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RESEARCH

Tracking ancient glass production in India: elemental and isotopic analysis of raw materials

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Abstract

In India, the organization of the ancient glass industries that produced, starting around the mid-1st millennium BCE, huge quantities of small drawn beads that were traded both locally and all over the Indian Ocean and beyond, is poorly known. Elemental compositions conducted on glass beads found in India and on Indian beads found elsewhere show a great variability that could be linked in some cases to different production regions (Northern, Southern, and Western India). However, a more precise provenance attribution and the identification of regionally distinct production centers was not possible without additional research. Ethno-historical references show that glass was often produced from one single ingredient, called *reh*. We collected raw material samples from selected regions within the subcontinent. This paper reports on both the elemental compositions of these raw material samples obtained using laser ablation—inductively coupled plasma—mass spectrometry and their isotopic compositions including Pb, Sr, and Nd. The results were compared to elemental and isotopic data available for known Indian glass artifacts recovered from sites within and outside India. Our results show that some regions within India are more likely than others to have been the loci of glass production in ancient times.

Keywords India · Glass · Elemental composition · Isotopic composition · Provenance

Introduction

Indian glass beads, often in the form of small (less than 5 mm diameter) monochrome drawn beads, have been discovered across a vast geographical area encompassing Asia, Europe, and Africa. Often referred to as Indo-Pacific beads, the circulation of these Indian glass beads around the Indian Ocean has long been documented (e.g., Tornati and van der Sleen 1960; Davison and Clark 1974; Francis 1990). Recent research, however, is providing new insights about the nature and importance of their diffusion in this region and beyond. For example, over the last 2 decades, glass bead research in Africa has intensified, with multiple studies in southern Africa (e.g., Robertshaw et al. 2010; Wood et al. 2009), coastal eastern Africa (e.g., Siu et al. 2021; García-Heras et al. 2021; Wood et al. 2022, 2017; Sarathi et al. 2022), inland eastern Africa (Walz and Dussubieux 2022; Denbow et al. 2015; Klehm and Dussubieux 2022), northeastern Africa (Then-Obłuska and Wagner 2019a, b; Then-Obłuska and Dussubieux 2021a, b) and western Africa (Brill 1994; Lankton et al. 2017). Indian beads are in burials, middens, or living areas, with some variability in terms of chronology and density. The density of the Indian glass bead presence seems correlated with distance from the Indian Ocean and so is comparatively low in western Africa, moderate in northeastern Africa, and fairly high in eastern and southern Africa. Indian beads are also known from earlier periods in Egypt, mostly 4th–6th c. CE (compared to the other African regions where they appear mostly from the 9–10th c. CE onward). In Europe, recent research has shown that the presence of Indian beads occurs during a relatively short time window corresponding to the Merovingian period (more specifically the mid-5th/6th c. CE) and is attested in burials in France, Belgium, and Switzerland (Poulain et al. 2013; Pion and Gratuze 2016; Gratuze et al. 2021). The beads, rather than being traded as individual ornaments, were clearly sewn

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onto imported garments made from exotic fabrics (Martin Pruvot and Gratuze 2019) as suggested by their location on the body of the deceased. These beads might have reached their destination traveling from the Indian Ocean to the Mediterranean basin via the Red Sea. A single Indian bead, found in Tel Anafa, Israel, dating possibly from the 9th to 13th c. CE (Larson and Dussubieux 2022), could have used the same route, whereas numerous specimens recovered at Kish in Iraq suggest another extension of the Indian bead trade network through the Persian Gulf (Dussubieux 2022). The dating of the beads found in the Middle East is quite uncertain but may more or less correspond to the early to the mid-1st millennium to the mid-2nd millennium CE.

The earliest known occurrence of Indian beads outside India comes from mainland Southeast Asia where they were recovered mostly from burial contexts but also in a few instances from production sites, dating to the mid-1st millennium BCE, in Thailand (Dussubieux and Bellina 2017; 2018). Early glass beads were also found in Vietnam (Tamura et al. 2020; Nguyen Kim Dung 2017; Dussubieux 2001). They are ubiquitous in Southeast Asia during a period covering well over two millennia (Dussubieux 2021). In Southeast Asia, not only were beads imported from India but raw glass as well that was transformed locally into beads (Dussubieux and Bellina 2017, 2018). The Bay of Bengal and the South China Sea trade spheres were certainly two important maritime trade networks involved in the circulation of Indian beads between mainland and island sites. These maritime routes, inland routes, or some combination, might be responsible for the diffusion of Indian beads around the beginning of the common era in China (e.g., Ma et al. 2022), Korea (e.g., Kim and Kim 2013; Brill 1995), and Japan (Katsuhiko and Gupta 2000), where they are also found in abundance.

It seems reasonable to hypothesize that a craft industry that could produce huge quantities of beads found over such a vast geographical and chronological spread may have been the product of a well-organized industry that was in turn well connected to internal and external trade networks. It must also have been an enduring and highly adaptable industry—able to thrive over time despite shifting political and economic circumstances both within India and in the regions where the beads were exported. Although we assume as such, based on the research done on finished glass artifacts outside of India, we have surprisingly scant information from within India to explain how the Indian glass bead industry was organized and how it evolved through time. With this study, our goal is to provide new information about India's early glass industry. First, we identified possible subregions where production may have occurred in the past. Then, to start shaping its broad geographic and chronological framework, we compared the elemental and the isotopic (Sr-Nd-Pb) compositions of raw materials from these subregions with the elemental and isotopic compositions of known Indian glass objects. In this paper, we will discuss preliminary results based on raw materials collected from Andhra Pradesh, Gujarat, Uttar Pradesh, Maharashtra, and Tamil Nadu.

Background: single ingredient glass recipe

The primary reason we can speak of an "Indian" glass is because there exists a type of glass called "mineral soda-high alumina" or "m-Na-Al" glass that is particularly abundant in India and, because of the high concentration of this glass in the subcontinent, was assumed to be of local origin (Brill 1987). This glass has a soda-rich composition ($Na_2O = 15–20\%$) with high alumina concentrations ($> 5\%$) and a low concentration of magnesia ($< 1.5\%$) suggesting a mineral source for the sodic flux. The high soda/high alumina composition of the Indian glass can be explained by the selection of rather immature sand, with compositions very close to that of the granite from which it derives, which contains a relatively high proportion of feldspar. These sands also contain high concentrations of a range of trace elements, including titanium, zirconium, the rare earth elements, and uranium. Generally, high concentrations of sodium in a glass are due to the deliberate addition of soda, found either in mineral form or produced from halophytic plants (growing in soils containing saline water) that are reduced to ashes. But for India, ethnographic data and scientific experiments indicate that glass makers were using a naturally occurring sodic efflorescence variously called *reh, usar, kalar, oos*, etc.—terms designating either the efflorescence itself or the sodic rich soil (Agrawal and Gupta 1968). *Reh* is the most widely known term for this efflorescence and will be used here. *Reh* contains large amounts of sodium salts (carbonate, bi-carbonate, and sulfate) and varying proportions of calcium and magnesium salts, often resulting in soils that are unsuitable to agriculture (e.g., Sharma and Chaudhari 2012). It occurs in areas where rivers draining from mountains contain dissolved salts that percolate through the subsoil until saturation. Rains dissolve these salts, which during the dry season then travel upward through the soil by capillary action, forming a white efflorescence on the surface (Wadia 1975:489, 501, 502). These efflorescences are present in arid or semi-arid regions and can be exacerbated by poor irrigation methods and/or poor draining, which in turn accelerate waterlogging and salt accumulation in the soil.

In India, published accounts mentioning the use of *reh* or sodic soils as the only ingredient necessary to melt glass are plentiful, particularly in British colonial records. Edward Balfour (1871:331) indicates "that wherever *reh* occurs over clean sandy soil, there is naturally formed a mixture of sand and alkali, which fuses into coarse lumps of bottle-green

glass." Elsewhere, he describes how glass is made in the Behar district: "The efflorescence of the soil (…) is collected and thrown in a cistern lined with clay. This is then filled with water, which is afterward allowed to evaporate. When dry the bottom of the cistern is found covered with a thick saline crust, (…). This soda makes glass without any addition as it still contains a sufficient portion of siliceous matter." The administration report, No. 480G of 1882, Department of Agriculture and Commerce, the Northwest Province, and Oudh describes "the native fashion" to produce glass by loading a closed furnace with *reh* soil in the Aligarh and Etah districts (Uttar Pradesh). After 8 days, a colored glass full of bubbles and impurity is obtained. The Journal of the Society of Arts (1900, Volume 48, p. 584) includes reports from A. Rogers about glass manufactured in Gujarat: "Glass is already made at Kapparvanj, in the Thásra Talúka, in Kaira, from a surface efflorescence of carbonate of soda and the silica which it is mixed, but as the materials are crude and impure, the glass produced is naturally very coarse and bad. It is used mostly for women's bangles and rough glass and bottles…." Abraham (2016) reports similar practices from British records about southern Andhra Pradesh where "the soil mixed with the soda is found to supply the necessary amount of quartz…" for the manufacture of glass (Cox and Stuart 1894–1895:165). This traditional method of manufacturing glass was in fact still in practice until a few decades ago. Kock and Sode (1995) describe how, until very recently, the beadmakers in the village of Purdalpur (close to Agra in Uttar Pradesh) "just had to dig" a sandy ground with a high natural sodium carbonate content "to get their raw material." In the same area, Gill (2017:254) reports that *reh* was "gathered from the surface upon its natural efflorescence." The same raw material was used by the glass workshops in Firozabad, located a few kilometers south of Purdalpur (Kock and Sode 1995; Brill 2003). As an experiment, Brill (1999) placed *reh* from a nearby field (approximately 12 km southeast of Shikohabad, a town in Uttar Pradesh close to Firozabad) in a furnace at 1100 °C for 2 h. He obtained a glass with 35.7% of soda (Na_2O), 9.9% of alumina (Al_2O_3), 1.7% of lime (CaO), 1.3% of magnesia (MgO), and 2.28% of potash (K_2O). Although the soda content was a little high compared to the concentration found in glass in general, the composition of this glass is fairly close to the compositions of the ancient m-Na-Al glass artifacts associated with South Asia.

Materials and methods

Collecting raw materials

We selected a limited number of regions in India from which to sample raw materials based on two factors: the known presence of *reh* and evidence of glass-making in the recent and/or more distant past. We assumed that any glassmaking workshop would be located near a *reh* source. We focused our research on seven distinct areas in India based on the amount of time and funding at our disposal, but we acknowledge that additional areas could be explored and should certainly be investigated in the future. The seven areas that form the basis of this project are eastern Tamil Nadu, Puducherry, and southeast Andhra Pradesh in south India; western, northeastern, and southeastern Uttar Pradesh in north India; and Gujarat and western-central Maharashtra in western India (Fig. 1). We tried to identify possible *reh* sources by seeking out non-cultivated plots of land with whitish coloring and little vegetation, and of course, we relied heavily on the knowledge of local residents. GPS coordinates were recorded at each sampling location and approximately 50 to 200 g of raw material were collected from the surface (Fig. 2). Although our main focus was *reh*, we also collected other sands in an effort to understand isotopic variability within a given region. Here, one hundred sixty-six samples will be discussed; more details about them are available in Table 1 of the Supplementary Materials. Below is further information about each region and its connection to glass.

South India

Two key locales in south India include the archaeological site of Arikamedu and the contemporary village of Papanaidupet, both of which have played a key role in determining the origins and manufacture of India's small monochrome drawn beads. In southern Andhra Pradesh, the village of Papanaidupet still manufactures drawn beads (Francis 1991; Kanungo 2016). Francis documented the glass waste from various stages of production from Papanaidupet (Francis 2002: 22) and compared the debris to glass wastes from the site of Arikamedu, an Early Historic port site on the Tamil Nadu coast (Puducherry district). Not only did Arikamedu have Indo-Pacific beads in layers dating to the first centuries BCE, but the glass wasters from those layers were virtually identical to those still being made in modern Papanaidupet village. Similar material was also found at the nearby site of Karaikadu (Francis 2002:27). In the vicinity of Arikamedu and Karaikadu, further evidence for premodern glass working was recently discovered from other sites including Manikollai, (Lahiri et al. 2016, Rajan 2011:186, Yathees Kumar 2011:198), Thiruchapuram and Ariyur (Yathees Kumar, 11: 198). At Manikollai, both raw glass and glass beads were reported, suggesting a bead-making site (Gratuze and Sarah 2012:133). Although Karaikadu (Dussubieux 2001) and Manikollai (Gratuze and Sarah 2012; Lankton et al. 2014) yielded m-Na-Al glass, the glass

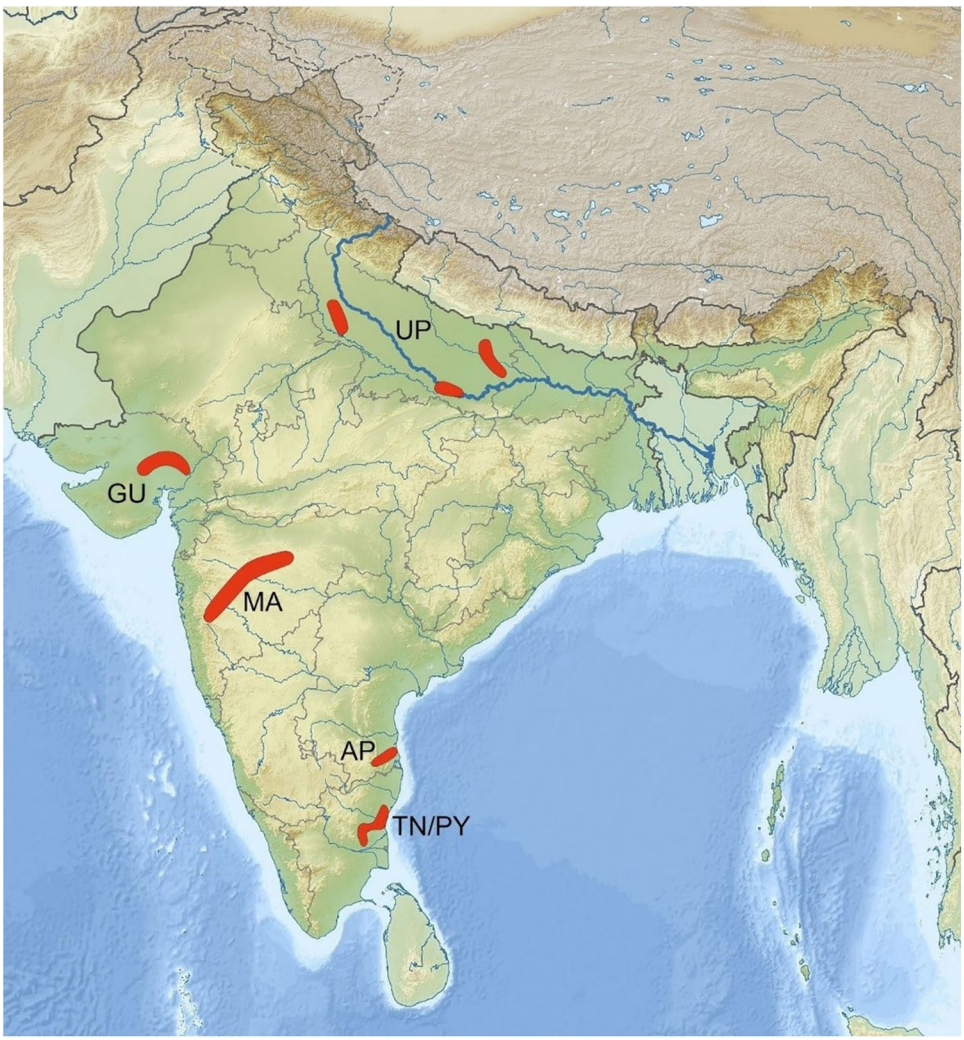

Fig. 1 Map of India with indicated in red, the sampled regions

found at Arikamedu belongs to two other glass groups (see Dussubieux et al. 2012 for more details) reflecting the use of different glass recipes involving ingredients different from *reh*. Francis concluded that the earliest producers of Indo-Pacific glass beads lived and worked in South India as early as 2nd c. BCE. In the greater region surrounding the village of Papanaidupet, recent archaeological, ethnohistorical, and ethnoarchaeological investigations have revealed further evidence for an extensive glass-working tradition in the area (Abraham 2016). In the same area, Kanungo (2002–2003) also reported evidence in the form of crucibles and glass slag to suggest possible glass production at Karakambadi. Finally, an early 20th-century British cottage industry report lists a number of villages along this river valley containing glass-making furnaces (Narayana Rao 1929). Both colonial records and recent reports have also noted the availability of glass ingredients in these southern Andhra districts (Indian Bureau of Mines 2012; Rameshchandra Phani 2014).

North India

The northern part of India, and Uttar Pradesh in particular, is heavily impacted by *reh*, and evidence of ancient and recent glassworking can be found in a number of places. The archaeological site of Kopia is located 12 km north of Khalilabad in the district of Sant Kabir Nagar, in a flood plain between the Churma and Ami rivers. Here, the discovery of hundreds of glass chunks and ceramic fragments coated with glass suggested that glass was manufactured at this site; however, most of the glass manufacturing evidence was found in undatable disturbed deposits. Charcoal found in a furnace pit did yield a date of 120 cal CE (+/− 120), and Kanungo (2010:473) suggests that "glass might have been produced here (in Kopia) from the Northern Black Polished Ware period (700–200 BCE) onwards." Elemental analysis of glass artifacts from Kopia, including raw pieces of glass, revealed the presence of m-Na-Al glass at the site.

Fig. 2 Dr Kanungo sampling potential *reh* from the surroundings of Kapadwanj

Approximately 250 km south of Kopia in southeastern Uttar Pradesh is the site of Kausambi, on the bank of the Yamuna River (70 km southwest from Allahabad), in a region containing salt-affected lands. This site may have been occupied from 600 BCE to 600 CE (Chakrabarti 1995). Gupta (1997) studied the beads found at the site, and Kanungo (2004) mentions Kausambi as a glassmaking site. This site yielded glass beads with mineral soda-high alumina compositions similar to those from Kopia (Lankton, unpublished report).

In western Uttar Pradesh, the town of Firozabad had been India's most important modern glass center for decades before its recent decline due to competition from modern Chinese production. In the same part of Uttar Pradesh, traditional glass working is still alive in the Purdilnagar-Susamayee-Jalesar-Akrabad region and although now raw glass is acquired from nearby Firozabad, *reh* glass used to be produced from local raw materials (Kock and Sode 1995; Gill 2017; Kanungo and Dussubieux 2022).

Western India

The western part of India is an important region, as this is where the exact same type of beads was identified as those found in different regions of Sub-Saharan Africa bordering the Indian Ocean (Dussubieux and Wood 2021). These beads were found at the archaeological site of Chaul, in Maharashtra state, a port site that quite likely served as a departure point for ships trading around the Indian Ocean (Gogte et al. 2006). It makes sense to hypothesize that a glassmaking center would have been established in this region to support this transregional bead trade. However, little archaeological evidence for glass manufacturing is present in this part of India, with the exception of the site of Nevasa, where evidence suggests that glass was produced during the Muslim Maratha period (Sankalia et al. 1960). At that site, beads are fairly abundant from an earlier period dating to 150 BCE–200 CE. Glass wastes dating from this

Table 1 List of samples included in this study with site where they were archaeologically excavated, the dating of the site, and basic description

Reference	Country	Site	Dating	Glass type	Artifacts	Color	Transparency
2.3.2	Thailand	Phu Khao Thong	3rd c. BCE-4th c. CE	m-Na-Al 1	Bead	Red	Opaque
JAM049	Malaysia	Sungai Mas	9th-11th c. CE	m-Na-Al 1	Bead	Turquoise blue	Translucent
JAM069	Malaysia	Sungai Mas	9th-11th c. CE	m-Na-Al 1	Bead	Turquoise blue	Translucent
JAM054	Malaysia	Sungai Mas	9th-11th c. CE	m-Na-Al 1	Bead	Black	Opaque
JAM063	Malaysia	Sungai Mas	9th-11th c. CE	m-Na-Al 1	Bead	Red	Opaque
CIB024	India	Chaul	9th-19th c. CE	m-Na-Al 2	Bead	Turquoise blue	Opaque
CIB021	India	Chaul	9th-19th c. CE	m-Na-Al 2	Bead	Red	Opaque
CIB015	India	Chaul	9th-19th c. CE	m-Na-Al 2	Bead	Green	Transparent
S1.10	Thailand	Phu Khao Thong	3rd c. BCE-4th c. CE	m-Na-Al 3	Waste	Red	Opaque
1.2.1	Thailand	Phu Khao Thong	3rd c. BCE-4th c. CE	m-Na-Al 3	Bead	Green	Transparent
PKT5.4.2	Thailand	Phu Khao Thong	3rd c. BCE-4th c. CE	m-Na-Al 3	Waste	Green	Transparent
KSEK034	Thailand	Khao Sek	4th c. -2nd c. BCE	m-Na-Al3	Bracelet	Red	Opaque
Burma002	Myanmar	Htan Ta Pin	mid-late 1st mill. BCE	m-Na-Al 3	Bead	Red	Opaque
Burma007	Myanmar	Htan Bo	mid-late 1st mill. BCE	m-Na-Al 3	Bead	Red	Opaque

Early Historic period were also recovered, and a glass furnace dating from the 3rd–4th c. CE was identified. It was made of clay and had a diameter of 75 cm. Bicolored glass fragments along with lime and cow dung were found close to the furnace. Approximately 200 km east of Nevasa is the Lonar Soda Lake where a local glass industry existed at least starting around the middle of the 18th c. CE (Brown and Dey 2008:427), suggesting that glass making was a developed craft in this part of western India as well.

In Gujarat, the glass industry can be traced back to the 17th or 18th c. CE (Dikshit 1968) in the town of Kapadwanj, and it is still active today (personal observation). Dikshit (1968) reports that the ingredients used to manufacture Kapadwanj glass were a local salt efflorescence and a sand collected in Jaipur, but Brill (2003) indicates that a few years back glass was manufactured from local *oos* (sodic efflorescence) which when melted produced a glass with a composition that was typical of medieval Indian glass.

Glass objects for isotopic comparison

As indicated in the introduction, we compare the isotopic data obtained from the sand samples with data already published in the literature (Dussubieux and Pryce 2016; Dussubieux et al. 2021) from different Indian glass samples (Table 1) in an attempt to determine artifact provenance. From the analysis of a wide range of finished glass artifacts (mostly beads) from sites within and outside India, a growing number of different sub-groups have been identified for Indian m-Na-Al glass. These sub-groups are based on the varying concentrations of the following elements: Mg, Ca, Sr, Zr, Cs, Ba, and U. For more details on differences in the compositions of the m-Na-Al sub-groups, see Dussubieux et al. (2008; 2010) and Dussubieux and Wood (2021) We hypothesize that these different m-Na-Al sub-groups correspond to glass artifacts produced from regionally distinct *reh* sands across India, and hope that isotopic signatures for these sub-groups will help identify their raw material sources. Among these sub-groups, we will focus our attention on glass artifacts from three sub-groups—m-Na-Al 1, m-Na-Al 2, and m-Na-Al 3—the only sub-groups for which Sr and Nd isotope data are available in the literature (Dussubieux and Pryce 2016; Dussubieux et al. 2021).

Five glass samples used in this analysis, all beads, belong to the m-Na-Al 1 glass sub-group. Four samples were found at Sungai Mas in Malaysia, a site dated from the 9th to the 11th c. CE (Dussubieux and Allen 2014). One sample is from the site of Phu Khao Thong in Thailand and is dated from the 2nd c. BCE to 4th c. CE (Dussubieux et al. 2012). The isotopic signature of the m-Na-Al 1 glass sub-group, when compared with published geological data, seems to point toward a southeastern Indian provenance (Dussubieux et al. 2021). Evidence for glass production in south India was reviewed earlier. Glass artifacts belonging to the m-Na-Al 1 sub-group were also found at two south Indian sites which also contained evidence for glass working, Karaikadu (Dussubieux 2001) and Manikollai (Lankton et al. 2014).

Three samples with a m-Na-Al 2 composition are glass beads found at the coastal port of Chaul in Maharashtra (Dussubieux et al. 2008) and correspond in elemental composition to glass beads from east African coastal sites. Initially, the m-Na-Al 2 glass artifacts from both Chaul and the east African sites were dated from the 9th to the 19th c. CE (Dussubieux et al. 2008), but recent analyses of additional beads from east Africa have refined that broad chronology and now suggest a time frame for m-Na-Al 2 glass from the 14th c. CE onward (Dussubieux and Wood 2021). We had assumed early on that the glass from Chaul was local (Dussubieux et al. 2008) but further research based on isotope analysis negated this hypothesis and suggested instead a provenance from the Delhi environs (Dussubieux et al. 2021).

Finally, we considered six glass samples belonging to the m-Na-Al 3 glass sub-group. Three samples (one bead and two glass chunks) come from Phu Khao Thong (2nd c. BCE to 4th c. CE), the Thai site mentioned earlier during the discussion of the m-Na-Al 1 glass sub-group. One sample, a bracelet fragment, comes from Khao Sek, another site located in Thailand dating from the 4th to 2nd c. BCE (Dussubieux and Bellina 2018). The last two last samples are beads from Myanmar and were found at two sites, Htan Ta Pin and Htan Bo in the Samon valley, and are dated from the mid-late 1st millennium BCE (Dussubieux and Pryce 2016). The presence of crucible sherds lined with greenish glass as well as raw glass fragments with a m-Na-Al 3 composition at the north Indian site of Kopia (Dussubieux and Kanungo 2013) strongly point to local glass production; however, Sr isotope analysis both of Kopia's m-Na-Al glass and of nearby raw material show two different signatures (Kanungo and Brill 2009; Brill and Stapleton 2012; Dussubieux et al. 2021).

Experimental: elemental analysis

The samples of raw material collected from these regions were heated to 1200 °C for 3 h to test whether they would turn into glass without the addition of any other ingredient(s). Traditional glass makers certainly used lower temperatures and longer melting times (e.g., Gill 2017; Kanungo and Dussubieux 2022); our goal, however, was not to reproduce these conditions but rather to test quickly whether a vitreous material could be obtained from the raw materials. Laser ablation—inductively coupled plasma—mass spectrometry (LA-ICP-MS) was used to determine the composition of the solid material. Although other techniques might have been more suitable for the kind of solid material we obtained, due to its heterogeneity, numerous LA-ICP-MS

studies on heterogeneous materials show the relevance of this technique in those cases (e.g., Simpson and Dussubieux 2018; Carter and Dussubieux 2016; Dussubieux et al. 2007).

The analyses were carried out at the Field Museum in Chicago, USA, with a Thermo Fisher ICAP Q ICP-MS. For direct introduction of solid samples, the instrument was connected to a New Wave UP213 laser for samples collected in 2018 and a ESI-Elemental Scientific Lasers NW213 laser for samples collected in 2019.

The parameters of the ICP-MS are optimized to ensure a stable signal with a maximum intensity over the full range of masses of the elements and to minimize oxides and double ionized species formation (XO^+/X^+ and $X^{++}/X^+ < 1$ to 2%). For that purpose, the argon flows, the RF power, the torch position, the lenses, the mirror, and the detector voltages are adjusted using an auto-optimization procedure.

For better sensitivity, helium is used as a gas carrier in the laser. The choice of the parameters of the laser ablation not only will have an effect on the sensitivity of the method and the reproducibility of the measurements but also on the damage to the sample. To be able to determine elements with concentrations in the range of ppm and below while leaving a trace on the surface of the sample invisible to the naked eye, we use the single-point analysis mode with a laser beam diameter ranging from 55 to 80 µm, operating at 40 to 70% of the laser energy and at a pulse frequency of 15 or 20 Hz. A pre-ablation time of 20 s is set in order, first, to eliminate the transient part of the signal and, second, to be sure that a possible surface contamination or corrosion does not affect the results of the analysis. Spots are from a few microns to a few millimeters apart from each other on one single sample. For each sample, the average of five measurements corrected from the blank is considered for the calculation of concentrations.

To improve reproducibility of measurements, the use of an internal standard is required to correct possible instrumental drifts or changes in the ablation efficiency. The element chosen as internal standard has to be present in relatively high concentrations so its measurement is as accurate as possible. In order to obtain absolute concentrations for the analyzed elements, the concentration of the internal standard has to be known. The isotope Si29 was used for internal standardization. Concentrations for major elements, including silica, are calculated assuming that the sum of their concentrations in weight percent in glass is equal to 100% (Gratuze 2016).

Fully quantitative analyses are possible by using external standards. To prevent matrix effects, the composition of standards has to be as close as possible to that of the samples. Different series of standards are used to measure major, minor, and trace elements. A standard reference material (SRM) manufactured by the National Institute for Standards and Technology (NIST), SRM 610 is a soda-lime-silica glass doped with trace elements in the range of 500 ppm. Certified values are available for a very limited number of elements. Concentrations from Pearce et al. (1997) are used for the other elements. Two other standards were manufactured by Corning. Glass B and D are glasses that match compositions of ancient glass (Brill 1999, vol. 2, p. 544).

Experimental: isotopic analysis

All isotopic sample preparations and analyses were conducted within laboratories of the Department of Geosciences, The University of Arizona, Tucson. Samples consisted of a portion of the original sand/*reh* sample, most of which had previously been analyzed by LA-ICP-MS. Typical sample weights used for isotopic analysis ranged from 25 to over 200 mg, depending upon estimates of their Sr, Nd, and Pb contents. All samples were ground in a clean agate or porcelain mortar with similar pestle, effectively homogenizing the constituents of each sample, and weighed. All acids were twice-purified and the water used for dilution was ultrapurified Milli-Q water. Dissolution of the samples for isotopic analyses was performed in screw-cap Teflon beakers using hydrofluoric-nitric ($HF\text{-}HNO_3$) acid mixtures on hot plates and hydrofluoric-chloric ($HF\text{-}HClO_3$) acid mixtures in open beakers at room temperature. The samples were taken in 1 N HCl and any undissolved residue was attacked in the same way. Any residual material remaining after digestion was removed by centrifuging. Separation of the Sr, Pb, and the bulk of the REE (for the Nd) was achieved via HCl and HNO_3 elution in cation columns. Separation of Nd was carried out using a LNSpec® resin, while Pb and Sr were separated from the same samples using Sr Spec resin (Eichrom). The highest procedural blanks measured during the course of this study were 180 picograms (pg) Sr, 18 pg Nd, and 100 pg Pb.

The filament loading and mass spectrometric analysis procedures were similar to the ones previously described by Pickett and Saleeby (1994). Sr and Nd isotopic ratios were measured using a VG Sector 54 multi-collector thermal ionization mass spectrometer (TIMS). The Sr isotopic ratios were normalized to $^{86}Sr/^{88}Sr = 0.1194$, whereas the Nd isotopic ratios were normalized to $^{146}Nd/^{144}Nd = 0.7219$. All $^{87}Sr/^{86}Sr$ measurements are reported in Table 2 of the Supplementary Materials. Estimated analytical $\pm 2\sigma$ uncertainties are $^{87}Sr/^{86}Sr \pm 0.0015\%$ and $^{143}Nd/^{144}Nd \pm 0.002\%$. External reproducibility, based on the range of multiple runs of standard NBS987 (for Sr) and LaJolla Nd (for Nd) is estimated to be ± 0.000014 for Sr, and ± 0.00001 for Nd. The grand means of isotopic ratios were corrected by an off-line manipulation program, which adjusts for fractionation correction and performs isotope dilution calculations.

Isotopic analyses of lead were made using a Micromass IsoProbe multi-collector inductively-coupled plasma mass

Table 2 Average concentrations with standard deviation indicated below for the raw materials collected in the different regions. Concentrations are either in w% of oxide or in ppm of element

	MgO	CaO	Sr	Zr	Cs	Ba	U
Andhra Pradesh	1.3%	3.0%	145	645	2	417	5
	1.7%	6.3%	138	2497	2	268	6
Gujarat	1.4%	6.1%	202	53	4	278	4
	0.7%	4.4%	101	21	3	240	5
Maharashtra	4.7%	7.4%	366	469	1	354	1
	2.8%	3.2%	403	756	0	373	1
TN/P	1.2%	3.0%	229	112	1	463	1
	1.2%	2.9%	143	383	1	327	1
UP-NE	0.7%	1.2%	72	647	5	317	9
	0.4%	2.0%	25	973	3	85	8
UP-SE	1.1%	1.9%	120	220	6	346	3
	0.4%	1.4%	32	360	3	54	2
UP-W	1.8%	2.6%	166	164	6	365	7
	1.6%	4.0%	211	236	3	81	8

spectrometer (MC-ICP-MS) at the University of Arizona. We analyzed NBS981 (Pb) in between unknown samples in the analysis sequence and all results were corrected for mercury and empirically normalized to thallium using the exponential law correction after Rehkämper and Mezger (2000). To correct for machine and inter-laboratory bias, all results were normalized to values reported by Galer and Abouchami (1998), for the NBS-981 standard ($^{206}Pb/^{204}Pb = 16.9405$; $^{207}Pb/^{204}Pb = 15.4963$; $^{208}Pb/^{204}Pb = 36.7219$). Internal error reflects reproducibility of measurements on individual samples, whereas external errors are derived from long-term reproducibility of NBS981 lead standard and results in part from mass bias effects within the instrument. In all cases, external errors exceed internal errors.

Results and discussion

Raw materials

Heating the raw materials resulted almost always in a solid block but we rarely obtained a fully vitreous material (Fig. 3). This can be explained by the elemental composition: the elemental composition of the raw material samples that became solid blocks revealed that they contain little or no sodium. Samples that did not melt were eliminated and were not added to the Supplementary Materials—Table 1.

Some samples had a high calcium concentration, suggesting the presence of calcium carbonate or other calcium-rich minerals. It should be noted that we collected the raw material samples during the summer because that was the only time of the year compatible with our schedules. We are aware that post-monsoon winter is a better time for *reh* collection, as this is when water evaporation brings sodium to the surface. Although the collection season may have an impact on the sodium levels, it should not affect isotopic composition, which is mostly influenced by the sand. We can simulate the presence of soda as found in the m-Na-Al glass by re-calculating the composition of the raw material samples, assuming a proportion of ~ 20% of soda. By doing so, a number of the samples appear to have a composition containing more than 5% of alumina (see Table 1 in Supplementary Materials). For these samples (with $Al_2O_3 > 5\%$), principal component analysis was conducted using the GAUSS Runtine v. 8.0 software available on the website of the University Missouri Research Reactor Archaeometry Lab with the elements Mg, Ca, Sr, Zr, Cs, Ba, and U, that were found useful in the past for separating different m-Na-Al sub-groups (see Dussubieux et al. 2010; Dussubieux and

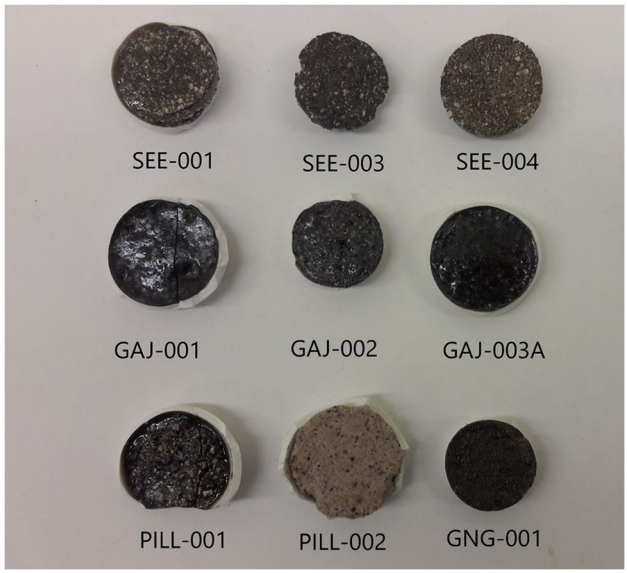

Fig. 3 Raw material samples after heating at 1200 °C for 3 h

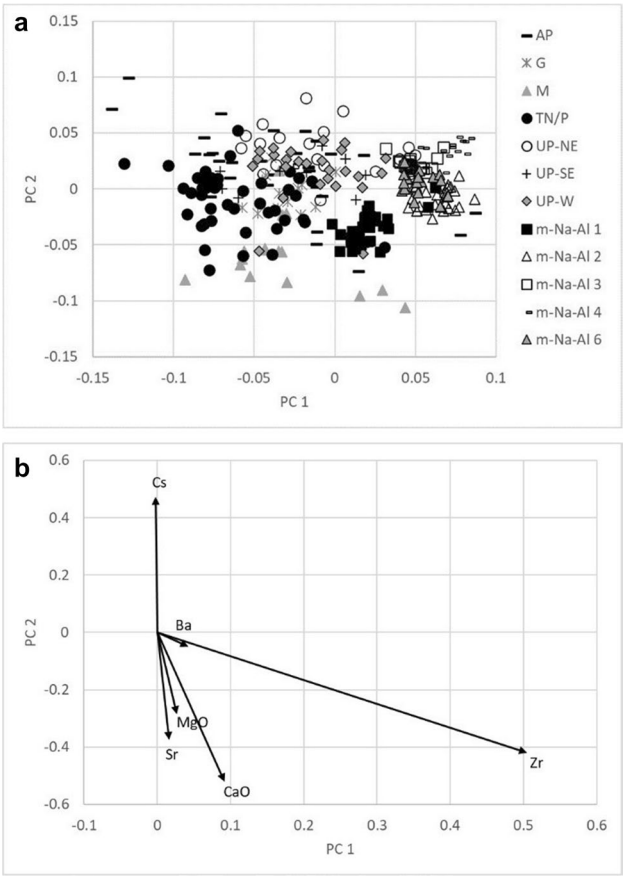

Fig. 4 **a** PC 1 and 2 calculated using Mg, Ca, Sr, Zr, Cs, Ba, and U concentrations for the raw materials with $Al_2O_3 > 5\%$ and for some samples belonging to the m-Na-Al 1 (unpublished data), m-Na-Al 2 (Dussubieux et al. 2008), m-Na-Al 3 (Dussubieux and Kanungo 2013), m-Na-Al 4 (Dussubieux 2009), and m-Na-Al 6 glass groups (Dussubieux and Wood 2021). AP is for Andhra Pradesh, G for Gujarat, M for Maharashtra, TN/P for Tamil Nadu/Puducherry, UP for Uttar Pradesh, NE for northeastern, SE for southeastern, and W for western. **b** Component loadings by element. PC 1 summarizes 47.5% of the total variability of the data and PC 2 18.8%

Wood 2021). Principal components 1 and 2 are shown in Fig. 4a and b. We observe an important sort of variability in the results from Andhra Pradesh; the compositions for this region overlap with most of the compositions of the other regions. The compositions for Maharashtra, Tamil Nadu/Puducherry, and Uttar Pradesh overlap slightly but in general, different trends are visible. The material collected in Maharashtra has the highest average MgO and CaO concentrations, while raw material from Tamil Nadu/Puducherry has the lowest Zr concentrations of the three greater regions. Globally, the samples from Uttar Pradesh have the lowest MgO and CaO concentrations but the highest Cs and U levels (see Table 2). The three Uttar Pradesh sub-regions cannot be separated based on the elemental composition. The Gujarat material has low Zr concentrations and appears quite similar to the Tamil Nadu/Puducherry material.

A comparison of the compositions of the raw material samples collected during this project with the compositions of known m-Na-Al 1, 2, 3, 4, and 6 glass artifacts show very little overlap. This suggests that the *reh* samples we collected do not correspond to *reh* sources used to produce the Indian glass artifacts studied so far. A few exceptions appear for samples from northeastern Uttar Pradesh that do overlap with the m-Na-Al 3 glass samples and possibly the m-Na-Al 6 glass group. It is important to emphasize the ubiquity of potential raw material sources for glass in India; isolating the precise sources used in ancient times is certainly a "needle in a haystack" kind of search, which is why isotopic data are also an important aspect of this project.

Our hypothesis included an analysis of the isotopic composition of the raw materials we collected, in the hope that these data would generate region-specific signatures that we could then match with the isotopic signatures of m-Na-Al glass artifacts (Table 2 in Supplementary Materials). Looking at the Sr and Nd data for the raw materials (Fig. 5), the samples collected from the relatively young and non-radiogenic deposits of the Deccan Plateau in Maharashtra in western India separate well from all other samples, as do the samples from Gujarat in western India and the south Indian regions of Tamil Nadu/Puducherry. The convoluted and deep geological antiquity of Andhra Pradesh in south India results in a wide range of highly radiogenic values for both strontium and neodymium that are distinct from all the other regions in this study. While Uttar Pradesh (UP) in north India also separates relatively well from all other sample regions, there is significant overlap in some of the values from sampling sub-regions, particularly within the western and southeastern Uttar Pradesh samples, all of which would be strongly influenced by the flow and alluvial depositions of the Ganges River. The northeast region of Uttar Pradesh separates better from the two other sampled regions (UP-SE and UP-W) in this state, likely due to its location along a different waterway—it originates from a different region in the Himalayas that has a slightly different isotopic signature. The lead isotope did not help us distinguish isotopically between western and southeastern Uttar Pradesh, confirming instead similarity in mixing of the geological source materials for these two regions (Fig. 6).

Comparison between raw materials and archaeological isotopic signatures

Returning to the Sr and Nd signatures of these different regions, we compared them to the isotope signatures of the three sub-groups of Indian glass: m-Na-Al 1, m-Na-Al 2, and m-Na-Al 3.

Three of the five m-Na-Al 1 glass data points match the signature identified for raw materials sampled from the Tamil Nadu/Puducherry area in south India, confirming at

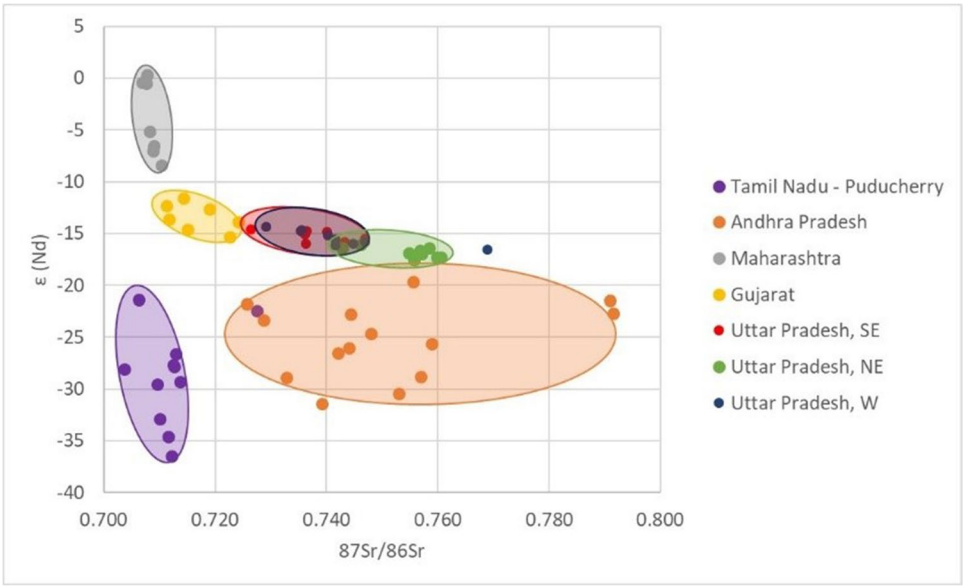

Fig. 5 $^{87}Sr/^{86}Sr$ and ε(Nd) for the raw materials collected in different regions of India for this study. Ellipses are not confidence ellipses but are just meant for help in visualizing the range of samples for the different regions

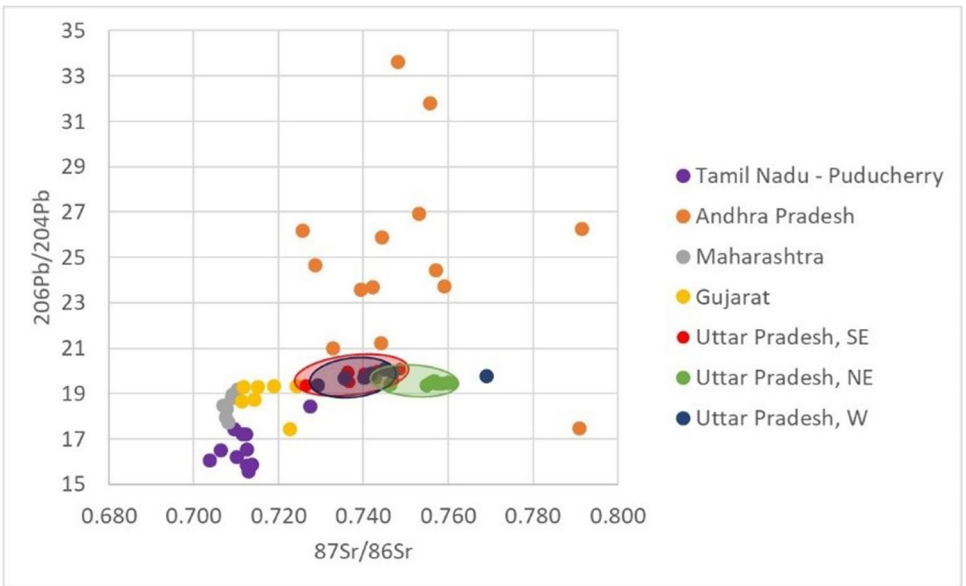

Fig. 6 $^{87}Sr/^{86}Sr$ and $^{206}Pb/^{204}Pb$ for the raw materials collected in different regions of India for this study. Ellipses are not confidence ellipses but are just meant for help in visualizing the range of samples for the different regions

least partially our previous provenance attribution (Fig. 7). Two samples fall outside of the isotopic region defined by the data produced in this study. More geological data would be necessary to confirm the range of the Sr and Nd signatures for the Tamil Nadu/Puducherry area.

None of the three Chaul samples (m-Na-Al 2 glass artifacts) match the geological data collected for Maharashtra (Fig. 7) which confirm what was observed in Dussubieux et al. (2021). In the same article, a glass provenance from the Delhi area was proposed. The immediate region around Delhi was not sampled as part of our study—the closest region we studied would be western Uttar Pradesh (~ 250 km southeast of Delhi). The Chaul Sr and Nd signatures do not match the data collected in that area. Instead, two of the glass samples fall in the Andhra Pradesh area while another point is in an area not associated with any of the regions we investigated. It seems that two different isotopic signatures exist for this small group of m-Na-Al 2 glass beads, suggesting maybe the existence of two sub-groups that were not recognized when considering only the elemental analysis.

With regard to m-Na-Al 3 glass, our results suggest that four of the m-Na-Al 3 beads might come from a glass manufactured either in southeastern or western Uttar Pradesh. The two other samples do not match the data for any of the regions we investigated and point to the existence of sub-groups for the m-Na-Al 3 glass that, like the

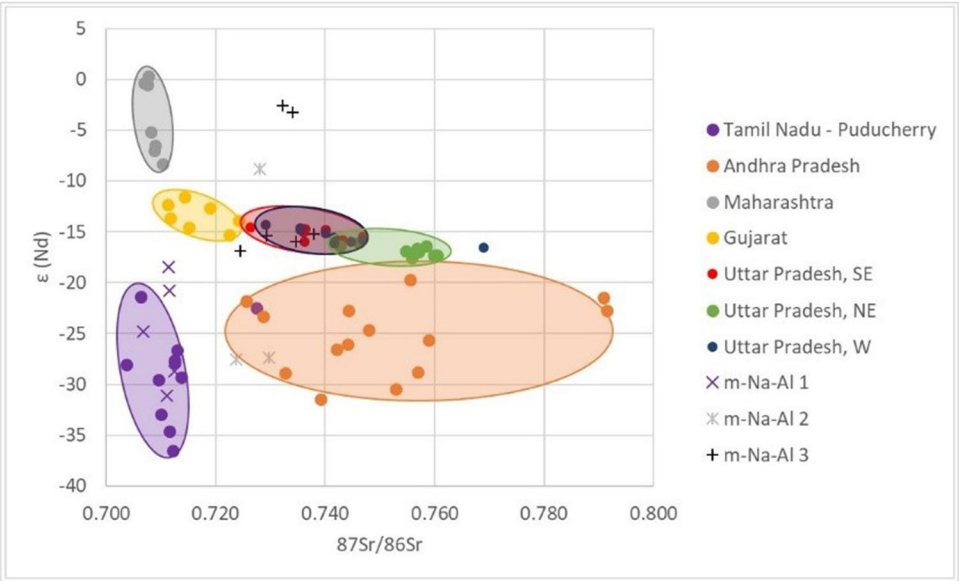

Fig. 7 $^{87}Sr/^{86}Sr$ and ε(Nd) for the raw materials collected in different regions of India for this study. Data for m-Na-Al 1, 2, and 3 glass samples were added to this graph. Ellipses are not confidence ellipses but are just meant for help in visualizing the range of samples for the different regions

two m-Na-Al 2 samples, was undetected with elemental analysis (Fig. 7).

Conclusion

This study shows the great potential of combining elemental and isotope analysis data to provenance ancient Indian glass, although the task is challenging given the ubiquity of the raw material and the huge size of the country. We are in the process of acquiring more data points for each of the regions, which will allow us to have a better idea of the variability of their isotopic signatures. We are also currently acquiring additional isotopic data for more Indian glass artifacts to further our comparisons with the raw material samples. Current results are encouraging, but given the geographic limits of this study, more extensive raw material sampling from additional areas is necessary. Unfortunately, ancient glass production in South Asia is a somewhat marginal topic in glass research in general; this project is a first step towards filling the lacunae. We hope this study will motivate more research and encourage others to systematically address this region when considering glass exchanges globally.

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Author contribution All authors participated to the sample collections, elemental analysis was carried out by LD and isotopic analysis by TF. LD, TF, SA, and AK contributed to the writing of the manuscript. All authors agreed to the publication of the manuscript.

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Data availability All data relevant to this manuscript were placed in supplementary materials.

Declarations

Competing interests The authors declare no competing interests.

Ethics approval The funding organization had no influence on the study design, data collection, and analysis, decision to publish, or preparation of the manuscript.

Consent to participate N/A.

Consent for publication N/A.

Conflict of interest The authors declare no competing interests.

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