

Research Paper

Radiocarbon Dating of Historical Bricks: Exploring the Unprotected Archaeological Mounds in the Environs of Excavated Site of Nalanda

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Nalanda was a renowned Buddhist monastery which is believed to have been active for 800 years from around 4th/5th to 12th centuries AD. We know about the site from the accounts of 7th century Chinese traveler (Xuanzang) and later from 19th century British surveyors (Buchanan, Cunningham and Broadley). There are many studies on this site, recent one among them is a geospatial analysis which has revealed a larger extent (~7.25 sq. km) of the site compared to what is protected at present as World Heritage (WH) inscribed by UNESCO (0.23 sq. km. as core zones and 0.58 sq. km. as buffer zone) and has identified several unprotected archaeological mounds in the vicinity. Since we have a range of period when the site was active, it is possible that the dates of structures in each unexcavated mound are decades or centuries apart. To understand the time sequence in which the site existed one could use absolute dating method. This paper reports result of preliminary dating analysis using the Accelerator Mass Spectrometry (AMS) technique on bricks collected from unprotected mounds in the environs of Nalanda. This paper also reports methods and issues in sample collection, preparation and discusses challenges to overcome in future efforts.

Keywords: Nalanda; Remote Sensing; Historical Bricks; DEM; ¹⁴C AMS; World Heritage; Archaeology

Introduction

Radiocarbon dating is a method for determining the age of an object using the radioactive properties of carbon (¹⁴C) which is derived from the atmosphere and incorporated in the sample. Radiocarbon is an unstable isotope of carbon, which decays to stable ¹⁴N by beta decay with a half-life of 5,730 years. This technique of age estimation of an object by counting beta particles was developed in 1940s by Willard Libby, which subsequently revolutionized archaeological studies as it turned out to be a reliable scientific way of providing quite precise chronology. However, its original application was somewhat limited because (a) complication in recognizing the right kind of sample for analysis. Since only samples containing organic matter can be tested, if one wanted to date an event (like construction of a buildings or establishment of settlement, metal making technology,

style of a sculptures), one had to depend on associated organic material found with these objects and their contemporariness with the event has to be weighed carefully (Bowman, 1990), (b) it requires large amount of sample (Fifield, 1999) and (c) very long measurement time to get enough counts of β particle for a reasonable statistics (Fifield, 1999; Litherland, 1980).

While the problem with (a) of the above still exists the emergence of Accelerator Mass Spectrometry (AMS) in last few decades for radiocarbon dating has widened the kinds of materials that can be carbon-dated as well as has significantly minimized the limitation (b) and (c) as this technique can precisely date even a few mg amounts of carbon-containing (organic/inorganic) materials. The condition is that the carbon should have been derived from the atmosphere where radiocarbon must have been

produced by cosmic ray generated neutrons except in the case of bomb pulse dating. In AMS, ^{14}C atoms are directly counted without waiting for their decay, therefore, the measurement time is significantly reduced. The use of sophisticated ion sources in AMS also dramatically reduces the sample amount needed. Only a few mg amounts of materials are usually sufficient to achieve considerable statistics. Therefore, AMS can date a wide variety of materials and has potential to reduce the limitation (a) also.

A new AMS facility in India has recently been established at Inter-University Accelerator Centre (IUAC), Delhi (Shrama *et al.*, 2018), and it has enabled archaeologists, scientists and researchers to work on radiocarbon dating and improve the methodology for dating archaeological samples. Although some past studies (Bonani *et al.*, 2001) have extracted wood, charcoal or reeds from bricks for dating, in this study we have used the bulk brick materials for dating. This paper reports preliminary investigations of dating archaeological building material such as brick samples, collected from the unexcavated and unexplored region around the protected site of Nalanda.

Study Area

The archaeological site of Nalanda Mahavihara (25.13712 °N, 85.44325 °E) is situated in the state of Bihar in India. Nalanda Mahavihara was a Buddhist residential monastery that was functional from the 4th to 5th century AD until at least the end of the 12th century AD and was much larger than the property currently inscribed as WH by UNESCO (see Fig. 1). Chinese pilgrim Xuanzang visited Nalanda in the 7th century AD and documented what he saw (Beal, 1884). Several British explorers and archaeologists—Buchanan (1812), Markham Kittoe (1847-48), Cunningham (1861-1865) and Broadley (1871)—surveyed and excavated the site in 19th century (Buchanan, 1922; Cunningham, 1871; Broadley 1872). Archaeological Survey of India (ASI) conducted excavation from 1930s to 1980s exposing several large structures (6 temples and 11 monasteries) together with smaller ancillary structures which is within the bounds of the property that is inscribed as core zone by UNESCO as World Heritage (WH) (<https://whc.unesco.org/en/list/1502/>). Figure 1 gives details of the features in the core and buffer zone and of

settlements in the vicinity of Nalanda. The structures in protected site are intensively restored (Asher, 2015), old bricks have been replaced with newer ones (of the same size and shape) and therefore, selecting genuine samples would be a challenge in addition to the regulatory difficulties. The samples for the present study have been taken carefully from unprotected mounds. Since these mounds contain ruins of old structures it is more likely that we can get old brick samples from these.

The larger landscape of Nalanda consists of several mounds, tanks and temples (Fig. 1). While some of these were identified in 19th century; recent studies using remote sensing have identified several more that lie outside the confines of the WH protected area (Rajani and Das, 2018). In order to establish the antiquity of the structures in these mounds, bricks from some of these mounds were collected and dated using the AMS facility at IUAC, New Delhi.

Conventionally bricks are dated using thermo luminescence (TL) or optically stimulated luminescence (OSL) (Sanjurjo-Sánchez, 2016) but since bricks are made of soil that could well have included organic material (if the bricks were quasi baked/fired) of quantity sufficient for AMS, this study has explored the potential of bricks to yield dates and has compared these with dates available from other sources (archaeology and art-history).

Materials and Methods

The geospatial analysis used for identifying archaeological mounds is reported in (Rajani and Das, 2018). The processes involved in the present study include: 1) Field visits to the specific mounds, identification of ancient bricks sample and collection, 2) Radiocarbon dating (using AMS technique) of the samples. We further discuss the dating results, their location and corroborative evidence in the section on results and discussions.

Field Visit and Sample Collection

The present samples may not be best samples for AMS dating of archaeological bricks in general, but these are best samples that we could collect from the vicinity of the excavated site of Nalanda. Within the protected property of the Nalanda site, the structures are heavily conserved for over a century with new bricks of the size and shape imitating original ones

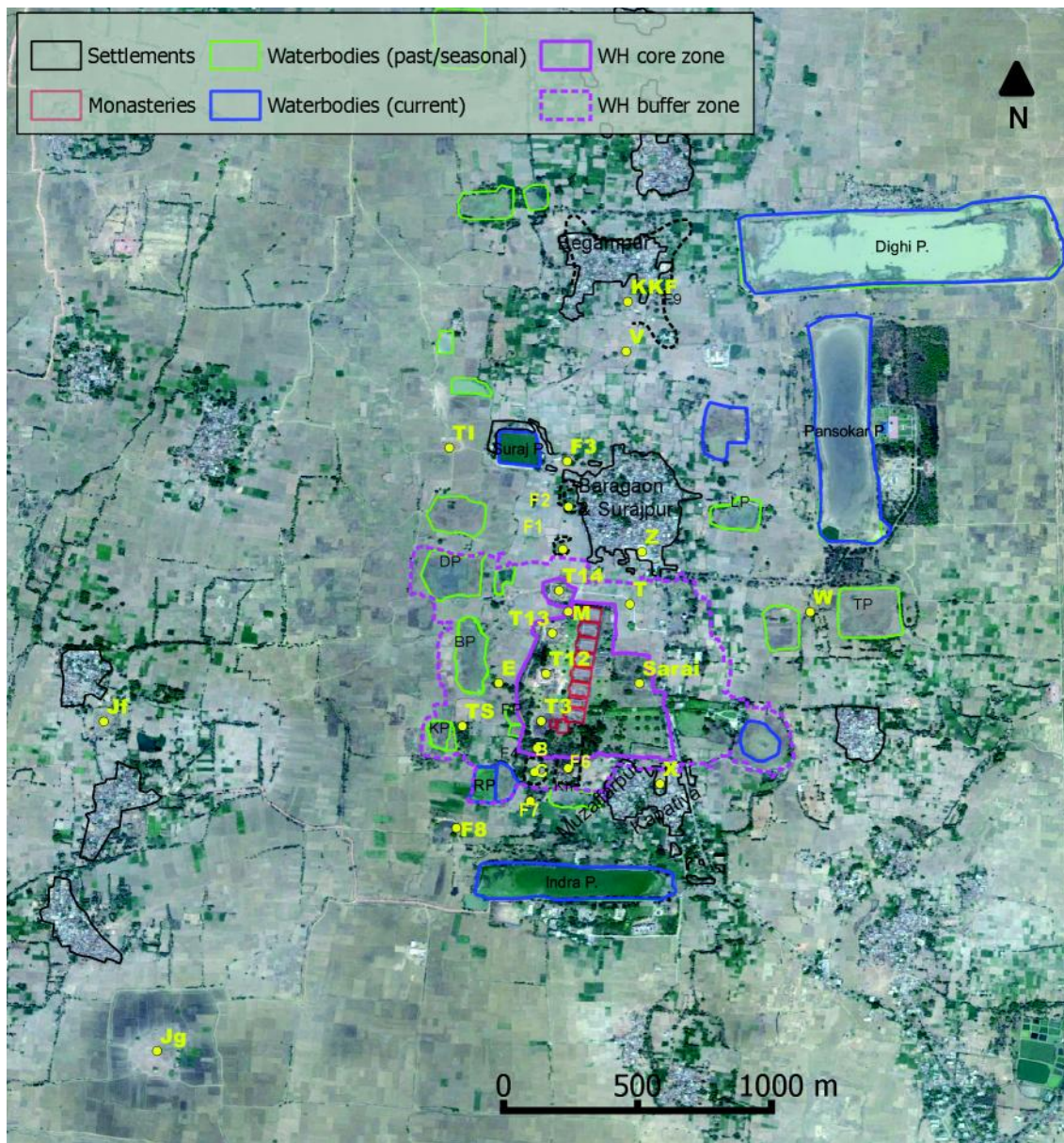


Fig. 1: A view of Nalanda and its vicinity showing features* as recorded. *Refer to Table 1 in Rajani M B and Das S (2018) *Archaeological remains at Nalanda: a spatial comparison of 19th century observations and the protected World Heritage site*. In: *Records, Recoveries, Remnants and Inter-Asian Interconnections* (Ed: Anjana Sharma) pp 247-250, ISEAS Singapore

and these activities have been poorly or inefficiently documented (Asher, 2015) making distinction of an original from a new one almost impossible. Hence, for the site of Nalanda, at this stage of its conservation, collecting samples from the surrounding mounds are the best means. In future if excavation is undertaken in any of these mounds it would then be desirable to collect samples from specific structures during excavation.

A field survey was conducted in December 2016 when we visited unprotected archaeological mounds, collected brick samples and also measured latitude/longitude coordinates of each location from where samples were collected using handheld GPS. We collected 2 samples from same mound so that the dating results of bricks can be compared. The samples we collected were bricks scattered on the mounds (see Fig. 3). These mounds have been identified by analyzing Digital elevation model (DEM)

and old maps made by Cunningham (1861) and Buchanan (1812). This is a preliminary study and we propose the samples collected from potential archaeological mounds to be best samples for 2 reasons:

1. The bricks from the structure (monasteries, temples) in the protected area have undergone extensive restoration. Hence by collecting samples from the mounds we have ensured that the bricks collected are historical.
2. Buchanan and Cunningham have recorded a larger extent of Nalanda and recent remote sensing based studies by Rajani (Rajani, 2016) have shown that the extent of the site is much larger than what is protected today. Hence the samples collected for this study are from potential archaeological mounds and should corroborate to the time period when Nalanda Mahavihara was active.

In some cases, these (bricks) were used by local villagers as weights on dry grass or leaves. Figure 2 has photographs of the samples and the context from where they were collected. Table 1 describes the context of each sample. None of the samples we collected were whole bricks, they were all broken

scattered pieces as we were keen to use non-invasive/non-destructive means (i.e. not disturbing the intact construction or architecture), at the same time took care in distinguishing old bricks from new based on their color, size, weight, etc. Figure 3 shows point locations from where bricks were collected. This helped us to visualize the range; that is the distance from one sample collection point to another and its context vis-a-vis the protected site.

Surface contamination of samples can lead to incorrect results; therefore, it was important to store and transport material carefully. The bricks were kept separately in clean plastic bags. Each bag contained a sheet with all the metadata about the sample (later the unique ID was painted on samples with enamel). These were arranged in carton box and transported to IUAC-AMS facility.

Radiocarbon Dating of Bricks

As a preliminary exercise five samples have been analysed for dating: two from Begampur mound (brick id 51A and 51B), two from Baragaon mound (id 46 and 47) and one from Kapatia mound (id no 39). Figure 3 gives location of all of them in the context of the protected site of Nalanda. Each sample was further divided in two parts, the suffix 'IN' represents the

Table 1: C% for each sample before Pretreatment

S.No.	Sample id	Sample wt. (mg)	C%	Location	Field Context while collection
1.	51B IN	76.21	0.14	Begampur	Brickbat lying on the surface in the field at the location marked as F9 (Rajani, 2016). This plot was being dug for agricultural purpose, when brickbats got hurled out. Following which archaeological authorities dug a trench and found a brick structure. In the process many bricks from the structure buried in the mound are scattered on surface.
2.	51B OUT	40.68	0.86		
3.	51A IN	41.84	0.20	Begampur	Same as above
4.	51A OUT	55.74	1.09		
5.	46 IN	59.52	0.17	Baragaon	In situ brickbat on the exterior of the mound (location marked as F3 in Rajani and Das, 2018) that could be released without force.
6.	46 OUT	70.19	1.37		
7.	47 IN	40.26	0.10	Baragaon	Same as above
8.	47 OUT	41.2	2.64		
9.	39 IN	62.44	0.26	Kapatia	<i>In situ</i> brickbat on the exterior of the mound (among roots of a tree; in location marked as X in Rajani and Das, 2018) that could be released without force.
10.	39 OUT	60.82	1.45		



Fig. 2: Below are photographs of the sample with scale and above are photographs at the collection locations

sample extracted from the center of the brick and suffix 'OUT' represents the sample extracted from outer portion of bricks by scouring the surface.

The methodology of AMS radiocarbon dating has been discussed (Jull, 2013; Jull and Burr, 2013; Sharma *et al.*, 2018). The process has two major steps: 1) pretreatment in which impurities and modern carbon is chemically treated and removed; 2) sample measurement using ion accelerator. Typically, bricks are not preferred for dating as they might not give required amount of atmospheric derived carbon. But for the present study, initially the samples were combusted without any pretreatment to ensure that the carbon percentage is sufficient for AMS dating (see Table 1 for the untreated values). The sample weight in table 1 is the amount taken for determining C%.

Pretreatment

The sample was first powdered and visually inspected for the presence of charcoal, wood etc. but none were found. The samples were transferred in cleaned 15mL centrifuge tubes and the tubes were marked with respective id. 10 ml of 0.5M HCl acid was added ensuring sample is fully dipped in the acid. Manually each tube was shaken and then left on thermo shaker at 60 degrees centigrade at 750 rpm overnight. The

sample tube was kept in centrifuge for 1 minute @ 3000 rpm and then washed with MQ water. This step was repeated till the pH 6 to 7 was attained. All the 10 tubes were wrapped in aluminum foil and kept in freezer for approximately 30 minutes. The samples were then put in freeze dryer (8-10 hours) and after this the samples were ready for graphitization. At the IUAC-AMS facility, Automated Graphitization Equipment (AGE) was utilized for graphitization.

The AGE device automates the two major steps that follow: combustion and graphitization. The basics of combustion and graphitization are discussed below:

Combustion

The sample is combusted at 900°C in the presence of oxygen and in this process carbon dioxide gas is produced. The resulting CO₂ is purified by passing through different columns and finally transferred the glass tube prefilled with small amount of Fe powder (as catalyst).

Graphitization

This step converts the sample CO₂ into graphite (elemental carbon). The purified CO₂ is mixed with a stoichiometric amount of hydrogen gas inside this glass tube. The glass tube is heated at 500 degrees centigrade and in 3 hours. The CO₂ converts to

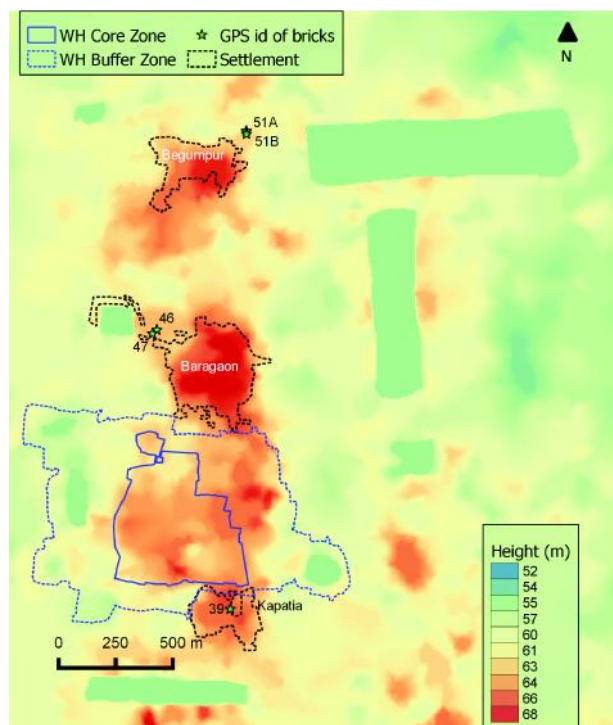


Fig. 3: A map showing topography of Nalanda generated using DEM and location of bricks

graphite. Water is trapped out as a by-product of the reaction. The samples after above processing steps are pressed into a capsule and used for AMS measurements. Further details about equipment, software and procedure has been discussed in detail (Nemec *et al.*, 2010; Wacker *et al.*, 2010).

AMS measurements were performed at the IUAC using a 500 kV Pelletron accelerator system. After the calibration of the AMS system with standard sample, the ratio of $^{14}\text{C}/^{12}\text{C}$ measured in the unknown samples is used to calculate the age of that sample. OX II (NIST standard SRM-4990C), is a standard sample having known $^{14}\text{C}/^{12}\text{C}$ ratio is used for calibration of AMS system. Delta ^{13}C values are corrected to -25 per mil for isotopic normalization. The background value during the measurement was (0.535 ± 0.018) pMC (Percentage modern carbon) and that corresponds to $^{14}\text{C}/^{12}\text{C}$ ratio $(5.3820 \pm 0.1810) \times 10^{-15}$. Data quality is monitored with secondary standard sample (IAEA-C7). Its consensus values (pMC = 49.53 ± 0.12) was matching with its experimental result (pMC = 49.48 ± 0.12) within the error range. The ages calculated from AMS measurement (Libby ages) are converted into calendar ages using OxCal online calculator (Ramsey, 1995).

Results

Table 2 shows the sample name (field id), lab id, pMC value (Percent Modern Carbon), Libby age and Calendar age. Out of ten samples (i.e. five pairs; each pair forms an IN and an OUT samples from same brick), one sample 47IN (IUAC ID: 17C1333) from Baragaon mound was not graphitized due to insufficient carbon content, hence we do not have dates for this sample.

Discussions

Several noteworthy points have emerged from our preliminary analysis. We would like to discuss each sample and their corresponding dates.

- 1) We were able to obtain dates using radiocarbon dating on the bricks using AMS technique. In the past charcoal and organic materials from archaeological and other sites have been used to obtain radiocarbon ages and historical bricks have not been dated using this technique to the best of our knowledge (Vahia *et al.*, 2016; Rajendran *et al.*, 2018; Meenakshi *et al.*, 2018; Raja *et al.*, 2018). Generally bricks are baked at high temperature due to which all the carbon escapes. However, these bricks had organic carbon (refer to Table 1) which indicates that these bricks were not baked upto high temperature. This study shows that with the pretreatment method we adopted it is possible to extract organic carbon in bricks and obtain radiocarbon dates.
- 2) The sample 51A of Begampur has yielded dates of 9th century AD (IN: 886 and OUT: 825). Rajani (2016) has identified this mound using remote sensing and has suggested that it has buried remains of a monastery similar in size to Vikramasila in Bhagalpur, Bihar, and Somapura in Paharpur, Bangladesh (both of which were built during the regnal years of king Dharmapala: c. 780 to 820) and therefore, may have been built around the same time. The AMS dating of this sample is consistent to the above interpretation. However, the other sample from the same site (51B) yielded a date that is a few centuries earlier. The reason of this inconsistency could be that the soil used to make this particular brick might have been collected

Table 2: ¹⁴C measurements of the samples from the site of Nalanda

S.No.	Sample name	Sample ID	pMC value	Libby age* (Median Prob.)	Calendar age probability range (2 sigma-95.4%)	CO ₂ final (microgram)	Wt. of sample (mg)	% carbon	Comments on the date (Calendar age) obtained through this analysis
Begampur Mound									
1.	51B OUT	IUACD#17C1327	97.272± 0.567	222± 46	1521AD-1950 AD(1751AD)	1026	142.72	97.272	Since the date is modern we are not considering this data point.
2.	51A IN	IUACD#17C1328	86.763± 0.773	1140± 71	695AD-1021 AD(886AD)	576	259.8	86.763	Date is consistent with the period when Nalanda was active
3..	51A OUT	IUACD#17C1329	86.152± 0.519	1197± 48	689AD-965 AD(825 AD)	1029	136.47	86.152	Date is consistent with the period when Nalanda was active
4.	51B IN	IUACD#17C1387	82.307± 0.591	1564± 57	385 AD-614 AD(489 AD)	576	259.8	82.307	Date is consistent with the period when Nalanda was active
Baragaon Mound									
5.	46 IN	IUACD#17C1330	71.599± 0.446	2683± 50	967BC-791 BC(848 BC)	415	347.18	71.599	Date is consistent with the period when Nalanda was active
6.	46 OUT	IUACD#17C1331	77.753± 0.475	2021± 49	166 BC-71 AD(27 BC)	1020	129.61	77.753	Date is consistent with the period when Nalanda was active
7.	47 OUT	IUACD#17C1333	92.325± 0.523	641± 45	1280 AD-1401 AD(1346 AD)	978	163.08	92.325	Since the date is modern we are not considering this data point.
Kapatia Mound									
8.	39 IN	IUACD#17C1334	76.151± 0.461	2188± 48	384 BC-112 BC(266 BC)	703	314.19	76.151	Date is consistent with the period when Nalanda was active
9.	39 OUT	IUACD#17C1335	96.737± 0.561	266± 46	1480 AD-1950 AD(1624 AD)	1033	142.28	96.737	Since the date is modern we are not considering this data point.

*Radiocarbon age = -8033 ln (pMC/100)

from different sedimentary layer (strata) or an older brick was reused.

- 3) A pattern can be noticed in the dates yielded by the pairs 39, 46 and 51B: IN dates are earlier than OUT dates. This suggests that the outer part of the samples are contaminated with modern carbon. The contamination of the outer part compared to inner part could be due to water or its exposure to environment.
- 4) The date provided by the 39IN sample of Kapatia mound (266 BC) is earlier than we expected as sculptures found at this mound, based on their stylistic representation, were dated to 9th century AD (Cunningham, 1871). However, archaeological excavation at Temple site no 3 (T3, which is on the southwest corner of protected area and closest to the mound in Kapatia) has material that dates to Maurya period (Mani, 2008). It is possible then that Kapatia mound also had remains from Maurya period (322-180 BC), in which case the dates yielded by sample 39IN would concur.
- 5) The date (848 BC) of sample from Baragaon mound (46IN) is earlier than expected. However, several scholars have suggested that the earth removed from digging tanks in the vicinity of Nalanda were used for making bricks to build the structures of Nalanda monastery (Patil, 1963). In this case, the soil for bricks in some portions of the site could have come from lower sediments and the very early dates yielded by samples from Baragaon mound may be the date of the death of organic material in this soil.
- 6) Since the samples we collected were from ruins buried in mounds and not from exposed structures, the dates obtained from the present analysis cannot be directly attributed to any structure. Ideally one could collect brick sample from an ongoing archaeological excavation where one could be more certain about the original location of samples. By collecting brickbats scattered or embedded in mounds we assume the brickbat belongs to the structure buried in the mound. In reality it may have moved from its original place. Buchanan (1922) and Cunningham (1871) have recorded that the site had become free source of bricks for

constructing buildings and paving road in all the villages in the vicinity (one of the compelling reasons that provided impetus for enactment of The Ancient Monuments Preservation Act in 1904 was to stop local villages to use sites such as Nalanda as a free source of bricks for fresh construction). However, considering the expanse of archaeological landscape and abundance of brick built structures we do not think a specific piece would have moved beyond a mound or maximum the confines of the larger Nalanda landscape. Since the objective of this study was to establish antiquity of the unprotected mounds in the larger landscape of Nalanda, the method we adopted for sample collection is adequate and all the results amply support the premise that the unprotected mounds around the excavated World Heritage site of Nalanda are archaeological in nature.

- 7) We plan to revisit our results after dating more number of samples using stepped combustion techniques as well as fine tune methods of sample collection. Previous studies (J. Mc Geehin *et al.*, 2001, Alvaro Fernandez *et al.*, 2014 and Rosenheim, B. 2013) on the use of stepped or ramped combustion technique for soil samples has shown better results and we also plan to try this technique to refine our findings.
- 8) The mean age of all the accepted results (refer Table 2 and Fig. 4) is 1814 ± 179 which translates to calendar age between 204 BC to 602 AD. This range of calendar age can be accepted assuming that the bricks would be younger than the date of samples (204 BC to 602 AD). It

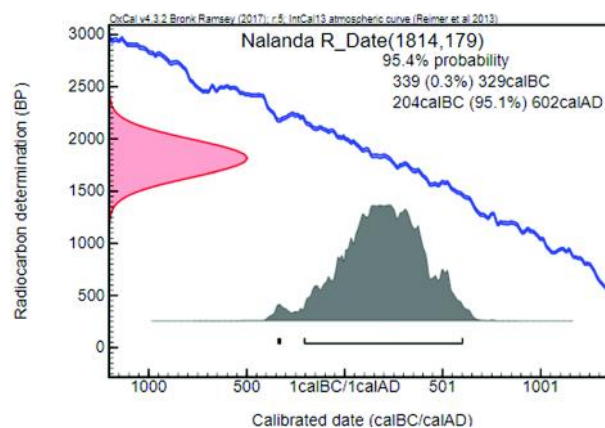


Fig. 4 : The mean of calibrated date for the accepted samples

thus establishes our point that the unprotected environs of Nalanda are archaeological.

Conclusions

Nalanda was inscribed as World Heritage by UNESCO in 2016 and is protected by ASI. Earlier research on the site Nalanda (from 19th century onward) has enriched our knowledge about the physical remains and sizes of structures of the excavated portion of the site. More recently remote sensing analysis has shown the site had a much larger expanse. The present study has added value by attributing possible dates to the archaeological mounds identified by geospatial techniques by dating bricks from these mounds using AMS technique and therefore has contributed to the continuing study of the site.

This study is based on dating of bricks collected from unprotected archaeological mounds in the vicinity of the excavated World Heritage site of Nalanda using AMS technique. The AMS dates may not correspond to the known dates of structures within protected site of Nalanda as these samples were collected from the unprotected area. The present study has produced two important results: 1) Ancient bricks from sites such as Nalanda can be dated using AMS technique; and 2). The unprotected mounds in the larger landscape of Nalanda (Begampur, Baragaon and Kapatia) are archaeologically very potential.

This kind of study can be further pursued on the site itself by trying to collect better samples. For example, studies in Rome, Italy and Sweden have shown that mortar can be dated (Hale *et al.*, 2003; Lindroos, 2018). We also believe that stepped combustion to isolate different fractions of organic and inorganic material will assist in the interpretation of future dating studies. If and when these mounds are excavated one could collect such binding material from these buildings and also date them. Considering the abundance of brick built archaeological structures in India and elsewhere, this study can be a first step towards potentially establishing dates for many structures.

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