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ORIGINAL ARTICLE

Nitrogen isotope values of *Pennisetum glaucum* (pearl millet) grains: towards a reconstruction of past cultivation conditions in the Sahel, West Africa

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Abstract
The nitrogen isotope compositions of charred wheat and barley grains reflect manuring intensity and have been used to reconstruct past manuring practices at archaeological sites across Europe and western Asia. To assess whether this analytical method can be applied to a staple crop in the West African Sahel, the nitrogen isotope values of *Pennisetum glaucum* grains in this region were determined and the effect of charring ascertained. *Pennisetum glaucum* ears were collected from fields in northeast Senegal, where the fertilisation histories of the plots (manure and/or household waste) were known. The nitrogen isotope values of these millet grains provide an insight into the values to expect for *P. glaucum* grains grown with low to moderate addition of manure/household waste in a semi-arid climate. Charring of *P. glaucum* grains by heating at 215–260 °C for 4–24 h increases their nitrogen isotope values by a maximum of 0.34‰. In light of these modern data, the nitrogen isotope values of millet grains recovered from the archaeological settlement mound of Tongo Maaré Diabal, Mali, can be interpreted as evidence for modest levels of manure/household waste input throughout the occupation of the site from cal AD 500–1150. This study demonstrates the potential for nitrogen isotope values of *P. glaucum* grains to shed light on past farming practices in West Africa.

Keywords West Africa · Charring experiment · Intensification · Manure · Palaeoethnobotany

Introduction

The notion that cultivators had to intensify agricultural production to support growing populations has held sway for many decades (e.g. Boserup 1965; Ellis et al. 2013), influenced not least by research focused on highly labour-intensive and productive irrigated farming plots in the early urban heartlands of southern Mesopotamia (Adams 1981; Algaze 2001). Increasingly, however, research is demonstrating that ancient farming comprised a whole spectrum of practices that are too heterogeneous to consider in terms of exerting simple direct effects on social organisation and stratification (e.g. Halstead 1989; Gurven et al. 2010; Bogaard et al. 2018a). Rather, food production strategies simultaneously shape and are shaped by the mechanisms that are in place to ensure sufficient nutritional resources. Reconstructing ancient farming practices is therefore fundamental if we are to examine and understand the relationship between agricultural and social trajectories in the past.

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The everyday labour intensive practices of growing crops, such as manuring, weeding and watering, have until recently proven difficult to detect archaeologically. In the last 10 years, however, novel isotopic and weed ecological methods have been developed and applied to ancient plant remains, allowing such practices to be identified in the archaeological record. This provides new possibilities for exploring how the labour intensity of agriculture changed through time. In this paper we focus on identifying the intensity of manuring by using the nitrogen isotope (δ^{15}N) values of charred archaeobotanical remains. Studies on the effect of manuring on cereal grain δ^{15}N values have thus far been restricted to Europe, southwest Asia and Morocco, and to wheat and barley. We focus our study on the semi-arid Sahel region of West Africa, where the climate and pathways to food production are very different from those of Europe and Asia. In Africa, herding tended to precede crop cultivation (Marshall and Hildebrand 2002) and ploughs were unknown in sub-Saharan Africa before the twentieth century (Blench 2014). Moreover, it is not wheat and barley, but *Pennisetum glaucum* (pearl millet) that is generally the staple crop in West Africa (Champion and Fuller 2018). West Africa therefore provides an exciting context for future crop isotope studies, allowing comparison with models of agricultural change otherwise heavily influenced by Eurasian narratives.

In this paper we determine the range in δ^{15}N values of modern *P. glaucum* grains to be expected when grown with modest manure/household waste inputs, building on and broadening a model used to identify manuring of wheat and barley in Europe and southwestern Asia (Styring et al. 2017a). Since the majority of archaeobotanical remains are recovered from archaeological sites in a charred (carbonised) state, we also determined the effect of heating on the δ^{15}N values of *P. glaucum* grains. We then discuss what the δ^{15}N values of charred *P. glaucum* grains from the archaeological site of Tongo Maaré Diabal (cal AD 500–1150), Mali, reveal about agricultural practice, demonstrating the potential for nitrogen isotope analysis of crop remains to enrich our knowledge of food production strategies in West Africa.

Reconstructing manuring practice from crop nitrogen isotope values

Addition of manure to soil has been found to increase the nitrogen isotope (δ^{15}N) values of cereal grains by up to 10‰, according to the amount and frequency of its application (Fraser et al. 2011). Moreover, it has been shown that the nitrogen isotopic composition of charred cereal grains is likely to remain unchanged for thousands of years (DeNiro and Hastorf 1985; Fraser et al. 2013; Styring et al. 2013). As a result of these findings, nitrogen isotope analysis of archaeobotanical remains has been used to reconstruct the intensity of manuring and determine how it varied spatially and temporally at a number of archaeological sites in Europe and southwest Asia. These studies have revealed that: (1) manuring was practised from as early as the 6th millennium cal BC in Europe (Bogaard et al. 2013; Vaiglova et al. 2014a); (2) there was differential manuring of wheats and barley, likely relating to their economic importance and culinary uses (Gron et al. 2017; Nitsch et al. 2017; Styring et al. 2017b); and (3) manuring intensity changed with the need to support urban centres and craft specialists, but the direction of this change was not universal (Styring et al. 2017a, b; Vignola et al. 2017).

Modern studies on the effect of manuring intensity on cereal grain δ^{15}N values have thus far been carried out in Europe, southwest Asia and Morocco on wheat and barley (Fraser et al. 2011; Kanstrup et al. 2011; Styring et al. 2016). It has been found that with decreasing annual rainfall, plant (and cereal grain) δ^{15}N values increase, independently from the effect of manuring (e.g. Craine et al. 2009). This means that it is not possible to equate a cereal grain δ^{15}N value with a particular manuring level without taking into account regional annual rainfall. A linear model was therefore developed to allow the manuring level of an archaeological cereal grain sample to be predicted, based on its δ^{15}N value and the estimated annual rainfall of its growing location (Styring et al. 2017a, Fig. 2). This permitted reconstruction of changing manuring intensity in the semi-arid climate of northern Syria, with an annual rainfall between 180 and 450 mm, between the 7th and 3rd millennia cal BC (Styring et al. 2017a). Nevertheless, it is desirable to independently verify the cereal grain values to be expected in the Sahel region of West Africa, given its distinct climate, and to determine the expected δ^{15}N values of *P. glaucum*, given its distinct physiology.

The effect of charring on crop nitrogen isotope values

The majority of archaeobotanical remains are recovered from archaeological sites in a charred state and therefore the effect of charring on the δ^{15}N values of plant remains also needs to be determined if we are to use the values of charred crop remains to reconstruct past manuring practices. There have been a number of studies on the effect of charring on the δ^{15}N values of wheat and barley grains and pulse seeds, but none have considered *P. glaucum* grains. A study by Charles et al. (2015) found that heating glume wheat (*Triticum monococcum*, einkorn, and *T. dicoccum*, emmer) grains in low oxygen conditions (achieved by wrapping in aluminium foil and burying in sand) at 220–240 °C for 2–24 h produced undistorted charred grains that closely resemble ‘well-preserved’ charred grains from archaeological sites. Well-preserved charred *Panicum miliaceum* (broomcorn millet) grains were discovered alongside einkorn and emmer wheat grains in storerooms at the Bronze Age Aegean site of Assiros, Greece (Jones et al. 1986). We therefore assume that they were heated at similar temperatures to the glume wheat grains and thus that 'well-preserved' charred *P. glaucum* grains preserved on archaeological sites were heated in similar conditions to the einkorn and emmer grains studied by Charles et al. (2015). We therefore determined what effect heating *P. glaucum* grains in a low oxygen environment at 215–260 °C for 2–24 h had on their δ^{15}N values. We followed the method used by Nitsch et al. (2015), who determined the effect of charring on wheat and barley grain and pulse seed δ^{15}N values within the same temperature and duration 'charring window'.

The modern study region in the West African Sahel

In Köppen-Geiger climate classification terms, the modern study region of northeast Senegal is semi-arid (BSh) (Fig. 1). Based on rainfall data derived from interpolation of average monthly climate data for 1970–2000, available from the WorldClim version 2 database (Fick and Hijmans 2017), annual rainfall ranges from 490 to 570 mm and generally increases from the north to the south of the study region (Fig. 1). It rains almost exclusively between the months of July and October. Daily rainfall records for 2016 and 2017 from weather stations located in Diourbel (14.650°, − 16.233°) and Kaolack (14.147°, − 16.051°; Fig. 1) were compared to the interpolated WorldClim annual rainfall values for these locations. The recorded 2016 annual rainfall totals of 523 mm and 586 mm for Diourbel and Kaolack respectively were very similar to the WorldClim estimates of 490 mm and 586 mm. In 2017, the reported annual rainfall totals of 605 mm and 614 mm at Diourbel and Kaolack respectively were higher than these estimates.

According to the Harmonized World Soil Database (http://www.fao.org/soils-portal/soil-survey/soil-maps-and-databases/harmonized-world-soil-database-v12/en/. Accessed 31 Aug 2018), the *P. glaucum* plots sampled in Thiès were located on sandy clay loam leptosols (very shallow soils over hard rock or in unconsolidated gravelly material) and regosols (soils with very limited soil development; Nachtergaele et al. 2009). In Fatick, the plots were located on arenosols (sandy soils featuring very weak or no soil development; Nachtergaele et al. 2009). In this area, however, farmers note two different soils, known as *Jóór* (*Dior* in the French spelling) and *Deg* (*Deck*), to which are ascribed different properties. *Jóór* soils are sandy with very low soil organic matter and little ability to retain nutrients, whereas *Deg* soils are more fertile (properties described in McClintock and Diop 2005). The type of soil for each plot in Fatick is noted in ESM 1. In Kaffrine, the plots were on lixisols (soils with sub-surface accumulation of low activity clays and high base saturation) and regosols (Nachtergaele et al. 2009). In general, all of these soils are characterised by low organic carbon content and a poor ability to retain soil nutrients due to their low cation exchange capacity and acidic pH (Table 1; Nachtergaele et al. 2009).

A total of 38 *P. glaucum* plots were studied, ranging from 0.08 to 5 ha in size (Fig. 2). The farmers practise crop rotation, usually featuring annual rotation of *P. glaucum* and

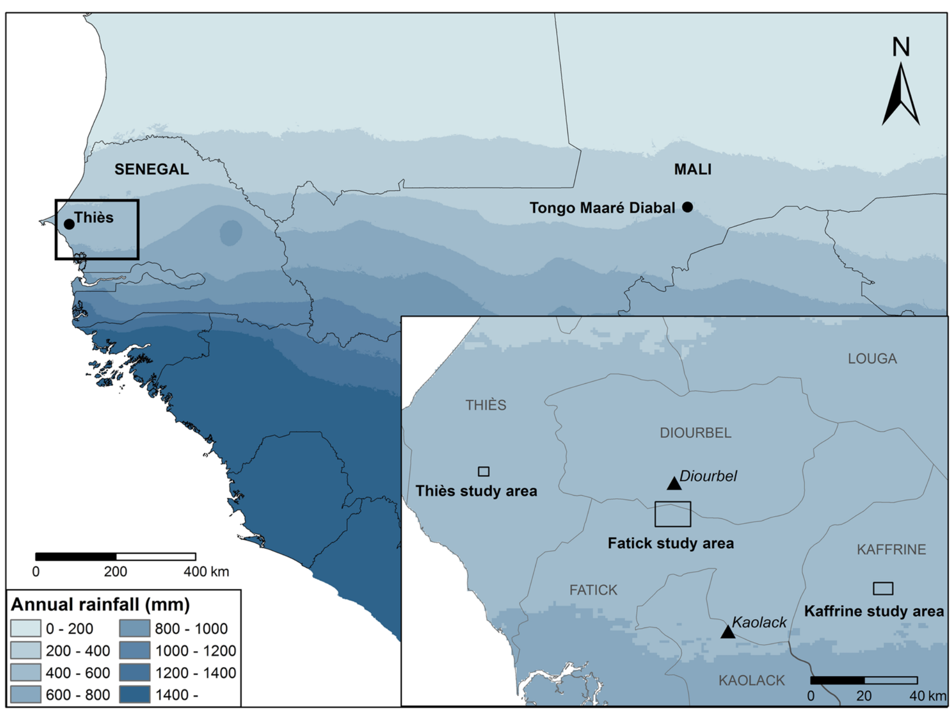

Fig. 1 Map showing location of sampling locations in Senegal and Tongo Maaré Diabal, Mali. Weather stations with rainfall data for 2016 and 2017 are marked as triangles. Annual rainfall data are derived from interpolation of average monthly climate data for 1970–2000, available from the WorldClim database, version 2 (Fick and Hijmans 2017)

Table 1 Summary of the study regions in northeast Senegal

Département	Thiès	Fatick	Kaffrine
Abbreviation	NOT	NJI	KAF
Approximate location	14.7° N, − 16.9° E	14.6° N, − 16.2° E	14.3° N, − 15.5° E
Interpolated annual rainfall (mm)	501	494	565
No. plots	11	18	9
Average plot area (ha)	1.2	0.6	2.2
Average crop height (cm)	236	263	233
Soils	Leptosols and regosols	Arenosols	Lixisols
Average soil organic matter content (%)	0.5	0.5	0.5
Average potential cation exchange capacity (mmol/100 g at pH 8.2)	33	31	22
Average soil pH (KCl)	5.6	6.7	5.9
Rotation regime	Mostly peanut-millet-peanut	Mostly peanut-millet-peanut; some millet only; one plot cowpea-millet	Mostly peanut-millet-peanut; two fallow before millet
Sowing time	July 2017	July 2017	June/July 2017
Tillage and harrowing	Horse-drawn plough in March/April 2017	Horse-drawn plough in July 2017	Horse-drawn plough in June/July 2017
Weeding	With a hoe		
Manuring	7 with manure/compost; 4 without	7 with manure/compost; 11 without	4 with manure/compost; 5 without
Harvest time	October 2017		

Fig. 2 *Pennisetum glaucum* plot NJI01 in the Fatick study area in northeast Senegal. The *P. glaucum* plants are c. 2 m high. Photograph by A. Styring

Arachis hypogaea (peanut) and sometimes annual rotation of *P. glaucum* and *Vigna unguiculata* (cowpea). *Vigna unguiculata* was sometimes intercropped with *P. glaucum* or *A. hypogaea* (Table 1). The *P. glaucum* seeds were commercially available varieties. Most farmers cultivated Souna 3 whereas half of the farmers in Kaffrine cultivated Thialack 2 (ESM 1). *Pennisetum glaucum* was sown using a seed drill pulled by a horse, between the end of May and the beginning of July 2017. After emergence of the millet seedlings, they were thinned to a density of about 4 plants/m^2, with row and plant-to-plant spacing of around 50 cm. Plots were weeded with a hoe up to five times (on average three times) during the growing season (Table 1).

All of the plots were organic, being managed without chemical fertilisers, and were not irrigated. Around half of the *P. glaucum* plots received additional organic matter in the form of composted kitchen waste, ashes, crop residues and animal manure. This material was raked into a pile to which additional material was added throughout the year. It takes about 6–10 months before the material is fully composted and resembles soil. This compost was then applied to the soil during preparation of the plots for sowing of *P. glaucum*, in May/June 2017. The amount of compost that each household produced was very variable and was most dependent on the quantity of manure that was available, which was largely a function of the number and type of animals the household owned. Manure generally comes from sheep, goats and horses stabled within the compound. Cattle are generally grazed outside the villages by contracted herders. Where farmers were able to give an estimate of the quantity of compost applied to their fields, it was generally in the range of 7–12 cartloads of around 1.5 m^3 compost each, which are estimated to weigh in total approximately 1,800 kg. This equates very approximately to between 1,000 and 3,700 kg/ha. These quantities are broadly similar to those quoted by a project carried out by the Rodale Institute promoting compost production in this region of Senegal. During this project, manure application was estimated to be between 1,100 and 1,500 kg/ha (McClintock and Diop 2005). In all cases, however, there was not enough compost to fertilise the entire area of land owned by the farmer. The application of compost was therefore done strategically, targeting areas that were noticed to have been unproductive in the previous year. A few plots received animal manure directly, either through penning of cattle, sheep and goats on the field for one or more months during the dry season after the harvest, or when pastoralists were permitted to drive their herds through the harvested fields. Plots that had received no additional organic matter for the last 3 years were classified as having received a low level of manure, after the classification in Styring et al. (2017a). There were three plots that had received compost 2 years before sampling took place and these were classified in the low manuring level, since the amount of compost added to fields was generally low. Plots that had received additional organic matter in 2017, either in the form of compost or manure from livestock, were classified in the medium manuring level. Full details of the cultivation histories of the plots are in ESM 1. Although the household waste probably differs in nature from that produced by households in the past, this subsistence agriculture without artificial fertilisers is as close as possible to the type of farming thought to have been practised in the Iron Age. Moreover, the type and number of animals kept within households, namely a few sheep and goats and perhaps a donkey, are likely to have been similar.

Fig. 3 Excavation Unit G at Tongo Maaré Diabal in February 2010, with Gandamia escarpment in the background. Photograph by N. Gestrich

Table 2 Summary of the chronology of the archaeological horizons excavated at Tongo Maaré Diabal (after Gestrich and MacDonald 2018)

Horizon	Date (cal AD)
5	1000–1150
4	900–1000
3, upper and lower	750–900
2	650–750
1	500–650

The archaeological site of Tongo Maaré Diabal, Mali

Tongo Maaré Diabal (TMD) is a 9 ha settlement mound situated on a narrow strip of flat land between two escarpments, 1 km north of the modern town of Douentza, Mali (Figs. 1, 3). Excavations carried out in the 1990s revealed archaeological deposits up to 4 m deep that were grouped into five horizons of superimposed earthen buildings. Continuities in architectural layout and radiocarbon dates on charcoal suggest continuous occupation of the site for 650 years, from around cal AD 500–1150 (Gestrich and MacDonald 2018). The site was abandoned in the 12th century AD, during a period of regional economic and social instability following the decline of the Empire of Ghana. The dates of the horizons, based on radiocarbon dates and thickness of deposits, are given in Table 2, following Gestrich and MacDonald (2018). More extensive excavations of the uppermost occupation horizon (c. cal AD 1000–1150) in 2010 uncovered household structures and buildings believed to have been used as working areas for blacksmiths (Gestrich 2013). The architectural remains have been interpreted as belonging to separate domestic compounds, inhabited by different kinship groups. The continuity in the location of these domestic compounds throughout the stratigraphy of the site suggests that the social boundaries between the domestic compounds persisted for centuries (Walicka Zeh and MacDonald 2004).

TMD was established on a low rise at the margins of an ancient floodplain. The plain contains clayey soils of tolerable agricultural quality, irrigated today by streams running from the escarpments, which collect in ponds during the rainy season. Palaeochannels in the western part of the plain indicate that floodwaters of the river Niger to the west reached the area during wetter periods both in pre- and recent history (MacDonald et al. 2017), but there is currently no consensus for the likely degree of flooding in the area during the occupation of the site. The closest proxy evidence for past rainfall at TMD is the analysis of sediments from the river Yamé, a tributary of the Niger 120 km south of TMD. Micromorphology and grain size analysis of the sediments provide evidence for variable hydrological conditions, both slightly wetter than (1630–1410 cal BP) and similar (1320–910 cal BP) to those of the present day

(Lespez et al. 2011). Given the high spatial variability of rainfall in the Sahel, however, it is difficult to assess how useful this proxy is for reconstructing past rainfall at TMD. Fish remains recovered from TMD include taxa that indicate seasonal proximity to floodwaters of the river Niger, particularly in Horizon 5 (cal AD 1000–1150), but it is also possible that fish which could not have survived in local ponding areas were brought to the site through trade (Gestrich and MacDonald 2018).

Bones of domesticated and wild mammals, and bird and fish remains, have been found at TMD. Domestic bovid bones (sheep and goat in particular) were the most numerous, followed by fish, dogs and domestic fowl. Cattle seem to have been rare at the site until Horizon 3 (post cal AD 750), suggesting that cattle-keeping or the presence of pastoral groups in the area may have become more prevalent at this time. The wild bovids identified in the assemblage, notably red-fronted gazelle and duikers, are adapted to savanna habitats, particularly to wooded grasslands (Gestrich and MacDonald 2018).

The archaeobotanical assemblage from TMD is currently being re-analysed by L. Champion as part of his doctoral dissertation. Preliminary results indicate that domesticated *P. glaucum* was the principal species recovered, accounting for around 85% of the assemblage (15,400 grains and involucres). Plant consumption at TMD thus seems to have comprised *P. glaucum* and *Digitaria* sp. (wild fonio), with sporadic consumption of fruits of *Sclerocarya birrea* and *Vitex* sp., local trees (3% of the assemblage). The plant remains from TMD do not appear to change throughout the deposit—only the introduction of *Sorghum bicolor* (sorghum) in very small quantities; 33 grains of the total 18,000 plant remains are noted in Horizon 3 (cal AD 750–900) and onwards. Results of preliminary studies on wood charcoal by Dirk Uebel are summarised in Gestrich and MacDonald (2018). They indicate that TMD was situated in a mosaic of fields and fallow land. Tree taxa such as *Faidherbia albida, Balanites aegyptiaca* and *Sclerocarya birrea* would have provided useful products (fruits/seeds/leaves) for human and animal consumption and thus tended to be protected within cultivated fields. *Guiera senegalensis* and *Combretum glutinosum* are also present in the charcoal assemblage. They recover well after cutting and are therefore indicative of fallow fields (Höhn and Neumann 2012). Tree taxa such as *Balanites aegyptiaca, Celtis integrifolia, Prosopis africana* and *Khaya senegalensis* also point to the presence of open woodland along watercourses, presumably beyond the cultivated zone. The lack of changes with time in the charcoal record suggests that TMD was founded within an already established mosaic of fields and fallow land and that metal working activity evidenced at TMD did not completely eliminate the woods in the vicinity of the site.

Surveys within a 10 km radius of TMD have documented 15 substantial sites that are assumed on the basis of pottery to be contemporary with TMD. Of these, ten are medium-sized settlement mounds (5–15 ha) and five are iron smelting sites. There are also indications from temporary campsite remains and the occasional presence of cattle remains in the TMD faunal assemblages that mobile pastoral groups inhabited the area at certain times of the year. The settlement mounds are evenly spaced across the landscape without any clustering, giving the impression of having been autonomous units rather than focused around an urban centre (Gestrich and MacDonald 2018). A conspicuously large amount of iron slag has been recovered at TMD and the surrounding settlements, suggesting that the settlements in this area were part of a proto-industrial iron production system supplying a wider regional trade network. Gestrich and MacDonald (2018) speculate that TMD was thus part of a wider state-organised economic landscape, the settlement perhaps having been established for iron-working specialists.

Materials and methods

Field sampling methods

Modern *P. glaucum* grains were collected in the first 2 weeks of October 2017, shortly before or after harvest, in three villages located in the départements of Thiès, Fatick and Kaffrine in northeast Senegal (Fig. 1). The village in Thiès was chosen because of its previous involvement in a programme promoting compost production conducted by the Rodale Institute. The villages in Fatick and Kaffrine were chosen due to their current involvement in a United States Department of Agriculture (USDA) funded programme entitled 'Projet Services Entreprises Mil Sénégal', the aim of which is to increase millet production and thus increase agricultural revenue. One element of the USDA funded project involves training farmers in composting kitchen waste and animal manure for use on their fields.

Ten panicles were collected randomly from each plot; in the cases where the crop had already been harvested, they were collected from the piles of panicles drying in the fields. Soil from 0 to 15 cm depth was also collected from each plot and air dried prior to storage in self sealing plastic bags at ambient temperature. The location of the boundary of every plot was recorded using a handheld GPS unit. The density of *P. glaucum* plants was estimated and the average crop height measured. Information about the sowing, preparation and weeding of each plot, including application of manure/composted household waste, was ascertained through interviews with farmers in Wolof (summary in Table 1, details in ESM 1).

Pennisetum glaucum grains were removed along the length of each panicle and 100 grains were sampled from each, giving a mixed sample of 1,000 grains per plot. For six plots (one cultivated with manure/household waste and one without from each village), 100 grains from each panicle were kept separate to determine the variability in *P. glaucum* δ^{15}N values within a single plot. For six panicles (one cultivated with manure/household waste and one without from each village), 50 grains were removed from five points along the length of the panicle to determine the variability in values within a single panicle.

For each plot, 30 grains from the 1,000 sampled were randomly selected and ground to a powder with a mortar and pestle. This number of grains was chosen as a compromise between the ten grains usually sampled (and available) in archaeological contexts and the need to homogenise a large enough number of grains to account for isotopic variability and obtain a representative δ^{15}N value for the plot. For each panicle, ten grains from the 100 sampled were randomly selected. The panicle sample identification codes (IDs) comprise the field ID followed by the randomised panicle number (1–10). For each position on a panicle, ten grains from the 50 sampled were randomly selected. The panicle position sample IDs comprise the field ID followed by the panicle number (1–10) and the position on the panicle (1–5). These grains were ground to a powder with a ceramic mortar and pestle.

Soil analysis

All soil analyses were carried out at the Institut für Physische Geographie, Goethe Universität, Frankfurt am Main, Germany. Prior to analysis, air dried soils were passed through a 2 mm sieve. Soil organic carbon content was determined using a Leco EC-12 carbon analyser, potential cation exchange capacity in mmol/100 g was determined following the procedure by Mehlich, described in DIN 19684 (1977) and soil pH was determined in 0.1 M potassium chloride using an electrode.

Charring experiment

Pennisetum glaucum grains were removed from a single panicle collected from plot NOT09 in Thiès, Senegal. Fifty randomly selected grains were charred for each temperature and duration combination, at 215, 230, 245 or 260 °C for 4, 8 or 24 h. Each of these batches of grains was weighed before and after charring. The grains were loosely wrapped in aluminium foil packets and buried in individual beakers of sand, following the method described by Fraser et al. (2013). Samples were heated in a Gallenkamp Plus II electric oven at the University of Oxford, UK. The oven was preheated to the desired temperature and then the entire batch of samples was placed randomly inside it, with batches being removed after 4, 8 and 24 h. The grains were allowed to cool to room temperature in their beakers. From each batch, three subsamples of ten grains (labelled A, B and C) were homogenised separately for isotopic analysis using an agate mortar and pestle. The sample IDs comprise the temperature followed by the duration of heating and the subsample A, B or C.

Archaeobotanical sampling

Pennisetum glaucum grains recovered by flotation from excavated units A and B at Tongo Maaré Diabal, Mali, were identified to species by comparison with reference collections, by L. Champion. Between seven and 15 grains (weighing 4–10 mg) were selected from 15 contexts, spanning Horizons 1–5 (cal AD 500–1150). Two of the samples were scraped clean with a scalpel, crushed and analysed using Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR) to look for the presence of carbonate, nitrate and/or humic contamination (Vaiglova et al. 2014b). No evidence for contamination was observed (ESM 2) and so the remaining samples were scraped clean with a scalpel before being crushed using an agate mortar and pestle.

Laboratory analysis

The homogenised powders of the fresh and charred *P. glaucum* grains were weighed into tin capsules for isotopic analysis on a Thermo MAT 253 continuous flow isotope ratio mass spectrometer coupled to a Thermo Flash 1,112 Series elemental analyser in the Institut für Geowissenschaften, Goethe-Universität, Frankfurt am Main, Germany. Isotopic data are provided in ESM 3. The nitrogen contents of the samples were calculated based on the area under the N_2 peak relative to the weight of the sample, calibrated using IAEA-N2. Following the method presented by Szpak et al. (2017), measurement uncertainty in %N was monitored using two in-house standards (DL leucine, %N 10.7%; DL glutamic acid monohydrate, %N 8.5%). Precision ($u(R_w)$) was determined to be ±0.4% on the basis of repeated measurements of calibration standards, check standards and sample replicates. Accuracy or systematic error ($u(bias)$) was determined to be ±0.5% on the basis of the difference between the observed and known %N contents of the check standards. Using the equation presented in ESM 4, the total analytical uncertainty was estimated to be ±0.7%. Stable nitrogen isotope values were calibrated to the AIR scale using IAEA-N-1 (δ^{15}N 0.4 ± 0.2‰) and IAEA-N-2 (δ^{15}N 20.3 ± 0.2‰). Measurement uncertainty in δ^{15}N was monitored using three in-house standards: LEU (DL leucine, δ^{15}N 6.46 ± 0.40‰), GLU (DL glutamic acid monohydrate, δ^{15}N − 1.87 ± 0.07‰) and MIL (millet flour from a single panicle from plot NJI11, δ^{15}N 3.14 ± 0.63‰). Precision ($u(R_w)$) was determined to be ± 0.27‰, accuracy or systematic error ($u(bias)$) was ± 0.55‰ and the total analytical uncertainty was estimated to be ± 0.61‰ using the equation presented in ESM 4.

The homogenised powders of the archaeological *P. glaucum* grains were weighed into tin capsules for isotopic analysis on a Sercon 20–22 isotope ratio mass spectrometer coupled to a Sercon GSL elemental analyser at the Research Laboratory for Archaeology and the History of Art, University of Oxford, UK. Stable nitrogen isotope values were calibrated to the AIR scale using Caffeine-2* (δ^{15}N − 2.90 ± 0.03‰; University of Indiana) and IAEA-N-2 (δ^{15}N 20.3 ± 0.2‰). Measurement uncertainty was monitored using one in-house standard: Alanine (DL alanine, δ^{15}N − 1.56 ± 0.03‰). Precision ($u(R_w)$) was determined to be ± 0.20‰, accuracy or systematic error ($u(bias)$) was ± 0.08‰ and the total analytical uncertainty was estimated to be ± 0.21‰ using the equation presented in ESM 4. Statistical analyses were performed in R v.3.4.3.

Results

Variability in *P. glaucum* δ15N within single panicles and plots

Figure 4 shows variation in *P. glaucum* δ^{15}N values within single panicles. The values vary by up to 0.7‰ within panicles (mean = 0.4‰, n = 6). There is no consistent directional change in values from the base to tip of the panicles, or vice versa. Figure 5 shows boxplots of the variation in δ^{15}N values of panicles sampled within single plots. The values differ up to 4.8‰ within plots (mean = 3.2‰, n = 6). Calculated from the standard error, the 95% confidence interval of *P. glaucum* grain δ^{15}N values within plots varies between ± 0.6 and 1.7‰ (mean = 1.1‰, n = 6). Levene's test for

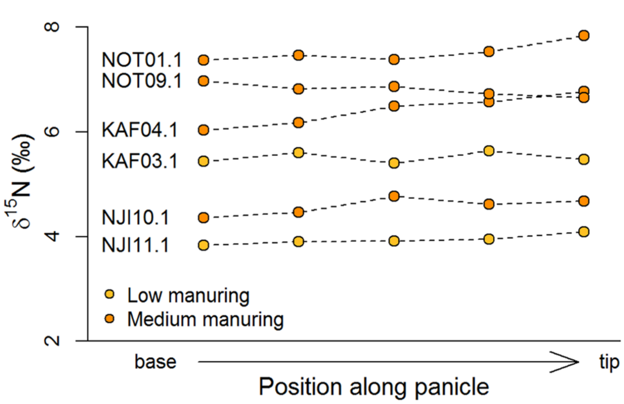

Fig. 4 Comparison of δ^{15}N values of *P. glaucum* grains sampled from different points along single panicles

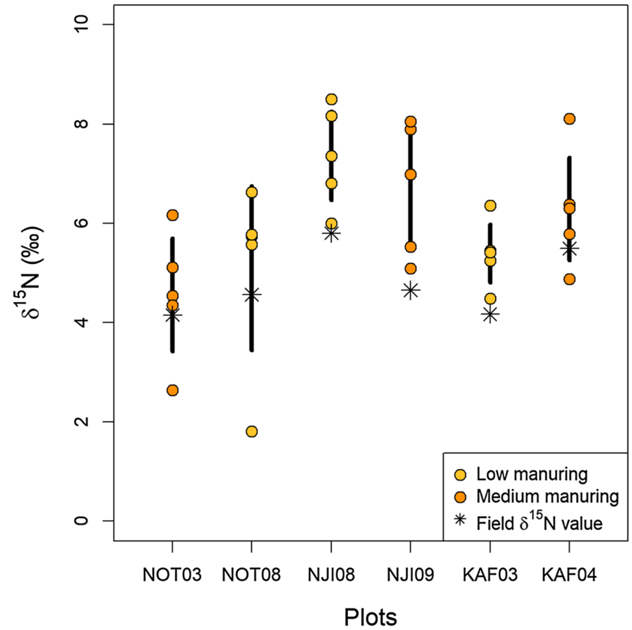

Fig. 5 Comparison of δ^{15}N values of *P. glaucum* grains sampled from different panicles from single plots. Vertical lines represent the mean ± 95% confidence interval for within plot δ^{15}N values. Stars represent the values of 30 grains homogenised from ten panicles from each plot

equality of variance found that the variances in panicle values were not statistically different between plots with low and medium manuring levels ($F(1, 4) = 0.01$, $p = 0.92$). The δ^{15}N values of the 30 grains homogenised from each plot are also shown for comparison (Fig. 5). This value overlaps with the mean ± 95% confidence interval for panicle values in only three out of six cases. It is likely that the five panicles from each plot whose grains were sampled and homogenised for the whole plot δ^{15}N value, but were not included in the within-plot comparison, have values outside the range of those that were included.

The effect of manuring on *P. glaucum* δ15N values

The *P. glaucum* δ^{15}N values from individual plots range from − 0.6 to 6.6‰. Figure 6 compares the values of grains grown on plots classified as receiving either low or medium levels of organic matter/manure. The data are not normally distributed and a Mann–Whitney U test was used to test whether grains grown in plots classified as receiving medium levels of manure had higher δ^{15}N values than those receiving low levels. This test was found to be statistically significant ($W = 129.5$, $p = 0.0459$). When the very low value from plot NJI02 (− 0.6‰) is excluded from the analysis, the data are normally distributed and thus an unpaired two-sample one-sided t test can be used to compare values from plots receiving low and medium levels of manure. In this case, the test

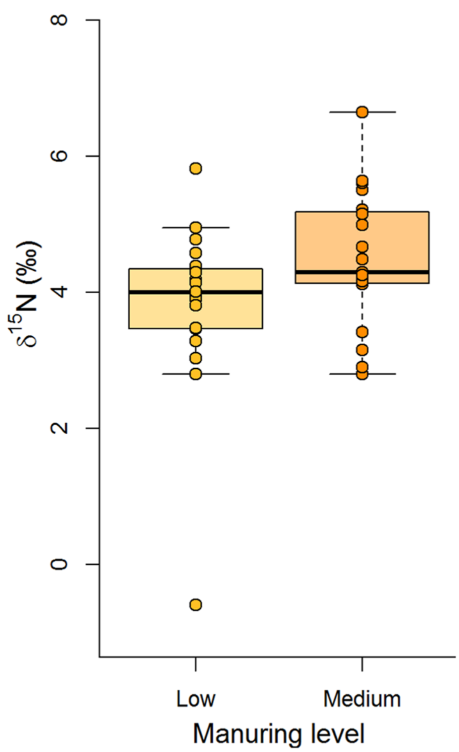

Fig. 6 Comparison of $\delta^{15}N$ values of *P. glaucum* grains from plots classified as receiving low and medium levels of manure/organic matter. Low level manuring means plots have received no manure/household waste for >2 years. Medium level manuring means plots received manure/household waste in the year prior to harvest. Boxes represent the quartiles, the bold line represents the median and whiskers represent 1.5 × the interquartile range

was found to be statistically non-significant at a significance level of 0.05 ($t(32.47) = -1.61$, $p = 0.059$, mean $\delta^{15}N$ value of low manure input = 4.0‰, mean $\delta^{15}N$ value of medium manure input = 4.5‰).

Fitting *P. glaucum* $\delta^{15}N$ values into the linear regression model

Figure 7 shows the *P. glaucum* grain $\delta^{15}N$ values plotted against the interpolated annual rainfall value for their location from WorldClim version 2 (Table 1). Figure 7a colour codes the samples by their observed manuring level and Fig. 7b by the manuring level that is imputed (value for missing data substituted) using a fitted linear model. This linear model regresses modern cereal grain $\delta^{15}N$ values sampled from Europe, western Asia and Morocco on mean annual rainfall, with a variance offset for crop type (wheat, barley or *P. glaucum*), and for each manuring level. The solid lines in Fig. 7 represent the fitted $\delta^{15}N$ values at each annual rainfall value and for each manuring level, evaluated using the restricted maximum likelihood estimate. The dotted lines in Fig. 7 represent the thresholds between manuring levels. In Fig. 7b each *P. glaucum* sample from Senegal is assigned to the manuring level whose fitted $\delta^{15}N$ value at the annual rainfall for its location is most similar to the measured value. For more details on the fitted linear model, see Styring et al. (2017a, supplementary information—statistical supplement). Overall the samples were correctly assigned in 50% of the cases (Table 3), comparing favourably with 33%, which is the correct assignment rate if manuring levels were randomly assigned. The majority of the low manured samples (74%) were assigned incorrectly as medium manured, whereas the majority of the medium manured samples (74%) were correctly assigned. Only one medium manured sample was incorrectly assigned to the high manure level (Table 3).

The effect of charring on *P. glaucum*

The majority of the millet which had been charred in the laboratory resembled optimally charred material that is identifiable to species from archaeological sites. However, heating at 260 °C for 8 h produced *P. glaucum* grains that were highly distorted, making identification to species difficult. The grains were therefore not heated for longer at 260 °C. Colour photographs of the different charred batches are given in ESM 5. Loss of mass increased with charring temperature and duration, resulting in up to 49% mass loss after heating at 245 °C for 24 h (Fig. 8, ESM 6). The %C and %N content of *P. glaucum* grains increased with charring up to 67% and 4%, respectively, and the molar ratio of C/N increased (Fig. 9, ESM 6). There is a general trend of increasing $\delta^{15}N$ values with increased charring temperature and duration of heating (Fig. 10, ESM 6). A multiple linear regression model was calculated, comparing the effect of charring (charred vs. uncharred samples) with different coefficients for temperature and time. The model produced a reasonable fit, with an adjusted R^2 of 0.56. The effect of charring is significant (Beta = 2.84‰, SE = 0.49‰, $t = 5.82$, $p < 0.001$) and the model predicts a 0.12‰ increase in $\delta^{15}N$ for every 15 °C increment of heating (Beta = 0.012‰, SE = 0.0020, $t = 6.07$, $p < 0.001$) and a 0.011‰ increase for every hour of heating (Beta = 0.011‰, SE = 0.0039, $t = 2.86$, $p = 0.007$). Since it is not feasible to identify the temperature and duration at which ancient grains were charred from visible inspection of archaeobotanical remains, it seems more sensible to calculate a 'worst-case' $\delta^{15}N$ value offset based on a predicted increase in $\delta^{15}N$ value with charring at 245 °C for 24 h—the greatest offset where *P. glaucum* grains are still identifiable to species. This calculation gives an offset of 0.34‰ between the values of charred and uncharred *P. glaucum* grains.

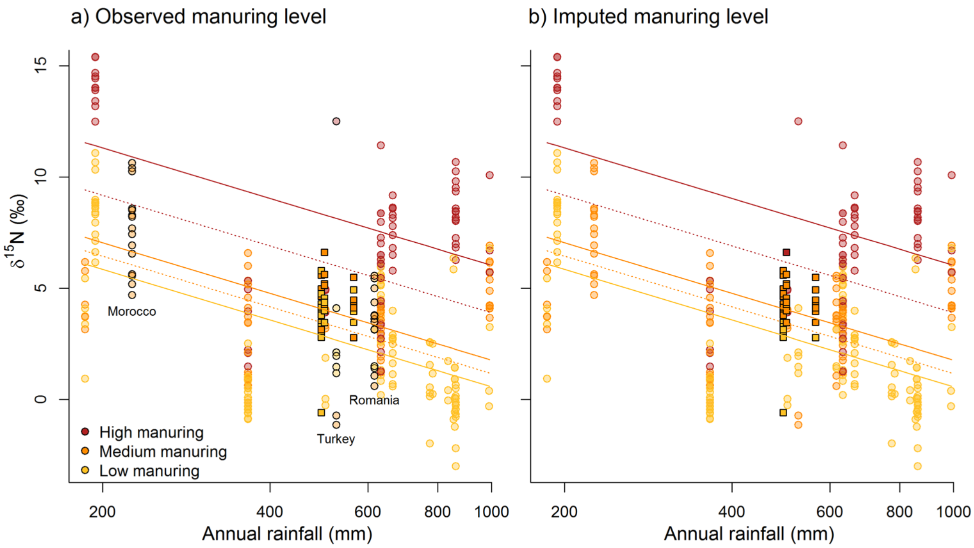

Fig. 7 **a** Modern cereal grain $\delta^{15}N$ values plotted against the natural log of mean annual rainfall, colour coded by manuring level. The solid lines represent a fitted linear model relating cereal grain $\delta^{15}N$ values to mean annual rainfall for each manuring level. The dotted lines represent the thresholds between manuring levels. Squares outlined in black represent *P. glaucum* grains sampled in northeast Senegal. Circles outlined in black represent cereal grains sampled from small-scale traditional farms (locations are given adjacent to these data points); **b** as for Fig. 5a, but *P. glaucum* grains sampled from northeast Senegal (squares outlined in black) are colour coded by the manuring level that is *imputed* using the fitted linear model. All crop $\delta^{15}N$ values, apart from those of *P. glaucum* from Senegal, have been previously published in Styring et al. (2017a)

Table 3 A confusion matrix for prediction of manuring level of pearl millet grains sampled in Senegal

	Predicted		
	Low	Medium	High
True			
Low	**26**	74	0
Medium	21	**74**	5
High	NA	NA	NA

Numbers are percentages

Figures in bold designate correct predictions. Rows sum to 100

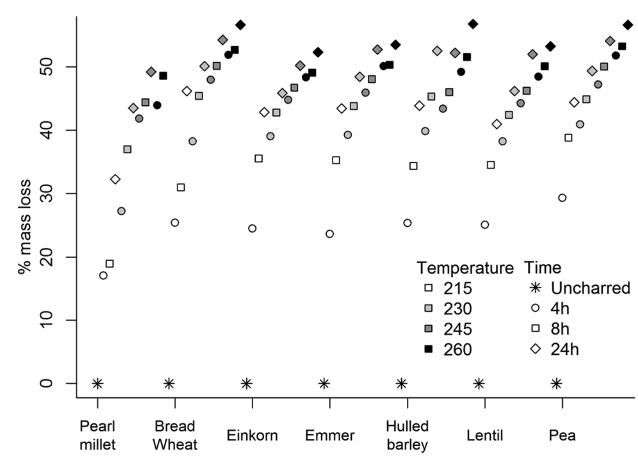

Fig. 8 Summary of % mass loss for seven different crop taxa charred at 215–260 °C for 4–24 h. The data for all taxa apart from *P. glaucum* have been previously published in Nitsch et al. (2015)

Pennisetum glaucum $\delta^{15}N$ values from Tongo Maaré Diabal, Mali

The archaeological *P. glaucum* $\delta^{15}N$ values from Tongo Maaré Diabal range from 4.0 to 7.1‰ (Fig. 11). The values are corrected for charring by subtracting 0.34‰ from the determined values. The data are normally distributed and an analysis of variance indicated no significant difference in values between archaeological horizons ($F(4, 10) = 0.97$, $p = 0.464$). The *P. glaucum* samples are colour-coded by the manuring level imputed using the fitted linear model (Fig. 11), regressing the determined *P. glaucum* $\delta^{15}N$ values against the interpolated average annual rainfall value for 1970–2000 from WorldClim version 2 of 391 mm (Fick and Hijmans 2017). Using the average recent rainfall gives a conservative estimate of the manuring levels of the *P. glaucum*, as there are also periods when it may have been a little wetter (Lespez et al. 2011, Fig. 12). In this preliminary study we focus more on *trends* in the $\delta^{15}N$ values during the occupation of the site, rather than seeking to reconstruct absolute manuring levels, which would require more accurate estimates of past rainfall. Figure 11 shows that the $\delta^{15}N$ values and manuring levels of the *P. glaucum* grains remain

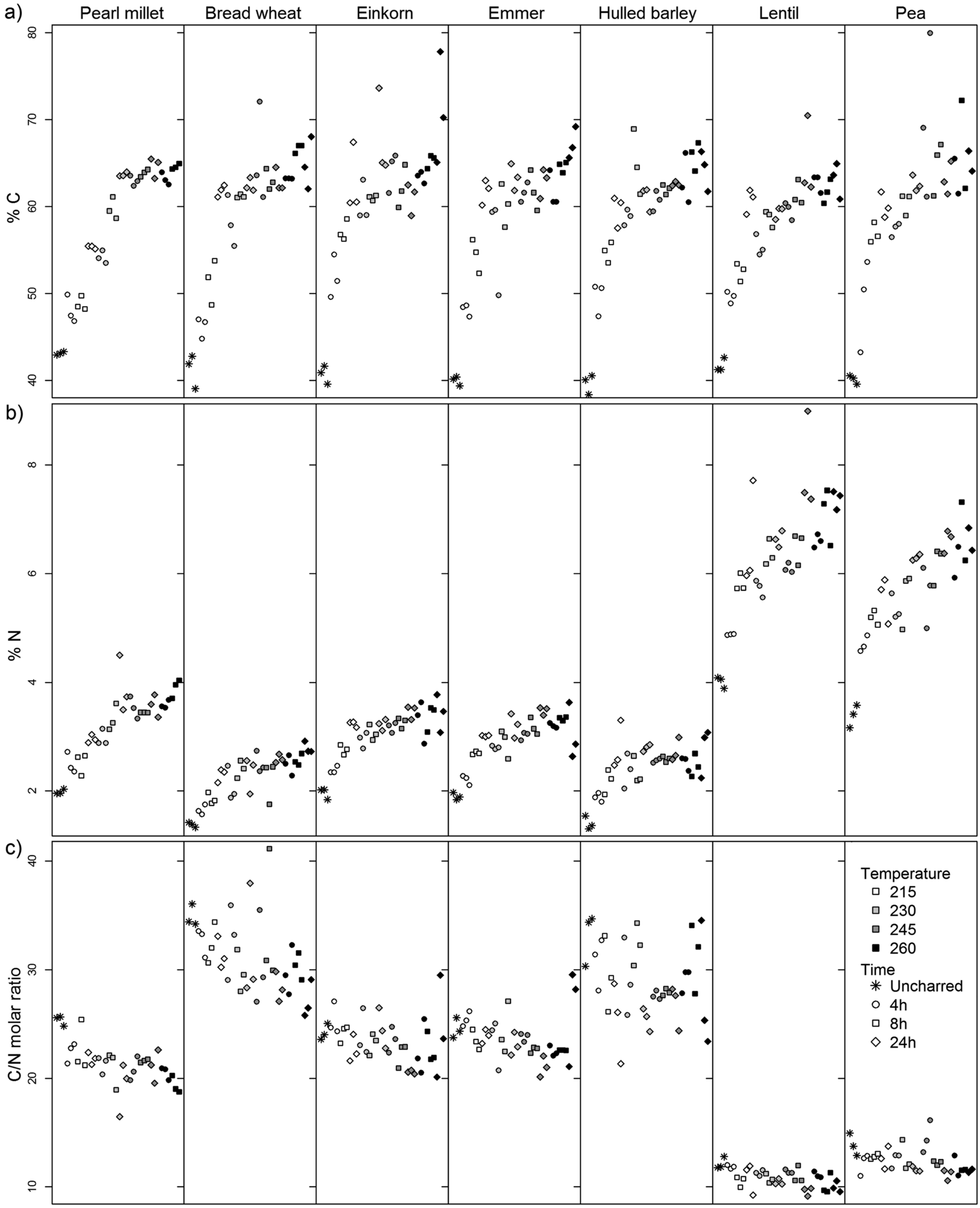

Fig. 9 **a** Changes in %C; **b** in %N; **c** C/N molar ratio due to charring, for seven different crop taxa charred at 215–260 °C for 4–24 h. The data for all taxa apart from *P. glaucum* have been previously published in Nitsch et al. (2015)

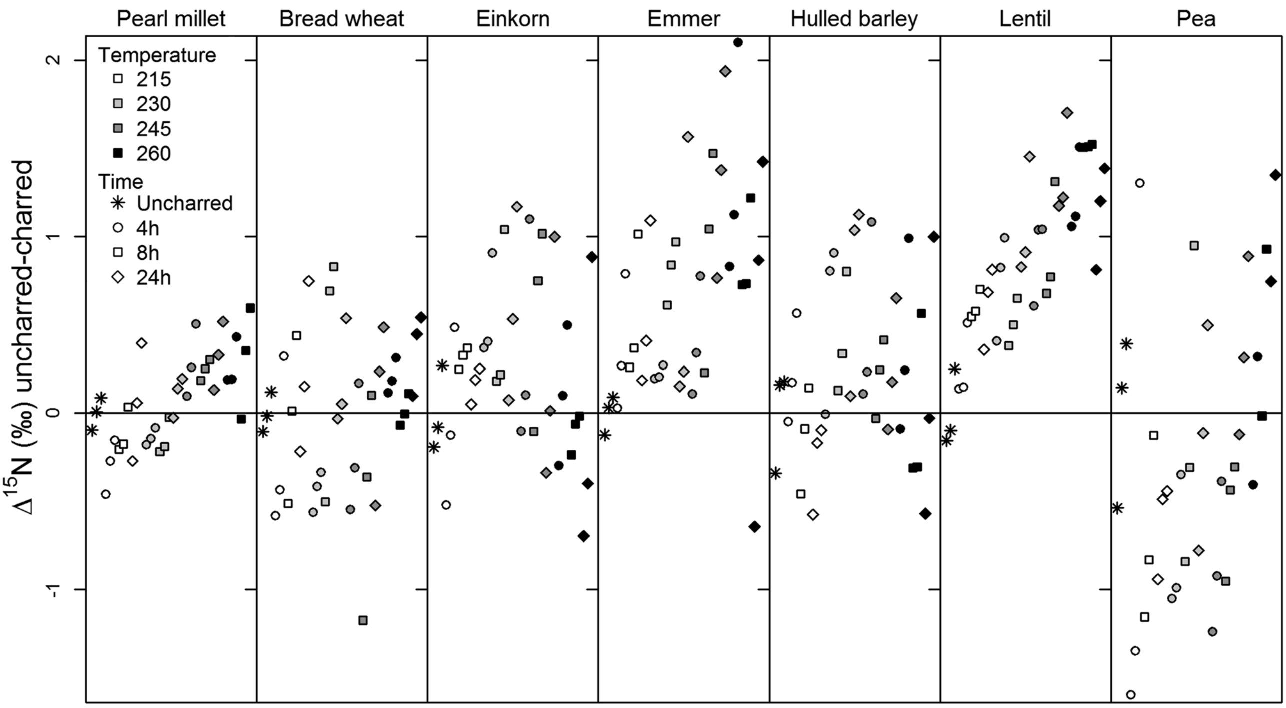

Fig. 10 δ^{15}N values of seven crop taxa charred at 215–260 °C for 4–24 h compared to the mean value of three uncharred replicates. The data for all taxa apart from *P. glaucum* have been previously published in Nitsch et al. (2015)

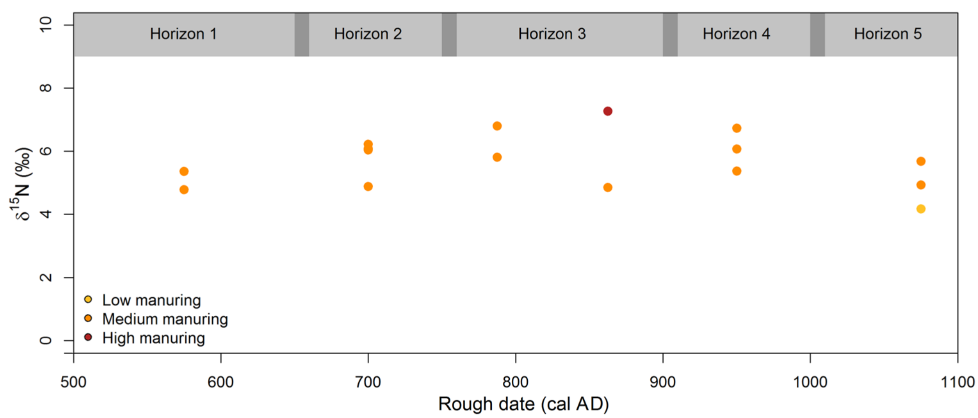

Fig. 11 δ^{15}N values of *P. glaucum* grain samples from Tongo Maaré Diabal, Mali, plotted against date. The crop samples are grouped by archaeological horizon and plotted against the midpoint of the date range for each horizon, apart from Horizon 3 where an upper and lower portion were distinguished in the archaeology. The horizons are dated according to stratigraphic relationships to radiocarbon dated contexts. The points are colour coded by their manuring level, which is imputed from the δ^{15}N values and the present-day rainfall at the site using a fitted linear model

relatively constant throughout the occupation of the site, with all but two of the samples being classified into the medium manuring level.

Discussion

Variability in *P. glaucum* δ^{15}N within single panicles and plots

The 0.7‰ maximum range in *P. glaucum* grain δ^{15}N values within single panicles is much less than the 5.7‰

range in the values of *Setaria italica* (foxtail millet) grains from within panicles of different accessions observed by Lightfoot et al. (2016). This range is also smaller than the 2.0‰ range in values of wheat grains sampled from within a single ear (Bogaard et al. 2007). All studies of intra-panicle/ear variability show that there is no consistent change in the values of grains sampled from the base to the tip of the panicle/ear, which means that random sampling of grains is unlikely to bias the δ^{15}N results.

The 4.8‰ maximum range in *P. glaucum* grain δ^{15}N values sampled within farming plots is large, but the mean 95% confidence interval of ± 1.1‰ is similar to the ± c. 1‰ proposed by Nitsch et al. (2015) to account for variability within a single growing context. It should be noted, however, that in three of the plots the 95% confidence interval is greater than this (up to 1.7‰). Lightfoot et al. (2016) found a 1.0‰ range in *S. italica* grain δ^{15}N values for a single accession grown in different positions in a growth chamber, but in this case the plants were grown in the same compost and were therefore not influenced by different soil conditions. They observed a 6.0‰ range in values for different *S. italica* accessions; some of this variation was found to relate to the latitude from which the local variety was sampled and flowering time of the accessions. Since the different accessions sampled by Lightfoot et al. (2016) derive from populations from diverse locations across Eurasia and Africa, it is unlikely that genetic and phenotypic variation in millet grains from within a single archaeobotanical assemblage would be as great as in this study. The variability in *P. glaucum* δ^{15}N values due to inadvertently sampling different land races is therefore unlikely to be greater than the variation observed within plots. Indeed, it is likely that the large variation we observe in values is due to the variability in manure/household waste application across individual plots, with farmers targeting *areas* of plots for manure application rather than applying it at consistent rates across an entire plot. It is important to bear this in mind when interpreting manuring practice in the past and argues for homogenisation of multiple archaeological grains to minimise the chance that they originated from a single plant growing in localised conditions.

The effect of manuring on *P. glaucum* δ^{15}N values

The effect of manuring on *P. glaucum* grain δ^{15}N values growing in three regions of Senegal was found to be significant with a non-parametric statistical test (Mann–Whitney U), but non-significant with a parametric statistical test (Student's t test) at a significance level of 0.05. This suggests that the intensity of and variability in manure/household waste application surveyed in this study does not result in significantly higher values. In the regions studied, application of manure/household waste is in fact managed in such a way as to *maintain* overall crop productivity rather than *increase* it in certain plots. Such variability in crop δ^{15}N values between manured and unmanured plots has been observed in other traditional, small-scale farming systems in Tighirt, Morocco (Bogaard et al. 2018b; Styring et al. 2016), Kastamonu, Turkey (Bogaard et al. 2016) and Sighisoara, Romania (Fraser et al. 2011) (Fig. 7a). In fact, the effect of manuring/fertilisation has to override the confounding effects of other factors, such as water availability, vegetation cover and soil properties, on the cycling and loss of nitrogen in order to result in significantly higher δ^{15}N values. It may also be the case that only prolonged differences in manuring practice over the relatively long term, for a decade or more, result in a very clear difference in crop δ^{15}N values. Fraser et al. (2011) found that the values of barley grains from a previously-manured plot that stopped receiving manure in 1871 had not decreased significantly after 11 years. The barley values only decreased by the date of the next isotopic determination, 30 years after manuring ceased (Fraser et al. 2011).

Nonetheless, the *P. glaucum* δ^{15}N values from Senegal provide an indication of the values to expect for *P. glaucum* receiving low to medium levels of manure in a similar climate. These values also fit well into the fitted linear model that regresses cereal grain values to mean annual rainfall, for low, medium and high manuring levels (Fig. 7). Although the model tends to classify plots receiving low/no levels of manure as having received medium levels of manure, only one of the medium manured plots is falsely classified as receiving high levels of manure. Nitrogen isotope values of *P. glaucum* can therefore be used to exclude or confirm *intensive* application of manure/household waste, rather than to distinguish medium level manuring from no/low manure application. These findings also underline the fact that exploring long-term trends in crop δ^{15}N values is more informative than trying to determine absolute levels of manuring from individual values.

The effect of charring on *P. glaucum* δ^{15}N values

Heating *P. glaucum* grains at 215–260 °C for 2–24 h or at 260 °C for up to 8 h results in well-preserved charred grains that resemble archaeobotanical samples that can be identified to species (ESM 5). This is a similar 'charring window' to that determined for barley, naked and glume wheat grains and pulse seeds (Charles et al. 2015). The decrease in mass, increase in %C and %N and increase in δ^{15}N values of *P. glaucum* grains with charring are similar to the changes observed in other cereal grains and pulse seeds (Figs. 8, 9, 10, ESM 6; Nitsch et al. 2015), resulting from loss of water and other volatile compounds (Styring et al. 2013). The 'worst-case' 0.34‰ increase in *P. glaucum* values with charring is very similar to the 0.31‰ average offset between uncharred and charred barley and wheat grain and pulse seed values calculated by Nitsch et al. (2015). In fact, when a multiple linear model is calculated for the effect of charring on all seven taxa including *P. glaucum* (Fig. 8), with coefficients for each of the taxa, the δ^{15}N value charring offset is calculated to be 0.28‰ (Beta = 0.28, SE = 0.12, $p = 0.0153$), with a 95% confidence interval of 0.06 to 0.51‰.

Reconstructing manuring practice at Tongo Maaré Diabal, Mali

The δ^{15}N values of *P. glaucum* grains recovered from Tongo Maaré Diabal (TMD) are similar to those sampled from present-day plots in Senegal that received low to medium levels of manure/household waste. It is difficult to reconstruct the annual rainfall during the period when the site was occupied, but according to Lespez et al. (2011, Fig. 12), the desiccation index in the area varied only slightly between AD 500 and 1150 and was similar to that observed today. It is therefore likely that the *P. glaucum* plots at TMD received low levels of household waste and/or manure from animals grazing after harvest. There is no obvious change in *P. glaucum* δ^{15}N values through time, although it is interesting that the only *P. glaucum* grain sample classified as receiving high levels of manure derives from a context post-dating AD 750, which is when the number of cattle bones on the site increased. Assuming that the rainfall did not change dramatically during the occupation of the site (as data presented in Lespez et al. 2011 would suggest), there is no sign of a decrease in manure application during the occupation of the site, implying strategic management of resources to ensure continuing crop productivity and soil fertility. This would have been particularly important, since the predominance of *P. glaucum* in the archaeobotanical assemblage would seem to preclude crop rotation or green manuring with legumes as alternative strategies for replenishing soil nutrients. Equally, the lack of increase in *P. glaucum* δ^{15}N values during the 650 year occupation of the site implies that manure/household waste was added relatively sporadically, rather than applied to the same plots annually. This continuity in manuring practice reflects the general lack of change in the seed and charcoal assemblages observed by Champion (this study) and Uebel (Gestrich and MacDonald 2018).

The apparent absence of a regional settlement hierarchy may mean that there was no external pressure to increase agricultural production to supply an urban centre. Nonetheless, the possibility that TMD supplied iron to a wider trade network connected to the Empire of Ghana makes it unlikely that the community was not influenced by increased demand for agricultural production to support iron-working specialists. The dispersed nature of the settlements means that it is unlikely that there was pressure on land availability to encourage agricultural intensification. In fact, if the increased number of cattle bones recovered from the site after AD 750 translated to an increase in the availability of manure, the continuity in *P. glaucum* δ^{15}N values could indirectly reflect an expansion in the cultivated land, with the larger quantities of manure spread over a wider area. This implies moderate agricultural expansion, on a scale where the level of manuring could be maintained but not increased.

It is likely that the low number of samples, which lack spatial resolution within archaeological horizons, precludes the identification of plots receiving high levels of manure/household waste. Future excavations will hopefully yield more *P. glaucum* samples and allow an assessment of the spatial variability in manuring practice—perhaps mirroring cultivation practices in the present and recent past where management and land use intensity tends to decline in concentric rings around household compounds (Pelissier 1966; Prudencio 1993). Further excavations may also gain a better insight into the changing economic role of TMD in the large scale production of iron over time, which would help to explore whether there were likely drivers for increased agricultural production. Future efforts to reconstruct the past climate in the region would also help to determine whether the *P. glaucum* δ^{15}N values were influenced by changing annual rainfall.

Conclusions

The δ^{15}N values of *P. glaucum* grains sampled from plots receiving low to medium inputs of manure in three regions in Senegal range between −0.6 and 6.6‰, largely consistent with values estimated from a model relating crop δ^{15}N values to manuring level and annual rainfall. The lack of a significant increase in values with higher inputs of manure reflects the variability in manuring practice and the lack of intensive and prolonged manuring of particular plots in the regions studied. Isotopic analysis of *P. glaucum* grown with high inputs of manure (similar to the 10 + t/ha applied in experimental farming plots in Europe; Fraser et al. 2011) is necessary in the future to definitively confirm whether *P. glaucum* values do increase significantly with higher manure inputs. Charring of *P. glaucum* grains by heating at 215–260 °C for 4–24 h increases their δ^{15}N values by a maximum of 0.34‰, comparable to the increase in values of barley and wheat grains, and pulse seeds. In the light of these modern data, the values of *P. glaucum* grains recovered from the archaeological site of Tongo Maaré Diabal, Mali, are consistent with low to medium levels of organic matter input throughout the occupation of the site from AD 500–1150. This is the first time that the δ^{15}N values of *P. glaucum* grains growing under known conditions in the Sahel have been determined and demonstrates what can be gained when botanical remains are collected at archaeological sites, and when modern traditional farming practices in climatically relevant areas are studied. This work has widespread implications for the reconstruction of farming practice and scale in the West African Sahel, where *P. glaucum* is a staple crop.

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