Baseline Bioavailable Strontium Isotope Values for the Investigation of Residential Mobility and Resource Acquisition Strategies in Prehistoric Cambodia.

Authors: Shewan, L. G[1]., Armstrong, R. A.,[2] and O'Reilly, D.[3]

1. School of Earth Sciences, University of Melbourne, Victoria, Australia.
2. Research School of Earth Sciences, the Australian National University, Canberra, Australia.
3. School of Archaeology and Anthropology, the Australian National University, Canberra, Australia.

KEYWORDS: Cambodia; Strontium isotopes; Baseline map; Paleomobility

ABSTRACT

Strontium isotope ratios ($^{87}Sr/^{86}Sr$) measured in human skeletal material can increase our understanding of the residential behaviour and resource acquisition strategies of past populations. We have mapped bioavailable $^{87}Sr/^{86}Sr$ variation in 183 plant and soil samples across Cambodia. Bioavailable $^{87}Sr/^{86}Sr$, as measured in plants, differs significantly between four major geological units. This dataset will support future investigations of skeletal material from Cambodian archaeological sites. Baseline $^{87}Sr/^{86}Sr$ data should be applied judiciously to skeletal populations, and in concert with other lines of evidence, to identify potential geographic outliers rather than to ascribe specific locations from which individuals may have moved.

INTRODUCTION

Cambodia has long held archaeological interest (Corre 1879; Mansuy 1902; Mourer 1994) where the primary focus has been on the rise and fall of the Khmer Empire centered on Angkor

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(Fletcher *et al*. 2006). Much of what is known about the Angkorian civilisation is derived from the magnificent religious monuments, artworks, and inscriptions and, in recent years, from the exquisitely detailed record of landscape modification obtained from high-resolution airborne laser scanning technology (Evans 2016; Evans and Fletcher 2015; O'Reilly, Evans and Shewan 2017). In the past 20 years, more research has been undertaken at archaeological sites that predate the foundation of the Angkorian Empire (C.E 802) and specifically about the populations who lived in these pre-state societies (Albrecht *et al*. 2000; Dega 2001; Ly 1999; O'Reilly and Shewan 2015, 2016 a, b; O'Reilly *et al.* 2008, in press; Phon 2004; Pottier *et al.* 2004; Reinecke *et al* 2009; Stark 1998, 2004, 2006; Yasuda 2013). The majority of the excavated prehistoric sites in Cambodia date to the Iron Age (c. 500 B.C.E. - 500 C.E.). This is a period of transformational change, characterised by increasing socio-political complexity, burgeoning inter-regional trade, technological transfer and developments in settlement use (Carter 2010; O'Reilly, Evans and Shewan 2017; O'Reilly and Shewan 2015, 2016 a, b). In the absence of epigraphic evidence for this period, our knowledge of pre-state social organisation, health, diet, material culture and mercantile activity is gleaned from residential and mortuary contexts including human and faunal skeletal assemblages and accompanying grave goods.

The measurement of strontium isotope ratios ($^{87}Sr/^{86}Sr$) ratios in human tooth enamel from skeletons interred in Iron Age mortuary contexts will be used to enhance our understanding of human settlement behaviour and resource acquisition strategies in this dynamic period prior to the rise of the Angkorian Empire. However, in isolation these isotopic measurements are of limited value unless baseline biologically available strontium variation is characterised throughout the study region; around the archaeological sites and surrounding areas. Here we present the first baseline map of biologically available strontium isotope ratios from selected regions in Cambodia. We have also compared the sample values between four major geological units (Jurassic-Cretaceous sandstone, basalt, young alluvium and old alluvium) in our sampled regions. The database is intended as a preliminary assessment of the spatial variation in bioavailable $^{87}Sr/^{86}Sr$ and will be updated as further sampling is completed and with the addition of archaeological faunal enamel from archaeological contexts. For the assessment of individual archaeological sites and human skeletal populations recovered from these contexts, it is advisable to use multiple lines of data. This includes, where feasible, higher resolution $^{87}Sr/^{86}Sr$ environmental sampling, archaeological faunal enamel from the same region, the use of other isotopic systems, and consideration of the material culture assemblage associated with the burials, to facilitate robust archaeological interpretation and understanding.

STRONTIUM ISOTOPES

Strontium has four naturally occurring isotopes (^{84}Sr, ^{86}Sr, ^{87}Sr, ^{88}Sr). ^{87}Sr is radiogenic produced by the radioactive decay of ^{87}Rb which has a half-life of 48,000,000 years (Faure and Messing 2005). Strontium isotope ratios measured in rock, soil, and groundwater vary according to the age and composition of the underlying geology of the region. In general, for rocks that are old (>100 mya) with high Rb/Sr ratios such as old granites, the isotopic ratio is high (>0.710). In geologically young rocks (1-10 mya) with low Rb/Sr ratios, the $^{87}Sr/^{86}Sr$ is low (<0.706). As a high-mass element this variability is carried with no measurable fractionation from weathered bedrock to vegetation and via diet to the teeth and bones (biological apatite) of animals and humans where strontium substitutes for calcium (Bentley 2006).

Strontium isotope ratios have been used in a wide variety of archaeological, palaeontological, ecological and modern source-tracing investigations (Bataille, and Bowen 2012; Beard and Johnson 2000; Bentley 2004, 2006; Bentley *et al*. 2005, 2007, 2009; Budd *et al*. 2004; Evans et al 2012; Hoddell *et al.* 2004; Hoppe *et al.* 1999; Montgomery *et al*. 2000; Price *et al.* 1994a, b; Sealy *et al*. 1995; Shewan 2004: Sillen *et al.* 1998; Slovak and Paytan, 2011; Willmes *et al.* 2018 among numerous others). In bioarchaeological research, the methodology has become increasingly utilised to identify non-local individuals in (pre)/historic mortuary populations and to investigate residential mobility and migration patterns.

The interpretation of isotopic values measured in human skeletal material requires the assessment of regional strontium isotope variability in the study region. While geological maps are a useful starting point to infer isotopic strontium variability in the study region, they reflect bedrock geology and not $^{87}Sr/^{86}Sr$ ratios available to living organisms. Biologically available $^{87}Sr/^{86}Sr$ may deviate significantly from whole rock ratios due to differential weathering of the source material, mixing processes, atmospheric deposition such as dust, rainfall and sea spray, and the use of fertilisers with potentially variable strontium concentrations and ratios (Poszwa *et al*. 2004; Sillen *et al*. 1998; Willmes *et al*. 2018). To gauge local biologically available strontium, $^{87}Sr/^{86}Sr$ ratios have variously been measured in plants, soils, soil leachates, water and faunal teeth from archaeological deposits (Maurer *et al.* 2012; Evans and Tatham 2004; Kootker *et al*. 2016). Ideally, all sample types should be included but this is not always feasible.

Here we provide the first baseline map of biologically available strontium from selected locations in Cambodia (Fig. 1.) based on plant and soil specimens sampled from areas near known Iron Age sites and other regions. restricted to areas accessible by road. Ongoing collection and analysis will extend sample coverage (particularly to the east) and will permit future updates of the database. The baseline map is intended as a resource for future bioarchaeological and zooarchaeological research and investigations concerned with material culture provenance.

THE GEOLOGIC SETTING

Cambodia covers an area of approximately 181,000 square kilometres and is divided into 25 provinces (including Phnom Penh). Topographically, much of Cambodia consists of a lowland plain with the majority of the country at an elevation less than 100m. The plain is framed by the Dangrek mountains to the northwest (highest peak 753m), the Damrei (Elephant) Mountains to the south (highest peak 1,081m), the Krâvanh (Cardamom) mountains to the southwest (highest peak 1,813 m) and a high plateau in the northeast reaching heights over 1,500m.

The dominant water features include the Mekong River and its tributaries and the Tonle Sap lake and river system. The Mekong, flows southward from the Tibetan Plateau through mountain gorges and valleys from China, Myanmar, Thailand, Laos and through the lowlands of Cambodia and Vietnam, debouching in the Mekong Delta into the South China Sea. The river drains a total of approximately 800,000 km^2 with with a long-term average sediment flux estimated to be about 144 ± 34 million tonnes per year (Kondolf *et al.* 2018). The Mekong joins with the Tonle Sap River near Phnom Penh and during the rainy season (May-September), excess sediment-laden water in the Mekong drains into the Tonle Sap (Great Lake) by reverse flow, flooding the alluvial plains surrounding the lake (Penny 2006). With the dry season, the water of the Tonle Sap flows back down the Tonle Sap River into the Mekong River.

While geological research in Cambodia dates from the end of the 19th century, geological information about Cambodia has steadily increased since the establishment of the Cambodian Department of Geology and Mines in 1985 (Sotham 1997). The stratigraphy of Cambodia is summarised in the United Nations, Economic and Social Commission for Asia and the Pacific, Atlas of Mineral Resources of the ESCAP Region (United Nations 1993) and other reviews

(Sotham 1997; Workman 1997). The geology is diverse and comprises sedimentary formations, metamorphic, and igneous rocks from the Precambrian through to the Quaternary (Sotham 1997) (Fig. 2). While there are no dated Precambrian rocks in Cambodia, Precambrian-Proterozoic metamorphic units including gneiss, amphibolites and schist can be found in the northeast of the country and near the western border near Pailin. Cambrian argillites are recorded in Stung Treng Province dated by a Trilobite specimen. Cambrian to Ordovician micaceous quartzite outcrops can be found in the southwest with Cambro-Silurian age schist and quartzite units found in the northeast and near the Laos-Cambodian border. Devonian to Carboniferous sandstone and shale can be found in the east, west and southwest of Cambodia and near Kratie with Upper Caboniferous-Permian limestone represented in Stung Treng Province and the Kampot area. The marine Permian limestone is recorded in Banteay Meanchey and Battambang in the west and Kampot in the south. Mesozoic deposits cover a large part of the region. Triassic rocks are common and include shale, sandstone, breccia, conglomerate, limestone and some volcanic deposits such as andesite, rhyolite and dacite. The early Triassic is considered to be more marine in character with continental phases more common in the late Triassic. In western Cambodia Triassic units have been found in Pailin, Pursat and Kompong Speu and in the east in Preah Vihear and Kratie.

The main characteristic unit of the Lower-Middle Jurassic is the *Terrain Rouge* comprising conglomerates, sandstones and siltstones of continental origin occupying large areas in eastern

Cambodia and extending into north central Cambodia with isolated outcrops in the west. This series can exceed 2000m in thickness. The Upper Jurassic-Cretaceous includes the *Gres Superieures* (upper sandstone) sequence represented in the southwestern Cardamom Mountain region and northern Cambodia including Phnom Kulen and the Dangrek Range, with isolated outcrops in Kratie and Stung Treng. This series consists mostly of quartz rich sandstone and conglomerates which can exceed 2000m in thickness. Volcano-sedimentary rocks of Jurassic age, comprising rhyolites, dacites and tuffs are represented in western Cambodia in Pursat.

The central plains are mostly covered by alluvial deposits of Quaternary to Recent age with protruding hard rocks. Neogene-Quaternary sediments are referred to as Old Alluvium. These sediments, varying in thickness, from thin to more than 8m, are composed mostly of claystone, silt and sand, overlying lateritized or conglomerate beds. Young Alluvium Quaternary sediments, generally occupying lowland regions of the central alluvial plain, 0-40m above sea level, are widespread and contain grits, pebbles, sand, silt and claystone. This alluvium can reach over 200m in thickness in some areas.

Intrusive suites of Paleozoic to Paleogene age are represented in the northeast, Preah Vihear, Kompong Chnang, Pursat, and in the south near Kampot. Volcanic rocks include Neogene-Quaternary basalts (occurring mostly in the east but also near Pailin, Preah Vihear and the Cardamom Mountains) and Permian-Mesozoic rhyolites, dacites and andesites.

METHODOLOGY

Strontium isotope ratios

Plant and soil samples were collected from a range of geologic provinces across Cambodia. Plant specimens consisted of shallow rooted grasses and were taken a few cm away from soil samples which were collected at a depth of between 0 and 5cm. Sample sites were concentrated in the western part of the country, in the regions where archaeological research is being conducted by the authors (Fig. 1.). We also sampled in other areas accessible by road. Access to eastern Cambodia was limited at the time of collection. Further sampling and analysis will become possible as other regions are explored. Sample sites were geo-located with a hand-held GPS device, samples dried and transported to Australia for analysis. Sample sites were classified into four categories including Jurassic-Cretaceous sandstone (n=12), basalt (n=4), young alluvium (n=38) and old alluvium (n=20), based on their location compared to geological maps (United Nations 1993). In addition, a single sample came from an area of exposed granite.

Plant and soil samples were placed in porcelain crucibles and ashed for 8 hours at 800C. 10-20 milligrams from each plant sample was placed in an acid cleaned Teflon beaker and digested in ultrapure concentrated nitric acid (HNO_3), and for the soils the addition of hydrofluoric acid (HF). Sr was separated and concentrated using Sr-Spec ion exchange columns. For soil leachates 1gm of soil was placed in a centrifuge tube with 1 ml ammonium nitrate (NH_4NO_3) shaking the suspension overnight to extract the bioavailable strontium component. Following digestion, Sr was separated and concentrated using Sr-Spec ion exchange columns. $^{87}Sr/^{86}Sr$ was measured using Thermal ionization mass spectrometry on two mass spectrometers over a period of several years at the Research School of Earth Sciences at the Australian National University. The earliest samples were analysed using a Finnigan MAT261 (with the samples loaded onto Ta filaments) and the later samples using a Thermo Finnigan Triton mass spectrometer (with the samples loaded with TaF onto Re filaments). All analyses were corrected for mass fractionation using $^{86}Sr/^{88}Sr = 0.1194$ and the $^{87}Sr/^{86}Sr$ ratios were monitored by frequent analyses of the NBS standard SRM987. The average values over the relevant periods for the two mass spectrometers were 0.71023 ± 0.00003 (2 SD; n= 30) for the Mat261 and 71025 ± 0.00002 (2 SD; n= 74) for the Triton. All values are reported normalised to a value 0.71023. Samples were compared using paired and unpaired Student t-tests as appropriate.

RESULTS

Plant and soil samples were analysed from 76 locations as shown in Fig. 2. and Table 1). As has been demonstrated in various studies in other regions, whole rock and soil strontium values do not necessarily correlate with bioavailable strontium (Evans and Tatham 2004; Price *et al*. 2002; Sillen *et al.* 1998). We have confirmed this finding in a monsoonal environment by measuring $^{87}Sr/^{86}Sr$ in bulk soils and comparing these results to $^{87}Sr/^{86}Sr$ values obtained from leached soils and plants. In the 45 locations where we had all 3 sample types (soil, leachate, grass) we performed a 2 tailed Student t-test for paired samples. This showed soil samples to be highly significantly different to plant samples (soil=0.71341 ± 0.00634 compared to plants=0.71062 ± 0.00310, p=0.0005). Analyses of soil leachates were intermediate, at 0.71172 ± 0.00391 between plants (0.71062 ± 0.00310, p=0.042) and soil (0.71341 ± 0.00634, p=0.02). These observed differences between leachates and plants are consistent with previous reports from other regions (Maurer *et al*. 2012; Willmes *et al*. 2018).

Plant samples

The range of $^{87}Sr/^{86}Sr$ ratios for plants across the study region varied from 0.7037–0.7198 (Fig. 2.). Generally, the most radiogenic $^{87}Sr/^{86}Sr$ ratios were measured in grasses growing on areas of Jurassic-Cretaceous sandstone and granite, while the least radiogenic values were recorded in plants growing on basalt (Fig 2-5). The highest average $^{87}Sr/^{86}Sr$ values were recorded in plant samples from Jurassic-Cretaceous sandstone (mean value ± SD 0.7131 ± 0.002) which was significantly higher than both basalt (0.7054 ± 0.002, p<0.0001) and Old Alluvium (0.7094 ± 0.002, p<0.0001), but not significantly different to Young Alluvium (0.7116 ± 0.003, p=0.14, ns). Plant samples from basalt areas produced the lowest $^{87}Sr/^{86}Sr$ values and significantly different from the three other geological units (sandstone p<0.0001; Young Alluvium p=0.0003 and Old Alluvium p=0.002). In addition, although considerable $^{87}Sr/^{86}Sr$ overlap exists between plant samples taken from Young and Old Alluvium, the difference between them was significant (0.7116 ± 0.003 vs. 0.7094 ± 0.002, p=0.004) (Table 2).

DISCUSSION

Cambodia is dominated by the broad central plain surrounding the Tonle Sap Lake and the Mekong River and tributaries, covered with Quaternary sediments with isolated hills of basement rocks. Areas surrounding the plain comprise metamorphic, sedimentary, volcanic and intrusive rocks of different ages and composition (Sotham 1997; Workman 1997). While ideally $^{87}Sr/^{86}Sr$ baseline maps for archaeological provenance studies might usefully compare all possible samples types including whole rock, soil, soil leachates, ground water, flora, archaeological and modern faunal enamel, this is not always practicable. In our study we have used plant samples to approximate bioavailable $^{87}Sr/^{86}Sr$ variability across the region. In areas where we analyzed plants, soils and soil leachates, from the same location, considerable divergence was recorded between plant, soil and soil leachate $^{87}Sr/^{86}Sr$ values, questioning the utility of using soil leachates in this region to approximate bioavailable $^{87}Sr/^{86}Sr$.

Our study has identified that plant $^{87}Sr/^{86}Sr$ ratios generally reflect the surface geology of the region and can discriminate between different lithologies. Furthermore, we find sufficient variation in bioavailable $^{87}Sr/^{86}Sr$ within the region to investigate the residential mobility of past populations using strontium isotope analysis of skeletal material. Plants growing on Jurassic-Cretaceous sedimentary units in areas such as the Phnom Kulen plateau, the Dangrek ranges and the Cardamom mountains ranged in $^{87}Sr/^{86}Sr$ value from 0.7084 to 0.7156. Lying south of the

Dangrek mountains and approximately 30 km north of Angkor, the Kulen region of north central Cambodia was an important quarry source during the Angkorian period (Carò, Douglas and Im 2010; Carò and Im 2012). Plants sampled from areas of basalt including locations in the Cardamom Mountains (southwest Cambodia) and north and central Cambodia display lower $^{87}Sr/^{86}Sr$ values; 0.7047, 0.7037 and 0.7044, respectively. A value of 0.7087 measured in a single plant specimen from the basaltic area in Mondolkiri (eastern Cambodia) presents a higher $^{87}Sr/^{86}Sr$ value. This may simply reflect a different rock composition (not measured) or may include strontium from other sources, including quaternary laterite deposits found in close proximity (Piilonen *et al.* 2018) or the presence of weathered soils modified by atmospheric input (precipitation and dust).

Plant samples growing on Old and Young alluvium displayed the widest range of $^{87}Sr/^{86}Sr$ values as would be expected given the range of source components in the alluvium and fluctuation in river discharge, sedimentation rates and river course throughout time (Fig. 6). $^{87}Sr/^{86}Sr$ measured in plants growing on Young Alluvium (sand, silt, clay and some gravel) ranged from 0.7064 to 0.7198 and generally increased in value from north to south, though no definitive gradient is discernible and outliers exist. Plants growing on Old Alluvium (sand, silt, clay, laterite and gravel) ranged from 0.7055 to 0.7133 reflecting different sources of strontium in the alluvial composition.

In areas of specific archaeological interest, where human skeletal samples will be analyzed to examine residential behaviour and landscape utilisation, the $^{87}Sr/^{86}Sr$ baseline plant data will be supplemented with the analysis of archaeological faunal enamel. Using animals that fed locally, and obtained from the same area and stratigraphic contexts as the human burial assemblage will help to refine local catchment $^{87}Sr/^{86}Sr$ variability. $^{87}Sr/^{86}Sr$ measured in the enamel of individuals interred at the investigated archaeological sites will be compared to the local strontium isotope signature to identify individuals who may be geologic outliers to the region rather than to nominate a specific childhood origin.

Archaeological landscape

Archaeological research has been conducted by the authors at Iron Age (c. 500 B.C.E. - 500 C.E.) sites in Banteay Meanchey at Phum Sophy and Phum Snay (O'Reilly *et al.* 2015; O'Reilly and Pheng 2001; O'Reilly *et al.* 2008), in Siem Reap Province at Lovea and Prei Khmeng (O'Reilly and Shewan 2015; 2016a, b; O'Reilly *et al.* 2017, b, in press; Pottier 2001) and at a historic jar/coffin site named Phnom Pel in Koh Kong Province (Beavan *et al.* 2012) (Fig. 7.). In Banteay Meanchey Province (northwest Cambodia), the Iron Age sites of Phum Sophy and Phum Snay are situated within Young Alluvium (mean 0.7117 ± 0.003). With the aid of this strontium baseline map, in conjunction with strontium isotope values measured in archaeological fauna enamel recovered from the sites, it may be possible to identify potential geologic outliers among the excavated human burials, should individuals have exploited food resources from more radiogenic areas such as Jurassic-Cretaceous regions or less radiogenic basaltic locations.

The Iron Age sites of Lovea and Prei Khmeng are located some 60km southeast of Phum Sophy and Prei Khmeng in Siem Reap Province. While also situated within Young Alluvium, plant samples taken in close proximity to these sites display higher $^{87}Sr/^{86}Sr$ values overlapping with more radiogenic Jurassic-Cretaceous regions to the north. As a result of this confluence, it may be more difficult to discern discrete human utilisation of these different geological areas, as significant mobility within regions of similar isotopic range, or indeed between spatially separated but similar isotopic regions, will not be perceptible. However, exploitation of less radiogenic areas such as regions of basalt and andesite will potentially be detected.

In Koh Kong province, the 15th-17th century jar/coffin site of Phnom Pel (Beavan *et al.* 2012) is located in the Cardamom Mountains, a region composed of Upper Cretaceous sedimentary formations (Grès Supérieurs) with outcrops of fine-grained basalt (unpublished observations). It should be possible to gauge whether this population, whose remains are contained in large ceramic storage jars and wooden log coffins, spent their childhood mostly within the dominant Jurassic-Cretaceous local environment or accessed resources from less radiogenic locales such as the areas of basalt within the Cardamom ranges and regions beyond.

Future research will usefully be directed at more intensive sampling around the archaeological sites of interest to enhance resolution and in areas not yet sampled.

CONCLUSION

In order to assess the residential mobility and resource acquisition strategies of past populations using strontium isotope analysis of human enamel, regional maps of bioavailable $^{87}Sr/^{86}Sr$ are required. Studies in other regions have relied on various types of samples to approximate local and regional $^{87}Sr/^{86}Sr$ variability including plants, soils, soil leachates, water, and faunal remains, against which to compare $^{87}Sr/^{86}Sr$ in skeletal remains. In this study, we have measured $^{87}Sr/^{86}Sr$ values in vegetation samples from across the region in an effort to create a baseline map of bioavailable $^{87}Sr/^{86}Sr$ for Cambodia. This database will assist in the examination and interpretation of skeletal material recovered from archaeological mortuary deposits excavated by the authors and to provide a useful resource for other provenance studies. The results of our study show that biologically available $^{87}Sr/^{86}Sr$ ratios, as measured in plant samples on average reflect the surface geology of the region and can be used to assist in the characterisation of the local $^{87}Sr/^{86}Sr$ signature. To further refine local $^{87}Sr/^{86}Sr$ values for archaeological regions of interest and to examine the mobility and habitat exploitation patterns of the individuals interred at these sites, we will use this baseline database in combination with the analysis of faunal enamel preserved in the same archaeological contexts as the skeletal population. Future research will result in more comprehensive coverage of the region with updates of the map as further regions in Cambodia are explored.

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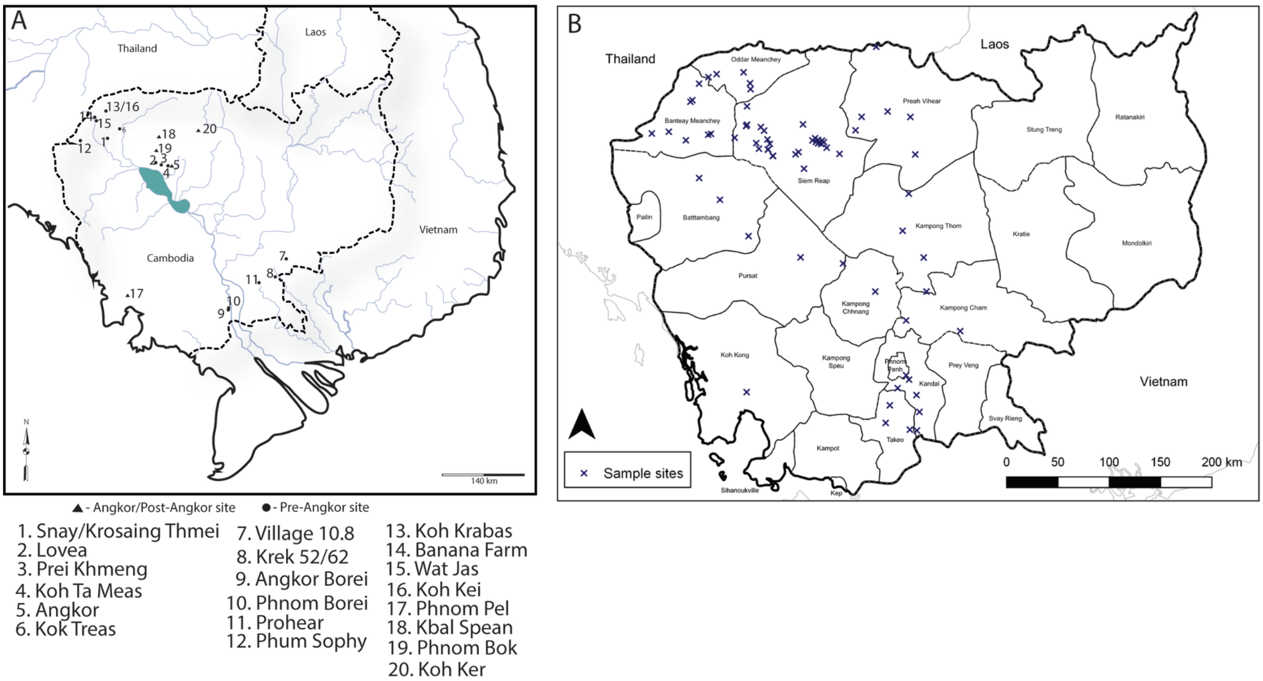

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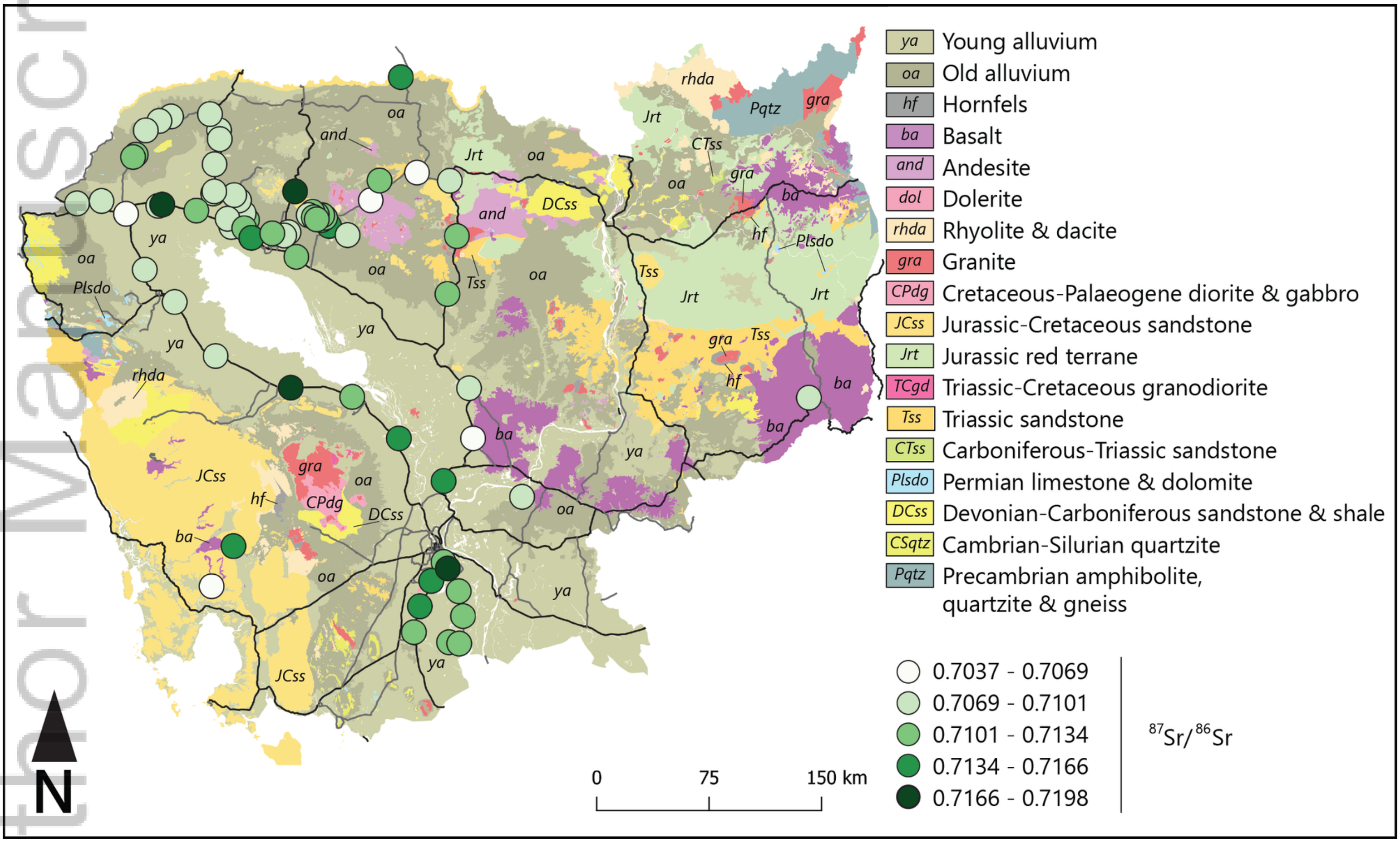

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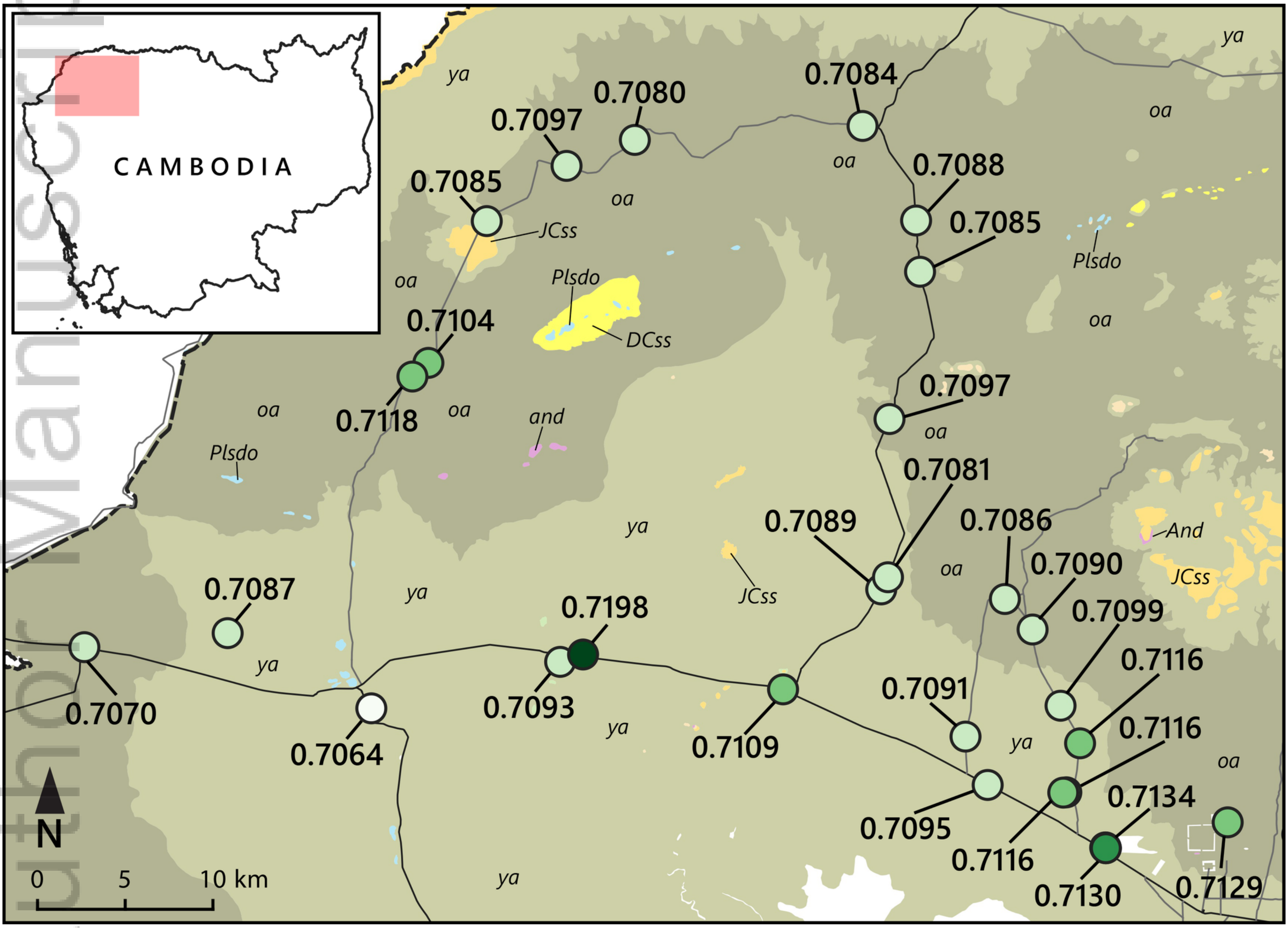

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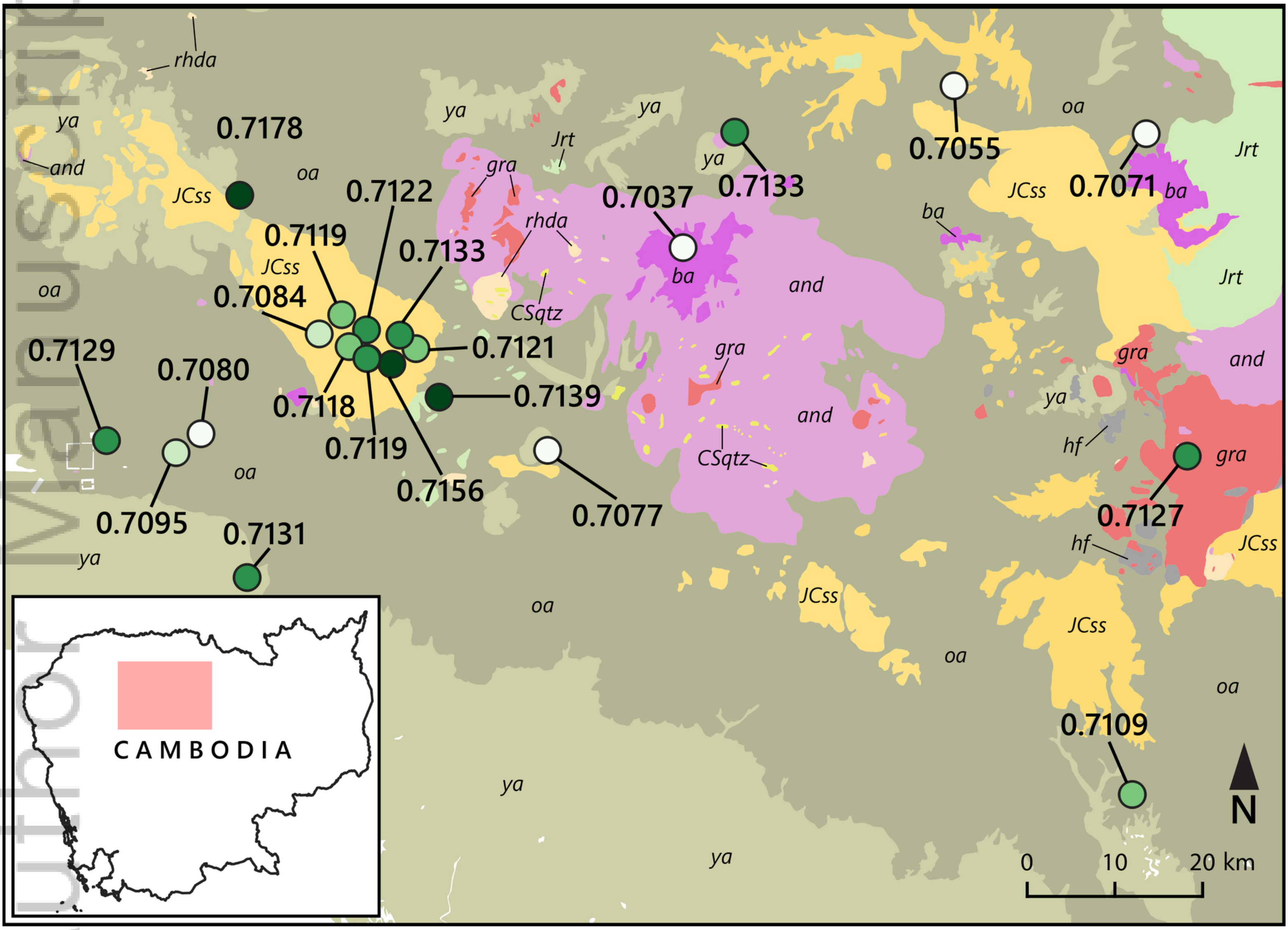

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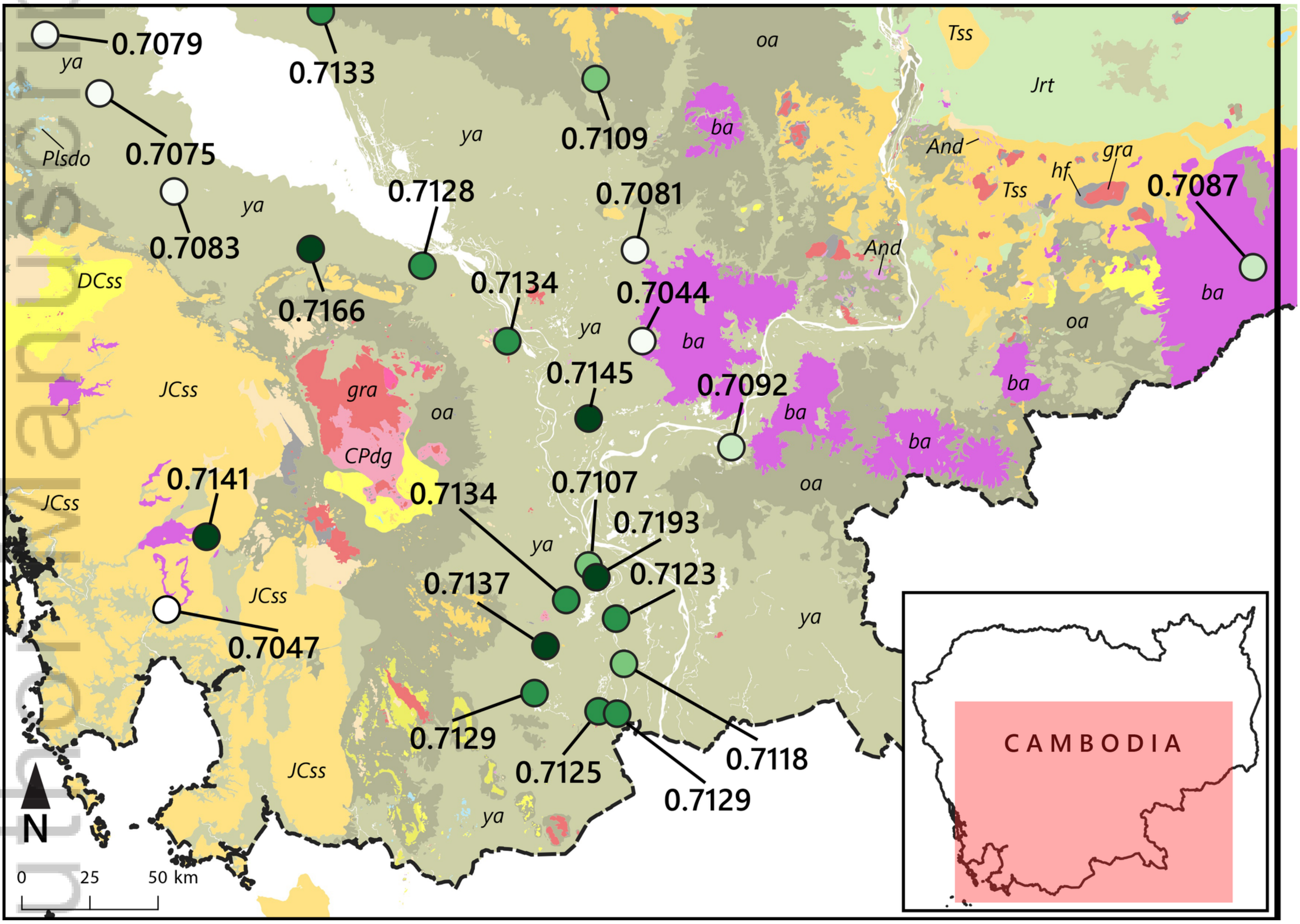

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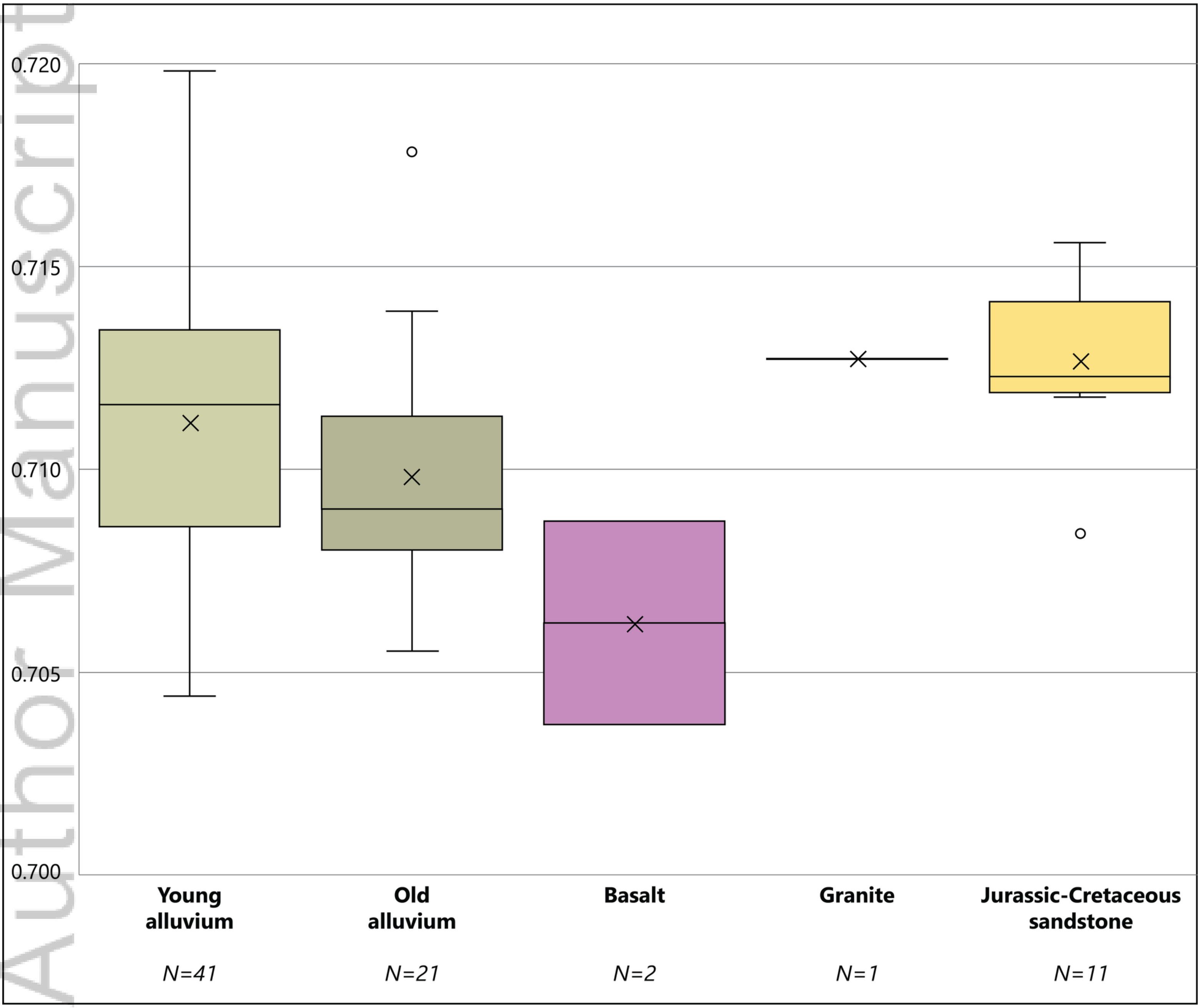

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