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CHRONOLOGY OF FOSSILIFEROUS LOCALITIES IN MANJRA VALLEY, DISTRICT LATUR, MAHARASHTRA, INDIA

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ABSTRACT. The present paper is an announcement of first absolute age dating directly on fossilized bones and teeth of the Pleistocene mammalian fauna from the Manjra valley, District Latur, Maharashtra, India. The fossilized samples were measured using the AMS facility at Inter-University Accelerator Centre, New Delhi, India. The results gave the time frame of 21,423 BP to 24,335 BP that correspond close to and the threshold of LGM and sheds important light on the palaeoecology of the area that supported diverse megafaunal species in the Upper Manjra valley. These calendar dates not only have wider significance in terms of first ever approximate chronological frame for the Pleistocene fauna in Peninsular India but also offer methodological innovations especially when the adequate bioapatite is absent in the fossilized bones and teeth from the fossil record.

KEYWORDS: accelerator mass spectrometry, Late Pleistocene, Manjra valley, radiocarbon dating, vertebrate fossils.

INTRODUCTION

The River Manjra, the major southerly tributary of the Godavari, is known for the occurrence of fossilized skeletal remains of large and small vertebrates, whose taxonomic diversity is only next to the Narmada valley in central India. It originates in the Ahmednagar District of Maharashtra and after flowing for a distance of about 724 km through the states of Karnataka and Telangana, meets the River Godavari near the village Kandhakurthi in Nizamabad District of state of Telangana, India (Figure 1). In the upper reaches, especially within the vicinity of districts of Beed and Latur, a fossil trove was unearthed some 45 years ago, which emerged as a landmark discovery in revealing the evidence of a rich prehistoric wildlife during the Late Pleistocene period in southeastern Maharashtra (Joshi et al. 1981; Sathe 1989). The taphonomic history of the fossil record shows the evidence of various events of accumulation and deposition that range from the drought(s) to fluvial contexts (Sathe 1989, 2004). Litho-stratigraphy of the fossil yielding horizons led to the assignment to the Late Pleistocene period (Joshi et al. 1981; Sathe 1989). Two of the rich fossil localities viz. Tadula (18º32′50″N, 76º23′E), and Wangdari (18º33′N, 76º24′25″E), located in Beed District of Maharashtra have reported abundant fossilized mollusks in association with vertebrate fossils which based on the lithostratigraphic association with bones and embedded matrix of shells indicated that temporally they shared the same horizon. These shells especially from Tadula and Wangdari were subjected to radiocarbon (^{14}C) dating at the Birbal Sahni Institute of Palaeosciences (formerly Birbal Sahni Institute of Palaeobotany), Lucknow and were dated to 26,820 ± 750 BP and 34,470 ± 2070 BP, respectively, which assign late Pleistocene age to the faunal assemblage in Manjra valley (Sathe 1989).

The recent discovery of a fossil bone bed at Harwadi (18º27′40″N, 76º35′38″E), about 45 km farther downstream of the aforesaid known fossil localities further added new fossil discoveries to that made the detailed examination of the fauna imperative. The undisturbed fossil assemblage with a minimal taphonomic modifications has been very helpful in explaining the environmental setting during the Late Pleistocene period in this region (Sathe 2005, 2015). The homogeneity in the degree of fossilisation, taxonomic diversity and lithostratigraphic contexts has helped in

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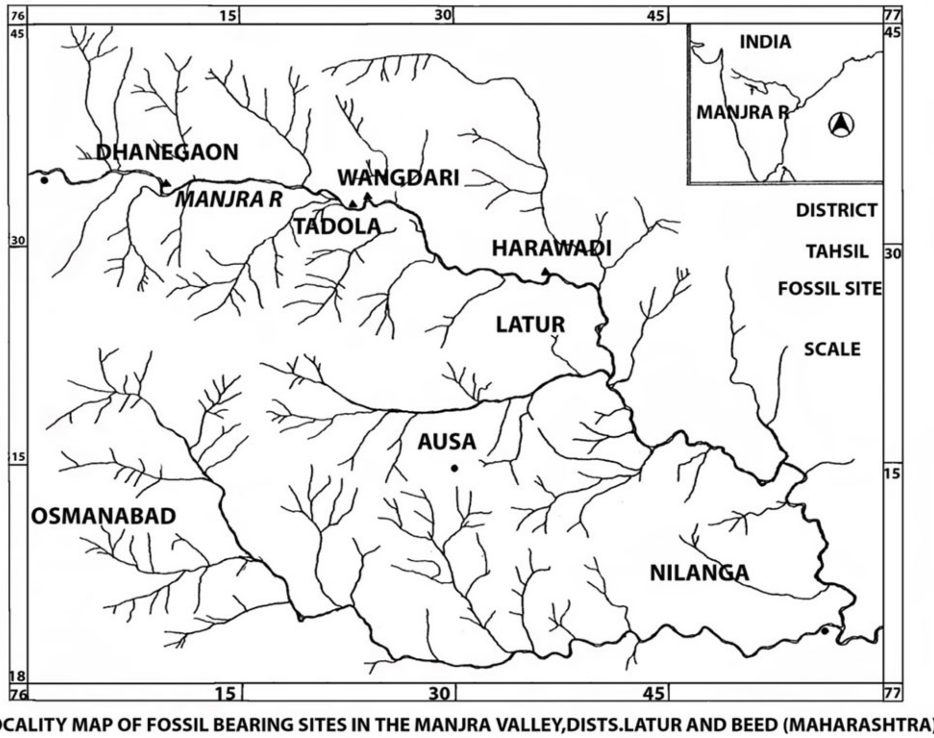

Figure 1 Locations of fossil-bearing sites in Manjra valley.

assigning the events of death and burial within the close proximity of Harwadi. This phenomenon is contemporary to the earlier known fossil bearing sites like Dhanegaon, Wangdari, Ganjur, and Tadula. There have been no further dates available and therefore, the earlier dates have remained the point of reference for chronology of this newly discovered fossiliferous horizon in the Manjra valley.

The age of large mammalian fossil yielding deposits in the Upper Manjra valley (Beed and Latur districts), has been dated to the time range between 22,000 to 34,000 years on the basis of conventional ^{14}C dating of molluscan shells. This is the first occasion to have bones directly dated using accelerator mass spectrometry (AMS) dating, which reflects precise chronology of megaherbivores that lived in the valley during the later part of the Pleistocene period in the Manjra valley. The present work has yielded new dates that can contribute to our understanding of the paleoenvironmental conditions, especially when the Terminal Pleistocene landscape was experiencing major climatic shifts prior to the LGM.

Faunal History of the Region

As with most of the fossil discoveries, the prehistoric faunal wealth of the region came to light when the alluvial cliffs were being dug on the banks of river Manjra in District of Beed in Maharashtra by the Irrigation Department of state of Maharashtra in early seventies of the last century. The discovery of the fossil potential prompted a three-year project undertaken by the geologists, prehistorians and a palaeontologist of Deccan College, Pune, who

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explored the entire Manjra valley from origin to its confluence with Godavari and brought to light in the context of alluvial flood plains several prehistoric sites with late Acheulian, Middle Palaeolithic, and Mesolithic tools but the fossil sites were found to be limited to the District of Beed in Maharashtra state (Joshi et al. 1981). Faunally and litho-stratigraphically, there appears a general uniformity in the faunal wealth of Manjra, Godavari and other river valleys of Western India which chronologically fall between 25,000 to about 40,000 years of history of their survival. The fossil record includes about 20 genera and 25 species of large to medium-sized mammals, reptiles, microvertebrates, and mollusks which along with the Early, Middle, and Upper Palaeolithic tools mark the region a conducive landscape for survival of prehistoric man for a continuous occupation from Late Acheulian to the Mesolithic (Tables 1–3).

The multispecies mammalian fauna in the Manjra valley is an admixture of megafauna that had either Ethiopian or Palaearctic origins as being the descendants of the Siwalik holdovers in the later part of the Pleistocene. However, of all the reported, there are eight genera (mostly artiodactyls) which are endemic to Indian subcontinent and appear to represent their wide home range throughout the Pleistocene (Turvey et al. 2021). It has been argued that the southward migration of post-Siwalik fauna led to a mosaic of palaeo-communities that thrived throughout Peninsular India until the known episodes of dry and arid phase triggered by the LGM.

Importance of the Site and its Faunal Diversity

Even if the Pleistocene faunal wealth of Peninsular India brings about a tapestry of diverse vertebrate fauna, its distribution and occurrence has been defined by fluvial behaviour and hydrodynamic sorting (Badam et al. 1986; Badam and Jain 1988; Sathe 1989; Patnaik et al. 2009). However, the fossil discoveries in the alluvial deposits of the River Manjra at the site of Harwadi provided minimal taphonomic modifications despite fluvial dispersal of the fossil assemblage. The faunal diversity supplying a rich data of palaeo-community is further authenticated by a vast body of evidence related to vegetation, palaeodiet and environment (Sathe 2015; Sathe et al. 2018) which has amply shown their role in maintaining the large mammalian population in the vicinity.

The wet land conditions enabled perennial grass cover and galleria forests along the river banks, which is also supported by predominant grazers preferring C4 diet along the forests, interspersed with open grasslands in the region. The presence of an immigrant carnivore (tiger) at Harwadi is a major landmark taxon (Sathe and Chakraborty 2016), especially given that its arrival in India has been considered to be not older than 12 kyr BP (Mondol 2011). Even if this is accepted, the tiger arriving in Indian Peninsula during the end phase of LGM shows the presence of ecologically supportive corridors in the southeast China, which enabled the movement of large felids into India.

The Indian Peninsula has lost only 9 from a total of 114 modern taxa, namely hippopotamus, two species of elephants, and horse, to environmental change and alleged anthropogenic intervention at the end of the Pleistocene (Jukar et al. 2021; Turvey et al. 2021; Sathe 2022). The African continent, in comparison, showed 24 species and 3 genera disappearing as part of the Late Quaternary extinction window (Faith 2014). Southeast Asia lost 2 elephant genera, the orangutan, hyenas, the giant panda, tapirs, rhinoceroses, and the giant Asian ape, due to climatic and the sea level changes (Louys 2007). Among all the megaherbivores,

Table 1 Pleistocene vertebrate fossil localities in the Deccan province, Maharashtra and Karnataka, indicating age, association with Stone Age tools and checklist of fossil assemblage found at individual site.

Fossil site	Material used for dating	Association of Palaeolithic cultures	Associated taxa	Period	Relative dating fluorine	Dating method (absolute)	Lab no.	Reference
Fossil sites in Western Maharashtra								
Gangapur (Godavari basin)	—	Lower Palaeolithic	*Bos namadicus* (Badam 1979; Kumar 1985; Pilgrim 1905)	Middle Pleistocene	—	26635 ± 425 (^{14}C)		Kumar 1985
Nandur Madhameshwar (Godavari basin)	Freshwater mollusc-an shells	Middle Palaeolithic	*Elephas antiquus, Bos namadicus (Pilgrim 1905)*	Late Pleistocene		27410 ± 425 (^{14}C)	BS163	Mishra 1995; Rajgopalan et al. 1982
Paithan (Godavari basin)	Ostrich eggshell	Middle Palaeolithic	*Bos* sp., *Elephas namadicus*	Late Pleistocene	—	—	—	—
Patne (Tapi basin)		Upper Palaeolithic	*Struthio camelus*	Late Pleistocene		25000 ± 200 (^{14}C)	GRN7200	Sali 1989
Rangna Jhola (Wardha basin)	—	—	*Bos namadicus, Bos/ Bubalus, Elephas namadicus, Antilope cervicapra, Hexaprotodon palaein-dicus, Trionyx sp.* (IAR 1997–1998: 56–57).	Late Pleistocene	—	C ~ 30–40 k	—	—
Hingoli (Painganga basin)	—	—	*Elephas antiquus, Hippopotamus palaeoin-dicus, Equus namadicus*	Late Pleistocene	—	—	—	—
Morgaon (Karha basin)	Volcanic ash, ostrich eggshells, bivalves.	Acheulian, Microliths	*Bos elaphus tragocamelus, Bos/Bubalus* sp., *Equus* sp., *Struthio camelus* c.f. *S.c. molybdophanes (?)* (Sathe 2008)	Middle Pleistocene – Late Pleistocene	1.69, 6.07, 6.39	984 ± 25 ka, 26, 37977 ± 10 ka(^{14}C), 22,485 ± 7320–310 ka (OSL)	BS1230	Westaway et al. 2011; Mishra et al. 2003; Sathe 2008

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Table 1 (*Continued*)

Fossil site	Material used for dating	Association of Palaeolithic cultures	Associated taxa	Period	Relative dating fluorine	Dating method (absolute)	Lab no.	Reference
Bori (Kukadi basin)	Volcanic ash	Acheulian	*Bos*, *Elephas*, *Equus namadicus*	Middle Pleistocene	3.22–8.05	815 ± 34 ka (±2σ)		Westaway et al. 2011; Kshirsagar 1993
Chirki (Pravara basin)	Volcanic ash, fossil bone	Acheulian	*Bos namadicus*	Middle Pleistocene	8.18–8.29	>350 ka 0.8 ma	—	Atkinson et al. 1990; Kshirsagar 1993
Kalegaon (Pravara Basin)	Fossil bone	Middle Palaeolithic	*Bos namadicus*	Upper Pleistocene	6.3	—	—	Kshirsagar 1993
Yeldari dam (Purna basin)	—	—	*Bos namadicus*, *Elephas hysudricus*, *Stegodon insignis* *Elephas* sp.	Late Pleistocene	—	—	—	—
Inamgaon (Ghod basin)	Freshwater molluscan shells	Middle to Upper Palaeolithic and Microliths	*Elephas maximus*, *Elephas hysudricus*, *Bos namadicus*, *Bubalus sp.* *Hexaprotodon palaeindicus*	Late Pleistocene	—	19290 ± 360 21725 ± 605 (^{14}C)	TF 1177 TF 1003	Agrawal and Kusumgar 1975
Chandoli (Ghod basin)	—	—	*Cervus unicolor*,	Late Pleistocene	4.1	—	—	Kshirsagar 1993
Kalabm (Ghod basin)	Freshwater molluscan shells	—	*Equus namadicus*, *Equus asinus*, *Equus* sp., *Sus palaeindicus*, *Canis* sp. (Badam 1988; Badam and Kajale 1977; Kajale 1979)	Late Pleistocene	4.03–4.33	—	—	Kshirsagar 1993

(*Continued*)

Table 1 (*Continued*)

Fossil site	Material used for dating	Association of Palaeolithic cultures	Associated taxa	Period	Relative dating fluorine	Dating method (absolute)	Lab no.	Reference
Harwadi (Manjra basin)	—	Late Acheulian (?)	*Hystrix cressidens(?), Panthera* cf. *P. tigris, Canis* sp., *Rhinoceros* cf. *R. unicornis, Equus namadicus, Elephas maximus, Elephas namadicus, Elephas hysudricus, Stegodon insignis-ganesa, Cervus duvauceli, Cervus unicolor, Axix axis, Bos namadicus, Bubalus palaeindicus, Boselaphus tragocamelus, Antilope cervicapra, Trionyx* cf. *Gangeticus, Crocodylus palaeindicus.* (Joshi et al. 1981; Sathe 1989, 2015)	Late Pleistocene	5.72	23981–23701 cal BC 26879–26277 cal BC 25271–24506 cal BC	IUACD#19C2580 IUACD#19C2581 IUACD#19C2582	Present study
Wangdari (Manjra basin)	Freshwater molluscan shells	—		Late Pleistocene	4.48	26820 ± 750 (^{14}C)	BS 562	Sathe 1989
Tadola (Manjra basin)	Freshwater molluscan shells	—		Late Pleistocene	2.5–6.00	34470 ± 2070 (^{14}C)	BS 561	Sathe 1989
Ganjur (Manjra basin)		—		Late Pleistocene	2.5–6.00	—	—	Kshirsagar 1993
Dhanegaon (Manjra basin)	—	—		Late Pleistocene	5.12	—	—	Kshirsagar 1993
Vadod (Purna basin)	—	Late Palaeolithic	*Bos namadicus, Bos bubalus Equus namadicus, Equus heminous khur, Antelope* cf. *cervicapra, Reptiles (turtles), Molluscan shells Elephas namadicus* (Badam et al. 2018; Thakur et al. 2018)	Late Pleistocene	—	—	—	—
Fossil Sites in Karnataka (Southern Deccan)								
Isampur, Hunsgi – Bhaichbal valleys, Dist Kalburagi	Bovid teeth	Early Acheulian	Bovids (Paddayya 2001)	Early Middle Pleistocene	—	1.27 myr (ESR dating)		Paddayya et al. 2002

Table 1 (*Continued*)

Fossil site	Material used for dating	Association of Palaeolithic cultures	Associated taxa	Period	Relative dating fluorine	Dating method (absolute)	Lab no.	Reference
Anagwadi, Dist. Vijaypura	—	Early Acheulian (Pappu 2001)	—	Late Middle Pleistocene	—	>0.08 myr		Deo and Rajaguru 2014
Khyab, Dist. Bagalkot	—	Early Acheulian (Pappu 2001)	—	Late Middle Pleistocene	—	—	—	—
Sadab, Hunsgi valley, Dist Kalburagi	*Elephas* molar	Acheulian Fartifacts	*Elephas* sp., Stegodont/ *Elephas*	Late mid Pleistocene	2.14	290,405 ± 20,999–18,186	—	Szabo et al. 1990; Sathe and Paddayya 2013
Kaldevanhalli Hunsgi valley, Dist Kalburagi	Travertine under-lying rubble containi-ng Acheuli-an artifacts	Acheulian artifacts	Cervids (Sathe and Paddayya 2013)	Late mid Pleistocene	6.72	174,000 ± 35,000[2]	—	Szabo et al. 1990; Kshirsagar and Paddayya 1989
Teggihalli, Hunsgi valley, Dist Kalburagi	Molar of *Bos* sp.	Acheulian artifacts	*Bos* sp., cervid (Sathe and Paddayya 2013)	Late mid Pleistocene	2.9	287,333 ± 27,169 –18180	—	Szabo et al. 1990; Sathe and Paddayya 2013
Yediapur, II – IV, Hunsgi-Baichbal valley, Dist Kalburagi	—	Middle Acheulian	*Panthera* c.f. *Panthera tigris, Equus namadicus* (Sathe and Paddayya 2013)	Late mid Pleistocene	2.57– 5.4	—	—	Kshirsagar and Paddayya 1989

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Table 1 (*Continued*)

Fossil site	Material used for dating	Association of Palaeolithic cultures	Associated taxa	Period	Relative dating fluorine	Dating method (absolute)	Lab no.	Reference
Hebbal, Hunsgi-Baichbal valley, Dist Kalburagi	—	Acheulian	Large mammals, Bos namadicus (Paddayya 1969; Sathe and Paddayya 2013)	Late mid Pleistocene	6.69–8.39	—	—	Sathe and Paddayya 2013; Kshirsagar and Paddayya 1989
Yedurwadi (Shirguppi), Dist Belagavi	Calcrete	Late Acheulian	*Elephas* sp., *Hexaprotodon* sp. (Kale et al. 1986)	Late mid Pleistocene	4.44–6.29	>350,000 BP, <0.8 Ma to ~350 Kyr	—	Atkinson et al. 1990; Deo and Rajaguru 2014; Kshirsagar 1993
Nittur, Dist Bellary	—	Acheulian	*Bos namadicus* (Ansari 1971; Sathe et al. 1986)	Late mid Pleistocene	—	—	—	—
Kolkur, Dist Kalburagi	—	—	*Hexaprotodon deccanensis, Equus namadicus,* (Anatharaman et al. 2005; Moitra et al. 2002)	Late mid Pleistocene	—	—	—	—
Hagargundi, Dist Kalburagi	—	Middle Palaeolithic	*Bos* sp, *Elephas* sp. (Paddayya 1969)	Late Pleistocene	4.59–5.28	—		Kshirsagar and Paddayya 1989
Chikdauli, Dist Belagavi	—	Palaeolithic implements	*Rhinoceros deccanensis, Elephas* sp, *Bos sp.* (Foote 1876)	Late-mid to Late Pleistocene	—	—	—	—

Table 2 First batch of samples, which included fossils from Manjra, Ghod, and Narmada river valleys.

Sample	Taxon	Element
INM 6	Bos/Bubalus	Mandible
4MSR 545 (BNJ)	Equus	Molar
4MSR 398 (BNJ)	Equus	Molar
INM 4	Hexaprotodon	Molar
MNJ 938	Cervus	M3
MNJ 1008	Hexaprotodon	Molar
WDH 1	Hexaprotodon	Molar
NMD 231	Hexaprotodon	Molar
MNJ 864	Hexaprotodon	Molar
24	Equus	Molar
MNJ 861	Cervus	M2
NMD 102	Hexaprotodon	Incisor
MNJ 3	Cervus	M2
NMD 53	Hexaprotodon	Molar
MNJ 940	Bos/bubalus	M2
MNJ EL	Elephas	Molar
MNJ 170	Stegodon	M3
MNJ 967	Rhinoceros	Molar
NMD 25	Equus	M2
MNJ 983	Elephas	Molar

Table 3 Second batch of samples.

Sample	Taxon	Element
MNJ dentine powder	Elephant	Ivory
MNJ dentine powder (acid treated)	Elephant	Ivory
MNJ crushed dental enamel	Hippopotamus	Molar
MNJ bone powder	Large bovine	Vertebra, pelvis

hippopotamus disappears practically from all places that shows significant climate change resulting in considerable loss of their habitat and access to perpetual water bodies. This ecological perspective needs to be understood in a chronological frame, especially in context of the rest of the world experiencing major climatic upheaval owing to the onset of aridity and glacial episodes.

There is an important need to focus on obtaining a large body of reliable ^{14}C dates obtained directly on securely identified megafaunal remains in this region of Peninsular India. So far there are majority of ^{14}C dates available on mollusks that are reportedly found in association of fossils or representing the same fossil bed. Relying on these dates requires the assumption that the shells and the megafaunal fossils are contemporary—which may not be the case depending on the taphonomic environment. In addition, studies have shown that the "hardwater" reservoir effect, and diagenesis of the outer layers of the shell can

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affect the age produced from these samples, rendering them less reliable (Preece et al. 1983; Yates 2016; Wright 2017).

Methods like fluorine dating are not entirely reliable except that they allow a concept of Late Pleistocene chronology to the fossiliferous beds but not the fauna that lived along the river valleys and its galleria forests. The strength of a chronology heavily depends upon the quality of dates. Therefore, the calibrated AMS dates freshly obtained on the megafauna from the site of Harwadi, using the methods described in the following sections, represent a beginning for the obtainment of direct AMS dates on fossil material—which can help in understanding the ecological proxies for the Terminal Pleistocene palaeoenvironment of the Manjra valley and Peninsular India.

Lithostratigraphy and Depositional Environment

The Quaternary formations in the area have a lateral extension of about 2 km and are confined to broad and shallow valleys cut into lower surfaces. Alluvial deposits are characterized by cut and fill types with thickness ranging from nearly 10 to 12 m. The Older Alluvium is represented by well cemented cross-bedded, poorly sorted, coarse-grained sandstone, and calcareous yellowish brown sandy siltstone, being the representative litho units in the region (Figure 2) The cemented sandy pebbly, poorly sorted, and coarse-grained sandstone has yielded all the fossils and the pattern of fossil scatter and mode of preservation and presentation renders it into the category of a bone bed.

The river Manjra originates in semi-arid plateau landscape at an elevation of 824 masl and flows throughout a similar landscape until its confluence with Godavari after completing a course of 724 km. The total area covered by the course of river Manjra within the district of Latur totals 126 km and the average rainfall is 800 mm, making it a wet land within the semiarid region of its course. Sedimentologically, the clay bands within the bone bed and lenticular patches of fissured clay represent pool phenomena that had adequate surface (channel pools, near channel, and wetlands) as well as groundwater recharging sand beds even in dry hot summers immediately before the LGM. This period also coincides with aggradational phase of river Manjra. Moderate slope of the valley, coupled with weaker currents eventually failed to transport the death assemblage and subsequent burial may have been possible only during the strong monsoonal floods.

The megavertebrate fossils are surface collected as well as excavated from the lithified sandy pebbly gravel or pebbly sandstone overlying cobbly, pebbly conglomerate and were subjected to detailed taphonomic analysis (Sathe 2005, 2015).

Sedimentology of the bone-bearing sandy beds reveals a 9–10 m of vertical section exposed along the left bank of Manjra. The bone-bearing deposit is a pebbly sandstone with reposited pebbles, silt and sand, with clayey bands, overlying well cemented calcareous sandy pebbly cross bedded gravel or pebbly orthoconglomerate above the Deccan Trap basalt. The fossils are found resting at an average dip of 20 degrees with a limited dispersal of the skeletal material, suggesting the moderate channel slope of the river. As the river was in aggradational mode, stream currents were too weak to transport the megavertebrates further downstream. As a result, some of the skeletal elements of hippos, elephants and bovids show a differential subaerial weathering indicating prolonged exposures prior to burial.

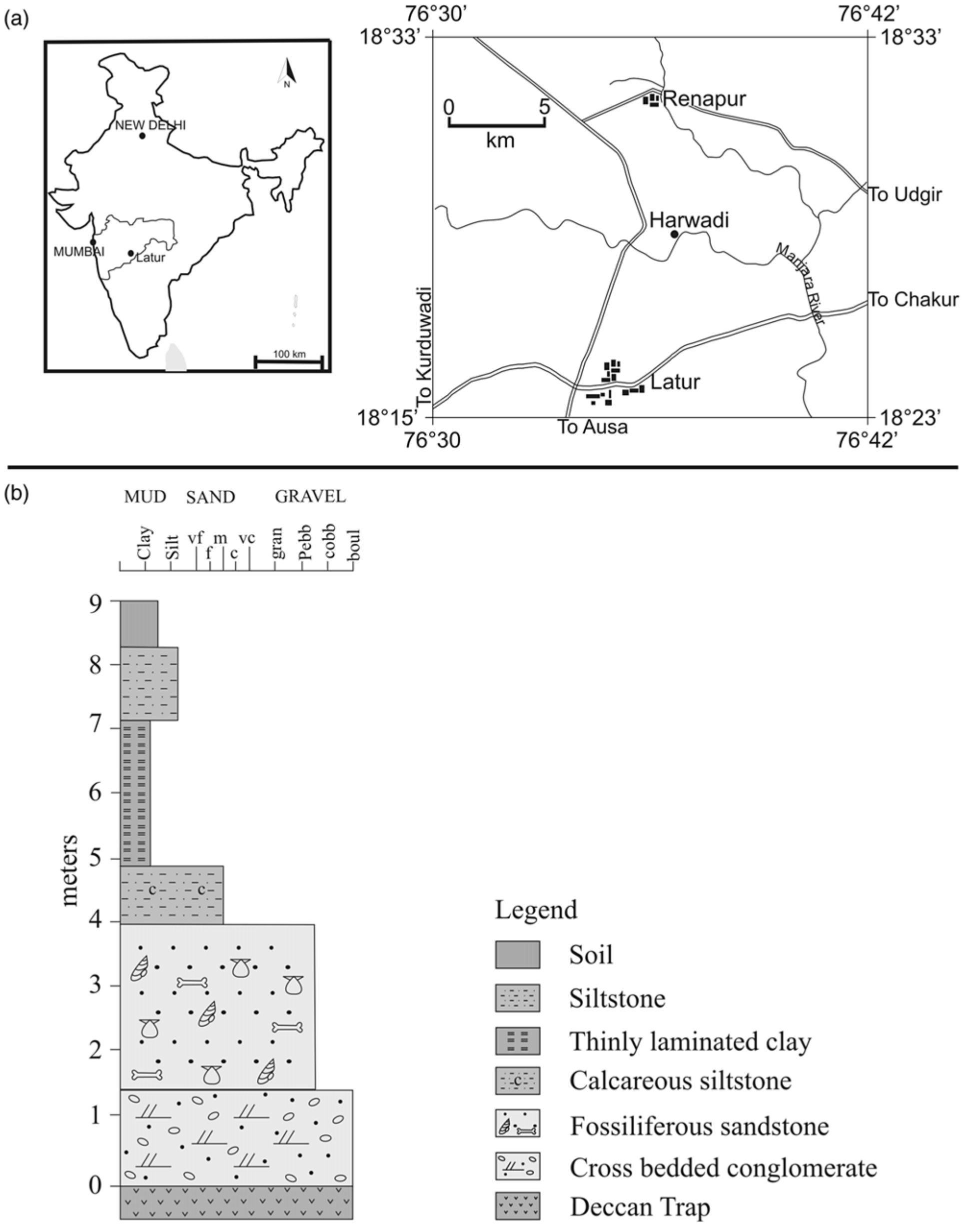

Figure 2 Lithostratigraphy of the fossil-bearing site of Harwadi.

Chronological Frame of Fossil Sites in Western Maharashtra with Special Reference to Manjra Valley

One of the first-ever chronological assessments of fossiliferous horizons in Peninsular India dates back to the beginning of the 20th century when Pilgrim (1905) compared the ossiferous gravels and fossils discovered by him at the banks of River Godavari in Nasik District of Maharashtra with that of Narmada valley. Based on litho-stratigraphical and fossil correlations he assigned them to the late Pleistocene period.

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Chronology of the Pleistocene fossiliferous horizons in Peninsular India traditionally finds litho-stratigraphical correlations of fossil occurrences, through osteological and morphometric approaches in assigning chronological horizons to type species and methods of absolute and relative dating, which collectively have been helpful in providing a time frame to the fossil record. In many cases, palaeolithic stone artifacts have been reported in association with vertebrate and molluscan fossils which also helps in providing a broad time frame that the typology provides to the lithic assemblage, besides throwing light on the prehistoric man-animal relationships.

Climatologically, the deposition of these fossil and implementiferous gravels correlate with phases of global aridity (Sadakata et al. 1995) but episodic aggradational events were favourable for the preservation of lithic and fossil evidence (Rajaguru and Mishra 1997). The sedimentary constituents of the Late Pleistocene sediments exhibit the high proportions of calcrete within the fine predominantly silt deposits, indicating distinctly arid climate with short spells of semi-arid climate (Deo and Rajaguru 2014). However, the occurrence of rich large mammalian fossil yielding localities in the Manjra valley strongly suggest a wet semi-arid phase chronologically falling into the MIS 3 phase. In other words, it is characterized by a wet phase within a prolonged period of aridity. This is sedimentologically indicated by the combination of fine and coarse sediments, sandy pebbly gravels with lenses of silt, seen in association with channel and near channel flood plain deposits.

A number of ^{14}C dates for the Late Pleistocene are available which cluster around 25 ka, 20–18 ka, and even stretch to the threshold of the Holocene to 12–10 ka, as shown in Table 1. However, most of these are on molluscan shells, Ostrich eggshells, volcanic ash, and calcrete. In contrast, very few dates are obtained directly on megafaunal fossil material and are obtained through U-Th series rather than ^{14}C. On the other hand, chemical methods of dating such as Fluorine dating have been used with considerable success in assigning a rough time frame to the events of burial and fossilization.

A vast body of samples of bones from Early Pleistocene (Upper Siwalik fossil from NW India) to the bones of modern mammals (early Holocene) have been evaluated for the presence of fluorine. The value of fluorine has shown a visible correlation with the dates that have been derived on radiocarbon dating. (Kshirsagar 1993; Sathe 2017)

AMS-^{14}C dating of Manjra Valley Samples and its Bearing on the Chronology of Fossil Sites in Peninsular India

The absence of direct dates on megafaunal fossil material also makes it difficult to assign precise dates for disappearance or extinction of many species especially in the context of Late Quaternary extinction in India (Turvey et al. 2021).

In addition to the "hard water" reservoir effect mentioned above, there is also taphonomic bias in the depositional history of vertebrates, making the mollusks' precise association with fossils difficult and contested. The present work marks the first time that fossilized bones have been dated directly through AMS, and the values are in close congruity with biostratigraphical, skeletal, and morphological parameters. The dates are a definite indication that the period to which the fauna belonged to was closely approaching the Last Glacial Maximum (LGM). Hence, the palaeoecological inferences drawn using isotope chemistry and histology of calcified tissues provide crucial link to the palaeoenvironmental conditions that prevailed in the Manjra valley in the terminal phase of the late Pleistocene (Sathe 2018;

Sathe et al. 2018). The presence of clay bands within the bone bed and lenticular patches of fissured clay (representing pool phenomena) indicates that climatic conditions in the Upper Pleistocene were also approximately the same, with adequate water to recharge both surface and groundwater features (Kulkarni, pers. comm.).

The occurrence of fossilized partial skeletons and isolated bones and teeth of six incisored hippopotami from the multi-species fossil scatter at Harwadi have provided vital information about the vegetational diversity, further attested by the palynomorphs of diverse plant species (Sathe 2015). The date of disappearance for hippopotami in India is known to coincide with the end of the Pleistocene, due to the extended periods of dry and arid conditions resulting in depletion of permanent water bodies during the LGM. This time period also witnessed the extinction of other large mammalian taxa like *Equus namadicus*, *Stegodon insignis ganesa*, *Elephas hysudricus*, and *Palaeoloxodon namadicus* from India.

However, it has also been postulated by Jukar et al. (2019) that the extinction of large megafauna at the end of the Pleistocene was due to anthropogenic factors rather than purely climatic or environmental, based on the dating of a single Hippotamus canine that yielded a date of 16,000–15,000 BP (Jukar et al. 2019).

Except a few examples from the Hunsgi-Baichbal valleys (Sathe and Paddayya 2013), the near total absence of human signatures in terms of cut and chopping marks on the fossilized bones from the Pleistocene fossil assemblages in India questions the role of humans as major factor of extinction of Pleistocene mammals in India.

Given that the date in Jukar et. al. (2019) was obtained on a single specimen and not directly on bone collagen, more dating of *Hexaprotodon* fossils must be carried out from the Indian Subcontinent in order to arrive at a clearer picture of their chronological position, especially with regard to their disappearance or Last Appearance Datum (LAD).

Sample Preparation and Analysis

Bone alteration during burial is the most significant diagenetic process which defines the future survival of the bone in the sedimentary environment. The diagenetic parameters include collagen content, histological integrity, porosity and crystallinity (Hedges 2002). Much collagen loss is correlated with microbial attack in the early stage of diagenesis, which is clearly defined by histological alteration of the fossilized tissue structures (Nielsen-March and Hedges 2000).

Besides the bacterial and fungal modes of attack (where the bone may lose even up to 80% of collagen), in warm climates, temperature sensitive collagen loss is inescapable, where hydrolytic mechanisms have an important bearing upon the loss of collagen. Therefore, it has to be borne in mind that the accuracy of a derived date is controlled by the chemical purification, geological provenance and taxonomic identification of the sample. This is evident in the attempt by Jukar et al. (2019), where the lack of visible collagen forced the authors to date the organic materials potentially found in the phosphates leached from the sample through HCl treatment as directly dating the collagen was not possible. It is due to the inevitable aspects like contamination, degradation and carbon-exchange issues, that fossil bone is invariably considered as one of the most unreliable materials for ^{14}C dating (Hedges and Klinken 1992).

In this context, we employed the treatment methods recommended in Cherkinsky (2009) for dating the bioapatite fraction. A batch of samples was taken from the samples collected

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from the site of Harwadi, Latur District, Maharashtra, comprising a section of elephant (*Elephas* sp.) ivory, a hippopotamus (*Hexaprotodon palaeindicus*) molar, and a section of ischium (pelvic) bone from a large bovine (Table 4).

As per the method described in Cherkinsky (2009), as a first step, each sample was physically or mechanically cleaned as far as possible, removing all of the surrounding matrix and any visible foreign matter. Following this, the samples were soaked in 1N acetic acid overnight and then cleaned by decantation, discarding all the material that was loosened from the fossil samples. The remaining material was then dried for a similar amount of time and again cleaned manually. The sample was then crushed to coarse fragments approximately <1 mm in size. Crushing to a fine powder was avoided to preserve bioapatite structure as stated above. These crushed samples were sent to Inter University Accelerator Centre (IUAC), New Delhi, and further sample processing was done in their graphitization laboratory of AMS facility. 2 g of sample was taken from each fossil specimen and treated with 1 N acetic acid in a 50 mL centrifuge tube. These centrifuge tubes were kept in a vacuum desiccator and samples were evacuated 10 times in 48 hr up to the point when no effervescence was observed. No effervescence was an indication of complete removal of secondary carbonates. Acetic acid was removed from the samples by cleaning with type 1 water (resistivity 18.2 MΩ-cm) and samples were dried in vacuum using a freeze dryer. 300–400 mg of such cleaned samples was taken in a 12 mL vial, flushed with helium gas for 10 min and then acid hydrolyzed using 1N HCl acid in the carbonate handling system (CHS) that is coupled with automated graphitization equipment (AGE). Samples were completely dissolved in 1N HCl acid at room temperature and no collagen chunk was observed in all these three samples. In the hydrolysis, produced CO_2 was trapped in the zeolite trap and graphitized using AGE (Sharma et al. 2019). Bioapatite content (carbon percentage) in these samples were varying from 0.17% to 0.24% as shown in Table 4. This content is less than the range (0.4–0.7%) described by Cherkinsky (2009). According to Cherkinsky (2009), if bioapatite content is more than (0.4–0.7%) indicates that diagenetic carbonates are not removed completely. In our case bioapatite content was less than the above range, implying that samples were cleaned with acetic acid for longer duration and some of the bioapatite fraction may have also reacted with acetic acid during the cleaning. Produced CO_2 in all three samples was sufficient for AMS radiocarbon measurements. ^{14}C AMS measurement was performed in the XCAMS (The compact ^{14}C accelerator mass spectrometer extended for ^{10}Be and ^{26}Al) at IUAC AMS facility (Sharma et al. 2019). Measured results for all the samples were normalized to the standard sample OX II and online delta ^{13}C values were used for the isotopic fractionation corrections. Radiocarbon ages were calibrated using the OxCal 4.4 software with IntCal20 calibration curve (Reimer et al. 2020). Sample ID, pMC values, radiocarbon ages, and corresponding calibrated ages are listed in Table 4. Taking the clue from Cherinsky (2009) and to cross check the results from bioapatite and collagen fractions, a replicate of one sample collected from the ivory specimen from Harwadi was processed for collagen extraction using the protocol by Longin et al. (1971). However, collagen yield in this replicate sample was too low to perform graphitisation and AMS measurements.

DISCUSSION

The present work has yielded AMS dates on vertebrate fossil material ranging between 23,701 to 26,879 calendar years BC, as seen in Table 4.

Table 4 Dates and calibration.

S. No.	Sample name	Sample ID	Weight (mg)	CO_2 produced after acid hydrolysis (μg)	Carbon percentage (%)	AMS Delta ^{13}C values	Percent modern carbon (pMC)	^{14}C age (BP)	Calibrated ages (95.4 % probability)
1.	MNJ bone powder: extinct large bovine	IUACD#19C2580	300	679	0.23	11.59 ± 1.10	6.946 ± 0.072	21423 ± 83	23981–23701 cal BC
2.	MNJ dentine: extinct elephant	IUACD#19C2581	300	726	0.24	3.44 ± 0.98	4.834 ± 0.059	24335 ± 100	26879–26277 cal BC
3.	MNJ dental enamel: extinct hippopotamus	IUACD#19C2582	405	705	0.17	–0.29 ± 1.68	5.992 ± 0.069	22610 ± 93	25271–24506 cal BC

These dates are a crucial step in confirming the Late Pleistocene age of the megafaunal assemblage at Harwadi. The presence of these important environmental faunal proxies like tiger, rhinos, hippos, elephants, and horses as well as several forest dwelling and tropical grassland plant species—at the Terminal phase of MIS 3, in the threshold of LGM—point to a stable population in a rich and diverse biological environment, which is far better preserved at sites like Harwadi compared to the rest of Peninsular India. This renders the Manjra valley assemblage as critically important in understanding the chronology of the megafauna of peninsular India in a primary context.

On another note, the material dated in the present study is co-terminous with dates obtained on molluscan shells from the site of Wangdari (Sathe 1989), which is the only Manjra Valley site to have yielded an isolated tooth of *Stegodon insignis ganesa*. This potentially extends the temporal frame of *Stegodon* in India a few thousand years further than what has been assumed till date, in the range of 30,000 BP (Mongabay 2021). The dates in the present study therefore could represent a new LAD for *Stegodon insignis ganesa* in Peninsular India.

CONCLUSION

The extraction of adequate bioapatite fraction from three samples from the Manjra valley enabled direct dating of fossilized bones, rather than associated materials such as molluscan shells, calcrete or volcanic ash, for the first time in the Indian Subcontinent. The method of sample preparation utilized in the present study has also seen success in the dating of similar collagen-deprived specimens from Brazil (Cherkinsky 2013).

Sathe et al. (2018) demonstrated that isotope analyses on Hippopotamus from the Manjra Valley have shown the continuity of a C4 type of landscape, indicating the abundance of grasslands (which is substantiated by palynological evidence from the site of Harwadi). The dates from the present work provide a definite timeframe for these climatic and vegetative conditions, which have hitherto been conjectural due to the lack of precise dates from the sites.

It is in light of these observations that the AMS dates reported by the authors assume great significance in placing the Terminal Pleistocene in Peninsular India into a definite chronological frame, confirming its proximity to MIS 3.

The new dates on these megaherbivores (including environmentally sensitive taxa such as *Hexaprotodon*) provide a fresh perspective of an ecologically stable landscape during the terminal stages of MIS 3, experiencing dry semi-arid to wet semi-arid phase.

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