. * range in starch grain size measurements

Table S2 ^{14}C dating and calendar calibration of samples excavated from the Lajia site

Lab. No.[a]	Stratum from which sample unearthed	Material age	Libby ^{14}C age[b] and its 1-sigma error based on $t_{1/2}$ 5568 y	Tree-ring corrected age (Cal BC)[c]	
				1σ（68.2% confidence）	2σ（95.4% confidence）
ZK－3132	H18	Charcoal	3574±73	2029 (54.8%) 1873 1844 (7.7%) 1815 1800 (5.7%) 1778	2136 (94.8%) 1742 1709 (0.6%) 1701
ZK－3133	H20	Charcoal	3685±42	2139 (66.0%) 2022 1990 (2.2%) 1985	2199 (7.4%) 2164 2152 (88.0%) 1951
ZK－3134	M3	Charcoal	3637±75	2133 (16.1%) 2081 2061 (52.1%) 1910	2268 (0.4%) 2260 2206 (89.2%) 1863 1851 (5.8%) 1772
ZK－3137	H33	Charcoal	4200±107	2905 (19.1%) 2829 2824 (49.1%) 2628	3084 (0.9%) 3065 3028 (94.5%) 2481
ZK－3179	T537 H45	Charcoal	3746±48	2272 (4.4%) 2259 2207 (43.1%) 2123 2092 (20.7%) 2043	2296 (94.9%) 2021 1992 (0.5%) 1984
ZK－3180	T537 H45(2)	Charcoal	3740±42	2204 (45.0%) 2124 2091 (23.2%) 2044	2286 (95.4%) 2028
ZK－3181	T539 H48	Charcoal	3828±43	2396 (2.5%) 2386 2346 (65.7%) 2201	2458 (90.4%) 2196 2172 (5.0%) 2146
ZK－3182	T539 H48 (2)	Charcoal	3846±43	2435 (4.9%) 2421 2404 (9.0%) 2379 2349 (34.4%) 2273 2258 (20.0%) 2208	2463 (95.4%) 2200
ZK－3220	H44	Charcoal	3684±41	2138 (67.2%) 2023 1988 (1.0%) 1986	2198 (6.7%) 2166 2151 (88.7%) 1951
ZK－3221	H49	Charcoal	3764±38	2278 (13.2%) 2251 2229 (3.4%) 2221 2211 (51.6%) 2135	2294 (80.8%) 2116 2100 (14.6%) 2038
ZK－3222	T529J2 (3)	Charcoal	3800±56	2337 (3.7%) 2323 2308 (64.5%) 2140	2459 (90.0%) 2128 2089 (5.4%) 2047
ZK－3223	H41	Charcoal	3778±66	2299 (58.8%) 2129 2088 (9.4%) 2048	2457 (4.0%) 2417 2409 (91.4%) 2030
ZK-3632	2000QMLF3I	Human bone	3565±25	1944 (68.2%) 1887	2014 (2.7%) 1998 1979 (86.5%) 1876 1842 (3.9%) 1820 1796 (2.2%) 1781
ZK-3635	2000QMLF4Ⅳ	Human bone	3580±20	1950 (68.2%) 1896	2011 (3.8%) 2000 1978 (91.6%) 1886
BA110817	F4-Ⅹ	Human bone	3575±40	2010 (4.3%) 2001 1977 (63.9%) 1884	2032 (83.5%) 1869 1847 (11.9%) 1776
BA110818	F4-Ⅶ	Human bone	3580±25	1956 (68.2%) 1891	2022 (8.9%) 1991 1984 (86.5%) 1882
BA110819	F4-Ⅺ	Human bone	3555±40	1955 (52.8%) 1876 1842 (9.1%) 1820 1797 (6.3%) 1781	2020 (5.1%) 1993 1983 (90.3%) 1768

Notes:

(a) ZK= Radiocarbon Dating Laboratory of the Archaeology Research Institute of the Chinese Academy of Social Sciences BA= Beijing University Accelerator Mass Spectrometry (AMS) radiocarbon dating tests

(b) Age according to conventional or ‘Libby’ radiocarbon dating measured to 1950CE. The half life of the ^{14}C used was 5568 years.

(c) Calendar age calibration is performed using the OxCal v4.2.3 program (Christophe, 2009), and the IntCal13 calibration curve (Reimer et al., 2013).

References

1 Christopher BR (2009) Bayesian analysis of radiocarbon dates. Radiocarbon 51: 337-360.

2 Reimer PJ, Bard E, Bayliss A et al (2013) IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55: 1869-1887.

Table S3 Changes in noodle length versus different production methods

Method	Results and details of noodle manufacture	See
1	5—10-cm-long extruded noodles, breaking up easily after cooking into 1—2-cm-long pieces	Fig.S1d
2	Up to 15-cm-long extruded noodles, breaking up easily after cooking into 2—5-cm-long pieces	Fig.S1e
3	30-cm-long extruded noodles, breaking up easily after cooking into 15—20-cm-long pieces	Fig. S1f
4	Breaking up easily after cooking, with 5 cm lengths commonly seen, up to approximately 10 cm	Fig. S1g
5	More than 40-cm-long extruded noodles, appearing after cooking as 15—20 cm long	Fig. S1h
6	More than 50-cm-long extruded noodles steamed for 15min, appearing after cooking as >30 cm long; the same steamed for 60 min, appearing after cooking as 20 cm long or less	Fig. S1i
Comprehensive method	Noodle lengths in excess of 120 cm	Fig.10-ª

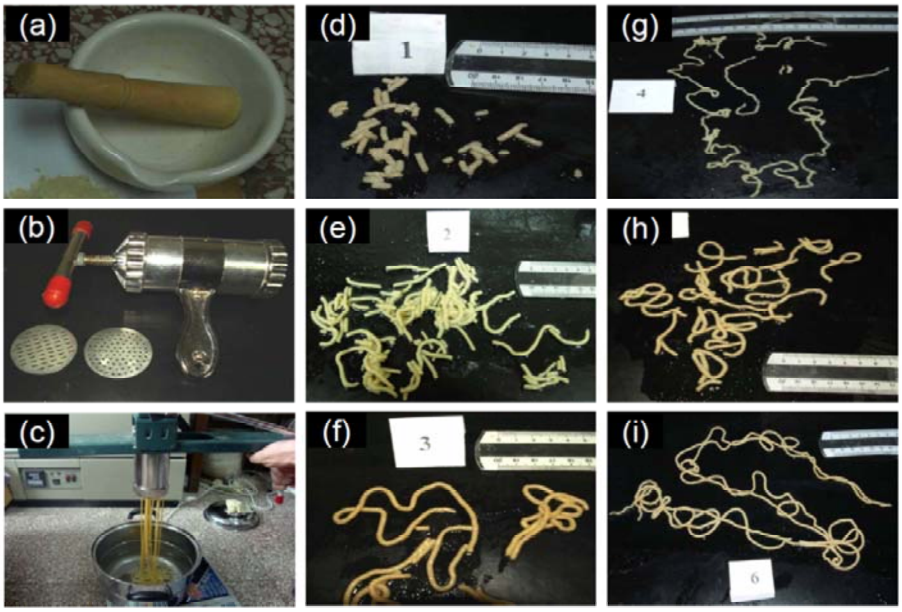

Fig. S1 Results of different noodle simulation methods. **(a)-(c)**, Equipment used in the noodle simulation trials; **(d)-(I)** results of different noodle simulation methods

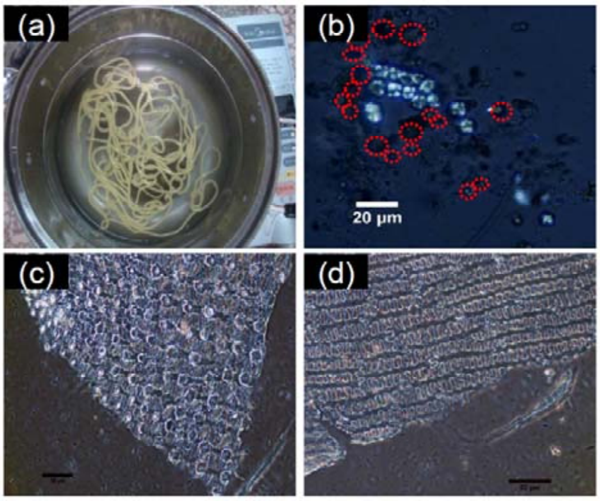

Fig. S2 Phytoliths and starch extant in the simulated noodles. **(a)** Analog heated-noodles; **(b)** analog-cooked noodle starch; **(c) – (d)** analog-cooked noodle phytoliths

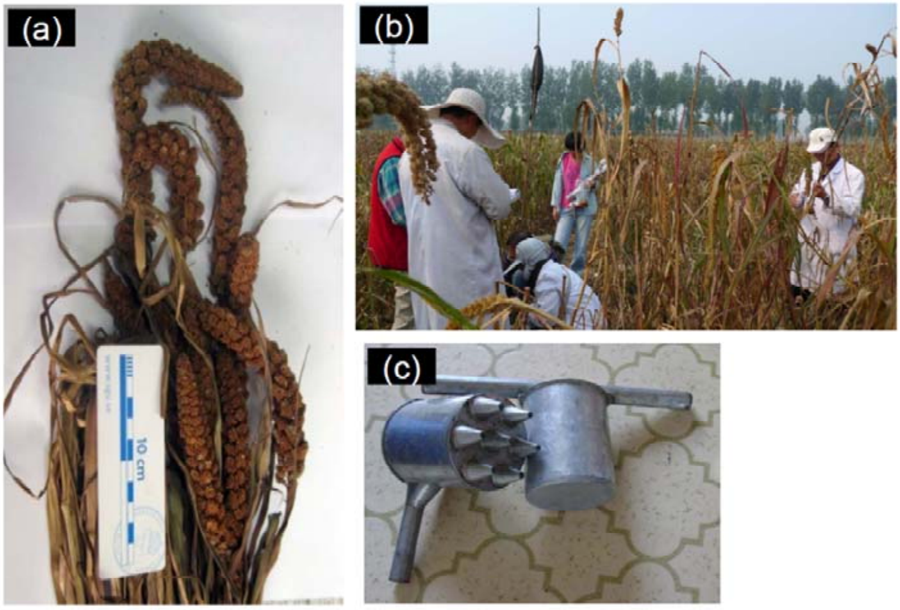

Fig. S3 Modern sticky millet sampling and a corn noodle *hele* machine. **(a)** Sticky millet collected from Suizhong County, Liaoning Province; **(b)** Chinese Academy of Agricultural Sciences sticky millet testing plot, Beijing suburbs; **(c)** Corn noodle *hele* machine, Suizhong County, Liaoning Province (supplied by Jin Guiyun)

Fig.S4 *Hele* noodle manufacturing process,Xun County, Henan Province (source: Hebi City Television Station, 2012). **(a)** Wooden *hele* rack; **(b)** *hele* millet noodle manufacturing process; **(c)** *hele* millet noodles