ANCIENT EGYPTIAN SULFUR BEADS¹

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The Ancient Egyptian and Near Eastern Collection at Tokai University (AENET), Japan, contains two unique necklaces made of an opaque yellow substance identified as sulfur through XRF and XRD analysis. Sulfur beads are rare and have not been adequately studied. We therefore undertook a study of the AENET beads and estimate that they date to the Ptolemaic and early Roman periods in Egypt. A digital-image comparison between the AENET beads and similar beads in another museum collection shows a strong correlation, suggesting that they share a single mold. An isotopic analysis also provides a specific fingerprint of the sulfur. Experiments to replicate the beads indicated that they were made by pouring molten sulfur into a greased mold. The process is simple, revealing that a small-scale cottage industry was sufficient to make them. The beads were used for funerary purposes (likely incorporated into broad collars) rather than in daily life because oxidized sulfur emits an unpleasant odor, discouraging people from wearing them every day.

INTRODUCTION

The Tokai University collection of ancient Egyptian artifacts includes two necklaces composed of opaque yellow beads (Figure 1). They are somewhat porous with visible crystalline structures. In that a previous paper (Yamahana and Akiyama 2017) provides a detailed description of each bead and discusses the dating, only a summary discussion is presented here. This article combines archaeological and scientific methods to determine the date, composition, and method of manufacture of the beads.

DESCRIPTION OF THE BEADS

The AENET collection contains approximately 6000 books, 15,000 images, and 6000 artifacts of archaeological value. They were donated to Tokai University in 2010 by the family of the late Professor Emeritus Hachishi Suzuki, who lived in Egypt from 1958 to 1968. Most of the artifacts were purchased from antique dealers in Cairo, and the two strings of yellow beads (reg. nos. SK 10 and SK 176) also appear to be purchased items.

One might speculate that the yellow beads unearthed in Egypt were made of glass because yellow glass beads were not uncommon to ancient Egypt. The beads do not appear to be glass, however, but are made of an opaque yellow porous substance with a matte texture and a peculiar needle-like crystalline structure on the reverse. They smell faintly of rotten eggs, suggesting the presence of a sulfur compound.

Common materials for ancient Egyptian beads are bone/ tusk, stone, clay, glass, and faience. Beads made of sulfur rarely occur in an archaeological context. A few sulfur bits of irregular shape were found at two ancient sites, Defenneh and Badari (Lucas and Harris 1962; Petrie 1888), but not in the form of beads. Comparable beads can, however, be found in the Louvre Museum in Paris (Keimer 1938:208), the Egyptian Museum in Cairo (reg. nos. JE71593a-c), and the Hirayama Ikuo Silk Road Museum (reg. no. NR103112) and the Kobe Lampwork Glass Museum (Habara 2015:7) in Japan. The beads in the first two museums were accessioned at the beginning of the 20th century, while the beads in the latter two were purchased from antique dealers and registered later. The AENET beads were most probably acquired between 1958 and 1968, when the late Professor Suzuki lived in Cairo. All of them, unfortunately, lack the original provenience.

SK 10 and SK 176 are the two strings of opaque yellow beads that are discussed in this paper. SK 10 has 18 bucranium ("ox head") and 50 12-petal floral beads, while SK 176 has 26 bucranium and 45 12-petal floral beads, for a total of 139 beads with 44 bucranium and 95 floral shapes. The beads are strung on modern blue cotton thread, indicating that the beads were recently formed into two necklaces and do not reflect their original context.

Radial grooves emanate from the center of the floral beads to represent petals (Figure 2, left). The back is flat and plain, though many beads exhibit a peculiar crystalline structure. The average diameter is a consistent 12 mm, but the thickness varies from 2 mm to 4 mm; even a single bead exhibits an uneven thickness. There is almost always a dark-colored disk bead about 2 mm in diameter in the center of



Figure 1. String SK 176 of yellow sulfur beads (photo: S. Miyahara; subsequent images are by the authors).

the bead, as if to represent the disk floret of a flower. A hole passes through the center of each bead perpendicular to the short axis.

The bucranium bead represents an ox head with two horns and small bumps between them (Figure 2, right). Two ears, eyes, and nostrils are indicated. The maximum width is 18 mm, with a thickness that varies from 3 mm to 6 mm. The perforation extends horizontally between the horns and ears. The average perforation diameter of both bead forms is approximately 1.2 mm.



Figure 2. A 12-petal floral bead and a bucranium bead.

DATING THE SULFUR BEADS

Dating the floral beads is difficult since that form was in use in ancient Egypt since the beginning of the pharaonic period. Conversely, the bucranium beads have unusual bumps between the horns that may allow us to determine a specific date.

The decorative row of bumps between the bucranium's horns has no parallel in dynastic Egypt. There are, however, instances of bucrania crowned with floral garlands during the Ptolemaic and Roman periods. The bucranium most likely represents the goddess Hathor since she is one of the few deities depicted by a bucranium/human frontal view. In contrast, most gods and goddesses are represented by their profiles in ancient Egypt.

Hathor takes the form of a human, a bucranium, or a human with cow ears and horns. She is one of the most popular deities in the ancient Egyptian pantheon. Since she was the goddess of motherhood, worshiping her became popular, especially during the latter part of ancient Egyptian history. Thus, the bucranium beads most probably date to the period between the Ptolemaic (304-30 BCE) and early Roman (30 BCE to probably the end of the 2nd century AD) periods.

Nearly identical beads are held by the Egyptian Museum in Cairo, the Louvre, the Hirayama Ikuo Silk Road Museum (Kamakura, Japan), and the Kobe Lampwork Glass Museum (Kobe, Japan). All museums, including the AENET, share the 12-petal and bucranium beads. In the Hirayama Ikuo Silk Road Museum collection, however, the beads are in the form of Bes, an ancient Egyptian protective deity who gained popularity throughout the Mediterranean coastal areas from the Late period to the early Roman period (from approximately the 7th century BCE until the 2nd century CE). The Egyptian Museum in Cairo also has 15-petal beads made of the same yellow material. In all, four variations – 12 floral petals, 15 floral petals, bucranium, and the Bes figure – are known, for a total of 342 beads (Table 1).

The photographic measurements taken of both the 12-petal and bucranium beads are identical. The beads in other collections were also photographed and compared with the AENET specimens. The measurements of beads in other Japanese collections are almost identical to the AENET beads. Although the beads in Cairo and Paris were inaccessible for study, the approximate measurements obtained from their photographs also show a close similarity to the AENET beads. The uniformity of shape and size, together with the rarity of the material, suggest that the beads were produced at one time in the same locality.

ARCHAEOMETRIC ANALYSIS

Elemental XRF Analysis

As noted above, the two strings of yellow beads (SK 10 and SK 176) emit a distinctive sulfurous odor. The beads were analyzed using non-destructive X-ray spectroscopy

(XGT-2700 HORIBA). The analyzed points were yellowbased materials of the floral and bucranium beads and a purple bead embedded in the center of the floral beads. The XRF setting was Rh as a target, 30kV, 0.8mA, 150 seconds, and a measured diameter of 100 μ m. The bead samples were analyzed in a vacuum chamber.

Analysis revealed that both bead forms have identical compositions. The material shows a strong sulfur peak around 2320 eV (Figure 3, left). Quantitative analysis indicates that the yellow base is 95 wt% of sulfur. The purple-black disk bead, on the other hand, is composed of 63 wt% of silica, 9 wt% of calcium, and 8 wt% of iron, with the remaining 20% consisting of manganese and other minor elements (Figure 3, right). This is most likely a sodium-silicate vitreous material with a dark purple colorant. The use of manganese-iron black is an indication of the authenticity of the beads since this particular colorant was widely used in ancient Egyptian vitreous materials such as faience and glass, especially during the Late period to the early Roman period (ca. 7th century BCE to the 2nd century CE).

The use of sulfur is rarely mentioned in ancient Egyptian texts, only appearing in a medical text on treating eye diseases such as pterygium (Bryan 1930). Archaeologically, a sulfur nugget was found in a pot together with an organic spice at Defenneh (Petrie 1888). Several nuggets were also found near Badari, but their chronology and function remain undetermined (Keimer 1938).

Structural Analysis by XRD

An X-ray diffraction analysis (Bruker, D8 Discover) of the beads was also conducted to examine their crystalline structure. The purple-black disk bead was cut to expose a fresh section to eliminate contamination. The XRD setting

Beads	Museums						Ratio
	AENET (SK 10-1)	AENET (SK 176)	Egyptian Museum	Hirayama Ikuo Silk Road Museum	Kobe Lamp- work Glass Museum		
12-petal floral	50	45	72	28	33	228	66.6%
15-petal floral	0	0	2(?)	0	0	2(?)	0.5%(?)
Bucranium	18	26	46	2	15	107	31.2%
Bes	0	0	4	1	0	5	1.5%
Total	68	71	124(?)	31	48	342	100%

Table 1. Quantities of Sulfur Beads in Known Museum Collections.



Figure 3. XRF spectrum of a bucranium bead (left) and a purple-black disk bead in the center of a floral bead (right).

was Cu K α as X-ray source, 30kV, 15mA, collimator diameter 0.1 mm. Figure 4 compares the XRD pattern of a bucranium bead to that of orthorhombic sulfur. The intense X-ray diffraction around 23.1 degrees (2 θ) indicates that the crystal is polycrystalline orthorhombic sulfur. The presence of a halo reveals that the material also contains amorphous features.

A comparison of the XRD result of a small purpleblack disk bead and that of quartz is provided in Figure 5. A prominent silica peak and a slight indication of the amorphous phase are its principal characteristics. A distinctive amount of calcium is present in the disk bead (Figure 3), indicating that the material is not ceramic (unglazed and glazed pottery or porcelain). Although it has a smooth vitreous surface and looks like glass, it is actually faience, a precursor of glass. Faience first appeared in Mesopotamia and Egypt around 4500 BCE and spread throughout the ancient Near East and Mediterranean. Its production died out after the Roman conquest of Egypt and it was no longer being made by the beginning of the 2nd century CE. We can, therefore, assume that the sulfur beads embedded with the faience disks were made before this time.

Isotopic Analysis

Some fragments of beads of strand SK 10 were also examined using stable isotope analysis (34S/32S ratios) to determine the provenience of the sulfur, which provides clear evidence of the origin. The investigation was conducted by Professor Mizota of Iwate University and Dr. Yamanaka of Tokyo University of Marine Science and Technology (Mizota, Yamanaka, and Yamahana 2018).

δ34S/δ32S ratios for native sulfur were measured online using a continuous flow mass spectrometer coupled with an elemental analyzer (Isoprime EA: GV Instruments, Cheshire, UK). The result is summarized in Table 2. The isotopic composition has a narrow range from +3.3 to +4.0%, with an average value of +3.7%, while the standard deviation of the measurement is 0.2%. The tested specimens proved to be reasonably homogeneous in nature.

Ras Jemsa, Bir Ranga, and Ras Benas are historically known sources of sulfur, all of which are located on the coast of the Red Sea. There is also a small sulfur deposit called "sulfur springs" at Helwan (Lucas and Harris 1962). Unfortunately, the isotopic composition of the sulfur from



Figure 4. XRD patterns of a bucranium bead (left) and orthorhombic sulfur (right).



Figure 5. XRD patterns of a purple-black disk bead in the center of a flower bead (left) and quartz (right).

these Egyptian sites has not been investigated. Thus, it is impossible to perform a comparative study to determine the provenience of the AENET beads. Relatively pure sulfur is obtainable by heating a sulfur-containing mineral to a temperature high enough (140 °C) for the sulfur to melt.

BEAD PRODUCTION TECHNOLOGY

The sulfur beads must have been produced by either molding or carving. The former method consists of either filling a mold with powdered sulfur and compressing it into a solid mass or pouring liquid sulfur into a mold. Carving involves shaping crystalline sulfur with a sharp tool. There are some archaeological finds of sand molds for metal production, terra cotta molds for making faience objects, and gypsum molds for an unknown purpose. We made terra cotta and gypsum molds to test the feasibility of these methods. In the first experiment, we filled a mold with sulfur powder and then pressed the mold to solidify it. The results demonstrated that neither mold could withstand the pressure. The second possibility, pouring liquid sulfur into a mold, will be discussed below.

Carving a bead from solid sulfur was another possible manufacturing method. The XRD analysis indicates that

Table 2. Isotopic Composition of Three BeadFragments from String SK 10-1.

Sample no.	$\delta^{34}\!S_{vs.V\text{-}CDT}$ values (%)			
1. Small	+3.7; +4.0			
2. Medium	+3.3; +3.7			
3. Large	+3.6; +3.8			
Average standard deviation $+3.7 \pm 0.2$				

the ancient beads are made of polycrystalline sulfur. It is, however, possible that the sulfur was once a crystal and later assumed a polycrystalline structure. Crystal sulfur has a translucent yellow color and looks like a semi-precious stone, making it attractive enough for ornamental jewelry. It is as soft as gypsum, rating a hardness of 2 on the Mohs scale. In a second experiment we made a glassy crystal of sulfur from carbon sulfide. Carving it was challenging due to cleavage. It was also fragile under pressure and shattered easily, revealing that carving beads from crystallized sulfur was not a realistic choice (Yamahana and Akiyama 2017).

The last possibility involves pouring molten sulfur into a mold. Figure 2 shows macro images of a floral and a bucranium bead. Both exhibit untrimmed excess material (flashing) along the edges clearly resulting from casting. Moreover, the backs of the beads are concave, and needle-like crystal structures are present on almost all of them (Figure 6). Monocrystalline sulfur is stable at high temperatures but becomes orthorhombic under 95.6 °C and



Figure 6. The back of a floral bead.

volume contraction occurs. This causes a slight depression in the back of the beads. The needle-like structures formed when the monocrystal transformed into an orthorhombic crystal. The evidence indicates that the beads were made by pouring molten sulfur into a mold.

There are 44 bucranium beads in the AENET collection and they are all similar. Their apparent similarity was tested using digital pattern matching (Figure 7). Horizontal lines were drawn on samples A and B, and every fifth line of A was replaced with the same line of B. The joint image (Figure 7, right) clearly retains the bucranium's facial traits, and shows distinctive, almost identical, facial features with uneven right and left eyes, eyebrows, and ears. The bucranium beads in the Ikuo Hirayama Silk Road Museum were also examined using pattern matching and are virtually identical to those at AENET. It is therefore reasonable to assume that the bucranium beads were all made in the same mold or molds made using the same master model.

On the other hand, the floral beads seem to have been made in several molds. Although the beads are all 12-petal floral, the reference lines do not always share the same pattern. Some are quite similar, but others appear slightly different. Digital pattern matching was difficult due to the rounded shape of the beads with no distinct cardinal point for comparison. In that floral beads comprise 66% of the entire sulfur bead collection, it seems likely that several floral molds were used to create the more than 200 beads.

The next step in our experiment was to make molds which could be used to replicate sulfur beads. To do this, we 3D-scanned the floral and bucranium beads with the cooperation of Abist Ltd. (Meshlab, v.32bit, 1.3.3.) (Figure 8). This is an effective way of examining valuable archaeological objects in detail without handling them. The images show the excess material along the edges of the floral beads and behind the ears of the bucranium beads. The molds were, therefore, relatively shallow but deep enough to accommodate the string hole. This is round and was likely made by putting a reed or twig in the mold. In this way, the artisans could mass produce the beads.

REPLICATING SULFUR BEADS

There is no archaeological evidence to suggest that molds were used to make the sulfur beads. There is, however, some material evidence that gypsum, terra cotta, and sand molds were used during the New Kingdom period (1550-1070 BCE). Sand molds can be excluded as potential candidates since the casting surface is not smooth enough to retain fine details, such as the eyes and nose, leaving gypsum and terra cotta as the probable candidates. Resin replicas of the beads were printed from the 3D-scan data. These were used as master models to make the molds (Figures 9-10). The depth of the molds was adjusted according to the measurements of the actual beads.

Since the melting point of orthorhombic sulfur (Ø-sulfur) is 112.8 °C and that of monocrystalline sulfur (β sulfur) is 119.6 °C, we heated the molds filled with sulfur powder to 130 °C. The sulfur liquified as expected, but it stuck to the molds, making it impossible to extract the beads without breaking them. A parting agent, which will be discussed later, was subsequently applied to the molds, but it fused with the sulfur powder during heating, failing its intended purpose.

We then undertook to pour liquid sulfur into the molds. The sulfur was heated to 140 °C using an alcohol lamp. This temperature is above the boiling point of water but low enough so that a reed or twig could be used to form the perforation. A blackish glass bead was placed in the center



Figure 7. Pattern-matching of bucranium beads. Sample A (left), sample B (center), and a digitally combined image of samples A and B (right).



Figure 8. 3D images of a bucranium bead and a floral bead.

of the floral bead mold and a plastic stick was set to make the string hole. Liquid sulfur was then poured into the molds of the two bead forms. Separating the solidified beads from the molds was more manageable than in the former experiment, but it was still challenging. It was also difficult to achieve the delicate impressions of the eyes and ears of the bucranium beads. Regarding the first problem, we realized the mold should be warm so that the liquid sulfur would not harden before the fine details could be copied. As for the second, we found that a parting agent definitely facilitated the removal of beads from the molds. In ancient Egypt, they used oils of such plants as castor, Tribulus, safflower, moringa, linseed, olive, almond, rapeseed, and sesame. Animal fat - such as beef tallow, lard, and sheep and goat fat - was also used for cooking and other purposes (Serpico and White 2000). Vegetable oil would have been more easily accessible to commoners than animal fat since the latter was often used in palaces and temples where numerous sacrifices were made daily. Any of the aforementioned oils could have been used as parting agents, though olive oil seems to have been the most common, being used since the beginning of the dynastic period. We therefore chose olive oil for our experiment.

After applying olive oil to the gypsum and terra cotta molds, we put a purple-black glass disk bead in the center of

the floral molds and heated them to 100 °C. We then poured molten sulfur heated to 140 °C into the molds and left them at room temperature for 20 minutes. For the terra cotta mold, the use of olive oil as a parting agent was successful and all the beads could be removed intact (Figure 11). The delicate impression of the eyes and ears of a bucranium were copied effectively, and the replicated beads also exhibited needle-like sulfur crystals. On the other hand, the olive oil was absorbed by the gypsum mold before the liquid sulfur could be poured and failed as a parting agent. Terra cotta is, therefore, the most plausible mold material for making sulfur beads.

CRYSTAL STRUCTURE OF THE REPLICATED BEADS

Liquid sulfur has a transparent yellow-green color. After pouring it into a mold, the fresh color gradually fades and turns into an opaque creamy yellow. As this happens, the needle-like crystal structure grows on the exposed side. The color change and the crystal growth indicate that the sulfur transforms into monocrystalline sulfur (β -sulfur) right after it is poured. The sudden shrinkage of volume causes the crystals to grow. The sulfur stabilizes into orthorhombic sulfur (α -sulfur) after being left below 95.6 °C (i.e., room temperature), causing the sulfur to turn opaque yellow.

Unique morphological transformation occurs when the hot sulfur cools. The change from monocrystalline to orthorhombic sulfur begins right after solidification. Figure 12 (left) shows the XRD pattern of a replicated bead an hour after it was made, while Figure 12 (right) is the pattern after three days. The intense X-ray diffraction, around 26 degrees (2 θ), indicates the crystal preferentially oriented on a {026} plane. The broad halo in Figure 12 (left), which shows the amorphous phase, vanished after three days and the material became orthorhombic sulfur (Figure 12, right). The



Figure 9. Reproduction gypsum mold.



Figure 10. Reproduction terra cotta mold.

crystalline structure of both the AENET bead (Figure 4) and the laboratory reproduction (Figure 12, left) are very similar; only the latter shows the presence of a broad halo. The high signal-to-noise ratio of the X-ray diffraction profile indicates that the crystal has a good crystallinity. Sulfur is, however, vulnerable to air, hot water, and bacteria, and contact with them easily oxidizes it, changing its chemical structure. Hence, the amorphous phase of the AENET beads (Figure 4) may have resulted from oxidization and degradation. The AENET beads emit a distinctive odor due to the presence of sulfur dioxide and hydrogen sulfide, chemical compounds formed when sulfur oxidizes.

THE FUNCTION OF THE BEADS

It is widely known that craftsmanship was highly developed in ancient Egypt, especially after the Ptolemaic



Figure 11. Replicated bucranium and floral beads, front and back.

period, when the AENET sulfur beads are thought to have been produced. Sophisticated products were made by the skilled craftsmen using the high-tech production methods and equipment of the time. They made high-quality objects using precious or semi-precious materials such as gold and silver, which have high melting points. Their customers were usually from the upper social class.

Conversely, the AENET beads did not require sophisticated technology to produce. Sulfur is easily melted and does not require special knowledge to manipulate. All that was needed was a mold, a small lamp, a parting agent, a reed or a stick to make the hole, and sulfur. The production of sulfur beads was likely more a small-scale cottage industry than a major operation. Given that there are fewer than 350 sulfur beads in collections worldwide, we may assume that the AENET beads were part of a one-time production in a local workshop.

The sulfur beads were definitely intended for ornamental purposes, but sulfur emits an unpleasant odor during oxidation, something the AENET beads still exhibit, even after more than two thousand years. It would, therefore, have been unpleasant for people to use sulfur beads in their daily lives.

In ancient Egypt, yellow pigments such as ocher or orpiment were used extensively in funerary contexts – such as tomb murals and coffin decoration – as a substitute for gold. The story, "Shipwrecked Sailor," written during the 12th Dynasty (ca. 1976-1794 BCE), mentions that god's skin is made of gold (Lichtheim 1985). We, therefore, assume that the sulfur beads were funerary ornaments, perhaps to adorn the dead.

There are two possible ornamental configurations for the beads: a single-string necklace or a broad collar (for details, *see* Yamahana and Akiyama 2017). If the former, several strands could have been produced, based on the number of beads in the collection. It is, however, more likely that they comprised a broad collar (*wesek*) which was worn by the gods and goddesses of ancient Egypt, and was essential funerary attire for the dead, the deceased being considered gods in the afterlife. Reconstructions of both forms were created using beads of yellow-dyed silicone made in the reproduction terra cotta molds (Figure 13).

Further support for the supposition that the sulfur beads were not made for everyday use is in the form of the suspension hole. Each bead has a single perforation that causes the bead to flip over when threaded. At least two holes are needed to keep the faces of the beads in position. If worn by the living, it would have been an annoyance to constantly flip them back to the proper position.



Figure 12. XRD patterns of replicated sulfur beads: one hour after synthesis (left) and three days after synthesis (right).

CONCLUSION

Our research has shown that the yellow beads were made of almost pure sulfur. The opaque yellow color with the needle-like crystal formation resulted from the transformation of monocrystalline to orthorhombic sulfur. The beads were mold-made at relatively low temperatures using simple techniques and tools. They indicate the presence of a beadmaking cottage industry during the later period of ancient Egyptian history, most probably from the Ptolemaic to the early Roman period. Although we are not sure how many sulfur beads were actually made and how their different forms were arranged when strung, they were most likely made for funerary use, as a broad collar, which usually comprises more than 300 beads.

The use of sulfur in ancient Egypt is little known because of insufficient archaeological and textual finds. Furthermore, except of the AENET specimens, no isotopic study of sulfur beads has so far been undertaken elsewhere. We hope to provide more insights into ancient Egyptian sulfur beads when the opportunity arises to find additional parallels.



Figure 13. Two possible reconstructions of the sulfur bead necklace: a single string and a broad collar.

ENDNOTES

1. This paper is based on two articles published in Japanese: Yamahana and Akiyama (2017) and Yokoyama et al. (2019).

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