Synchrotron Radiation Analysis on Ancient Egyptian Vitreous Materials

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Abstract
Ancient Egyptian vitreous materials, namely faience and glass, share the same elemental composition. But they appear to have originated separately. Faience objects appear as early as the Predynastic period, and glass was introduced from Mesopotamia during the New Kingdom. These faience and glass objects were not of daily use, rather they were regarded as religious symbols or luxury status goods. Most of the products were coloured blue, but we see an increased use of other colours during the New Kingdom (c.1550-1069BC). This tendency corresponds to the period of both territorial and political expansion of Egypt. A non-destructive SR-XRF experiment at Spring-8 was conducted last winter, aiming to determine the regional trait of elemental composition by examining the pattern and ratio of rare earth elements. As a result, we could observe some distinctive rare earth elements that may indicate regional variation.

1. Ancient Egyptian vitreous material – faience and glass
In the context of ancient Egyptian history, the category of vitreous material applies to both faience and glass. Faience is one of the oldest "high-tech" ceramics, composed of siliceous core and glassy coating (Figs 1, 2, and 3). It was named after the ceramics made at Faenze in northern Italy, since the blue-green colour resembles to the Egyptian counterpart. The ancient Egyptian called this, 'the shining (one)'. In the ancient Egyptian art and belief, colour plays an important role. Especially blue or green is a symbol of life, rebirth and regeneration. It added sacredness to the object to which the colour was applied. A number of divine figures, amulets, sacred animal figurines were thus made of blue-green faience.

Fig.1 Faience Ushabti, by courtesy of Institute of Egyptology, Waseda University
On the other hand, glass was probably derived from glaze production and began to be used independently. It was then introduced to Egypt and made explicitly at Pharaohs’ workshops. Some loanwords were applied to call this object in Egypt, or it was also referred in ancient Egyptian as ‘stone of the kind that flows’. Glass was one of the luxurious products of royal use, and Pharaoh would occasionally grant to their courtiers. The technique of shaping and decoration, as well as colouring was well established by the time glass reached to Egypt during the reign of Tuthmose III (ca.1504-1450BC). Egyptians began to make their glass products shortly after, probably from the reign of Amenhotep III (ca.1386-1349BC). They favoured to make small cosmetic containers, amulets, inlays, and cups. So there seems to be a distinct functional difference between faience and glass; faience had an intrinsic symbolic value associated with Egyptian ritual and notion of the afterlife, whereas glass was rather regarded as luxurious goods, substituting precious stones for the privileged people.

2. History of Ancient Egyptian Faience and Glass

The first glazed objects appear from c.5500BC both in Egypt and Mesopotamia in the form of beads. During the early dynasties of Egyptian history, small figurines of gods and animals, as well as ornamental tiles and beads were made. Most of these early objects were of blue-green, resembling turquoise.

It was during the 18th dynasty and later that the multi-coloured faience production was in boom. A number of such beads to make floral garlands or other products have unearthed from many sites in the vicinity of the royal residences at Malkata, Amarna and Qantir. The artifacts of high quality such as delicate cosmetic jars, inlayed tiles, openwork rings were manufactured for the members of the royal family. Such prosperity in artifact production corresponded with the widespread power of the Egyptian New Kingdom. During the early period of the New Kingdom, King Tuthmose III expanded Egypt’s territory to the maximum from modern Sudan to Syria. Egypt could then practice her power over the conquered regions, enjoying her subjects paying various tributes to their master. Egypt obtained a fair amount of gold from the south, and various objects such as horses, chariots, gold, silver, lapis lazuri, Syrian ivory, metals, metal ores, glass, wood planks, as well as the knowledge of new technologies from the north.

Glass manufacturing was one of the new technologies that were introduced to Egypt from the north. The first glass was invented around 1700BC, originating most probably in the kingdom of Mitanni, the kingdom that occupied the vast area of northern Mesopotamia. Then the manufacturing technique spread southward into Egypt during the 15th century BC. Perhaps the technique of constructing the glass-fusing kiln which could yield higher temperature was also introduced. The new technique of making glass and traditional faience production merged and reached to the most sophisticated state during the New Kingdom. But after the fall of the New Kingdom, Egypt could no longer hold a vast territory, and even her own land was segmented (Third Intermediate Period: c.1069 – 500BC). The Glass production rapidly declined, and it did not revive until the Late Period (c.500 – 332BC). The Late Period was the time of final struggle for independence against Persians. Faience, glass and many aspects of Egyptian objects display a revival of traditional culture, but gradually incorporated into Graeco-Roman world.
3. Ingredient and Manufacture of Faience and Glass

There are three techniques of making faience; Application, Cementation, and Efflorescence. Application is to apply a glazing powder or slurry to the faience core. Sometimes drip lines or brush marks that indicate the use of this technique are clearly visible.

Cementation is also known as ‘Qom technique’ since the faience objects made by this technique are still being produced at Qom, Iran. First, unglazed dry faience core is buried in a glazing powder which partially melts on heating. The powder reacts with the surface of the quartz core and so glazes it, though powder not in contact with it remains unaffected[1]. In these first two techniques, the core and glaze materials are made separately. The core is usually the open-textured silica, and the glaze is an alkaline calcium silicate mixture with addition of colourant. The last, efflorescence technique is unique in combining the glaze mixture in the core. Water-soluble alkali salts probably in the form of natron or plant ash are mixed with the siliceous core. The mixture of these ingredients is moistened and formed into a desired shape. Then, in the process of drying, the salts migrates to the surface of the object to form an efflorescent bloom. When fired in high temperature, usually above 1000 °C, this layer melts and fuses with the fine quartz, copper oxide or lime to create a glassy coating. The latter two methods are regarded as ‘self-glazing’.

Glass, on the other hand, was mostly shaped using the ‘core technique’. This is a process for making vessels by building glass around a removable core[2]. Other techniques, such as Mold Pressing and Cold Cutting were rather uncommon, but certainly in use from the 2nd millennium BC. Blowing and Mold Blowing were developed from the end of the 1st Century BC.

The elemental composition of faience surface glaze and glass is similar, and sometimes indistinguishable by the appearance of the final product. Especially from the New Kingdom when the production of both faience and glass was closely connected, glass powder was occasionally added to faience glaze to enhance its quality. Both faience glaze and glass are composed of silica, lime and alkali (natron or plant ash). These ingredients were locally available. But Egyptians must have obtained the colouring minerals through a long distance trade, since they were unavailable along the Nile Valley. One of the most important colouring agents was copper. Copper was in use since the Late Predynastic Period and the systematic exploitation of copper in Sinai peninsula began from the Early Dynastic Period (c.3100BC-). Copper was thus readily available mineral for faience production since the beginning of the dynastic period. The Egyptians made blue and red faience and glass, knowing the difference in oxidized state of copper could make two distinctive colours. Other colours such as white, green, purple, amber, black, yellow, and their variations were made by adding antimony, lead, tin, iron, manganese, cobalt and other minerals into the basic soda-lime-silica batch[3]. Among these minerals, antimony, tin and cobalt are thought to have been very rare in Egypt. Instead, these minerals

were likely to have been imported to Egypt from the north. The number of cobalt containing faience and glass increased during the New Kingdom, when Egypt was one of the most influential nations in the Near East. But the ratio of cobalt-containing objects diminished as the New Kingdom collapsed. The same tendency is observable with yellow colourant, which is a combination of antimony and lead. The colour also thrived during the New Kingdom, but vanished shortly after. The colour variation of the period represents not only the technological sophistication of the workshops, but also Egypt’s both territorial and political influence over other regions in the Near East. The origin of such colouring minerals is not exactly known, but we assume that they were mostly mined at the fringe of the Taurus mountains, northern Mesopotamia. We may be able to trace down the specific region where the minerals were mined, through the continuous effort to accumulate the data of scientific analysis.

4. Elemental Analysis using Synchrotron Radiation*  
A number of elemental researches have been conducted to clarify the regional traits of pottery, faience and other metal products. However, the ratio of major elements is more or less uniformed. Finding the regional variability with the major elements has been very difficult. In this analysis, we boldly set aside the major elements, but rather set our eyes on trace elements. We picked rare earth elements since the inclusion of those elements in minerals is affected by the geographical formation of the region. So, we expect to find the regional variability by analyzing the ratio and pattern of such rare earth elements. We examined approximately 45 samples including mostly of faience and glass beads of mid-14th century BC to 2nd century AD. Most objects we analyzed were the excavated samples from Iran and Egypt[4]. I have chosen the objects of two distinctive regions, to test our assumption that the geographical formation reflects in contents of rare earth elements.

X-ray fluorescence analysis is one of the scientific method by which the concentration of element can be measured. The method is widely used in the study of archaeological objects since it is non-destructive and comparably simple. We conducted our study using synchrotron radiation X-ray fluorescence (SR-XRF), the method which started to gain its popularity recently. Spring-8, where we undertook our experiment can provide $3.3 \times 10^{13}$ photons/s at 100 keV. This was the first time that the ancient faience and glass were irradiated by high energy X-rays (115 keV). Due to its high brilliance and energy, very small concentration of elements can be measured precisely.

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of fluorescence is obtained by the measurement system combined with a computer.

Figure 5 shows an X-ray fluorescence spectrum of a faience from Egypt. X-axis is the energy of fluorescence X-rays, and Y-axis is the counts. In this spectrum, Cu, Sn, Pb, Sr, Ba, La, Ce, Nd and Dy are detected.

Fig. 5 An XRF spectrum of Egyptian faience

Fig.6 shows the distribution of the faience with La/Ba peak ratio in X-axis and Ce/Ba peak ratio in Y-axis. In order to adjust the intensity decline of synchrotron radiation, we picked out La/Ba and Ce/Ba peak area ratios. La and Ce are rare earth elements. Though their chemical characteristics are similar, their ion radiuses are different because charge in nuclei can not be obstructed by inner shell electrons.

5. The result of analysis

Our analysis is still in progress, and we cannot, at this stage, draw any definite conclusion of the last experiment. We could, however, observe the effectiveness of trace element analysis. As shown in Fig.6, the distribution plots of Egyptian and Iranian faience cores seem to form two major clusters. These clusters appear to correspond to these two distinctive regions. Further accumulation of data may be able to draw clearer clusters. Among the samples from Iran, we had several fragments of Egyptian Udjat-eye amulet (see Fig.3 right, as an example). The art style of the amulet is clearly Egyptian, so they were thought to have been imports from Egypt. As a result of our analysis, most of them fell in the cluster of Iranian objects. Only one example belonged to Egyptian cluster. Though not certain at this stage, this outcome may indicate an existence of an Egyptian craftsman outside of Egypt, producing the Egyptian artifacts. More analyses on such objects which have been regarded as imports may help to gain the internationalization of the area, as well as the extent of the ancient trade network.

The experiment also yielded the compositional trend change in accordance with time. We detected the increased use of lead in faience glaze and glass. Our Iranian samples dating 5-4th Century BC contained a fair amount of lead compared to those samples of 10th Century BC. Perhaps sometime between these periods, people discovered that adding lead gives a great merit to vitreous material. It increases fluidity, thinness, and brilliance. Adding lead also lowers melting temperature, thus displaying better cost performance. The presence of lead was also obvious in the Egyptian samples, dating 2nd Century AD, while the New Kingdom samples contained very little. We expect to undertake more SR-XRF analyses in order to seek the compositional trend change.

Our SR-XRF study achieved to present the potential new data for further scientific analysis on archaeological objects.