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ELEMENTAL ANALYSIS BY FIELD-EMISSION SCANNING ELECTRON MICROSCOPE OF ANCIENT GLASS BEADS SAMPLE FROM PULAU KALUMPANG ARCHAEOLOGICAL SITE, (PERAK, MALAYSIA)

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ABSTRACT

Continuous research and excavation lead to the discovery of the 2nd A.D. prehistoric settlement in the mangrove area located in Pulau Kalumpang, Perak, Malaysia. The findings encountered included pottery and earthenware, bones and abundance of various colors and shapes of glass beads. Glass beads are a handy archeological finding for its convenience size, portability and attractive material. An analysis using Field-emission Scanning Electron Microscope (FESEM) conducted on the seventeen glass beads samples. The samples are the combination of seven major colors and three shapes. The analysis resulted to a composition that high in silica (52.0% to 78.0%), aluminum (9.0% to 20%) and sodium (3.0% to 19.0%), a key feature of South East Asia's Indo- Pacific glass beads. The composition also shows no similarities composition to the glass beads from China, Europe and India. Different trace elements compared to the glass beads Sg Mas, Kedah pointed that the glass beads found was made in Pulau Kalumpang and the raw materials from broken glass were originated from Middle East.

Keywords: Pulau Kalumpang, Indo-Pacific glass beads, material composition, provenance, Middle East, trading networks

1. INTRODUCTION

Glass beads are the common artifact that can be found in archaeological site all around Malaysia such as Lembah Bujang in Kedah, Pulau Kalumpang in Perak, Johor Lama and Kota Tinggi in Johor and Santubong in Sarawak (see Figure 1). These sites are the prominent trading ports of protohistoric age and also acting as Indo-Pacific beads making centers (Ramli et al. 2011; Ramli et al. 2017; Ramli et al. 2018; Bakar et al. 2018). Beads are significant key to clarify trading networks, local skills, and international connection as well as technology and mastery distribution patterns. It also may lead the investigation of social strata and status where its existence may determine the social class of the body found in archaeological tomb or sites (Verhaeghe et al., 2014).

Bead is something that designed to be wear or hang as personal decorations or used for calculation such as on abacus or rosary or similar items that used as amulet, seal or weight or, any object identified as bead (Francis, 1989). It is a great artifact for their small sizes, portability, and appealing appearance making them a trading object of prehistoric time (Francis, 1990). Beads are made of various material including organic materials (shell, ivory, bones, and coral), stones and glass. They are usually used as an ornament or for important rituals.

Glass is a non-crystallized amorphous solid used widely in various shape. The main substance of glass is silica, a main chemical composition for sand. Despite of that, glass is a difficult material to be put in exact classification due to its chemical composition, raw material and manufacturing method. Glass can be made from other material apart from silica. It also can be made through vapor deposition beside of melting method and may be produce from organic and non-organic material.

Glass bead is divided into two types, based on the method of making, drawn or wound. Wound is the glass bead which is made by twirling the heated glass whereas drawn is a method where the glass is pulled, cut into smaller segments and gone through the heat to cast off the rough edges (Francis1992). This type of beads is detectable from their materials and the bubbles or any other trapped substance will align accordingly to the drawn direction. Glass bead can be divided based on the original places where the raw sources may determine the element of the beads itself (Francis 1988-1989). There are few glass beads that have been associated by original places and element analysis such as China glass beads which consists high lead (Pb), low aluminum in Arikamedu's beads (Ramli et.al., 2011), where Middle East or Roman-Hellenistic-Byzantine have low lead and no barium in its element analysis (Lamb 1965, 36)

Indo-Pacific glass bead is small, drawn, monochrome, opaque or translucent glass bead. It was believed that the Indo-Pacific beads was first made in Arikamedu, India around 250 B.C. to 250 A.D, where there is a port called Ptolemy's Poduca Emporium, an advance and renown port existed for five decades (Francis 2002). The port was abandoned around 3rd A.D. due to a barbarian attack by people of Kalabras. Due to the that period, the people of Arikamedu has long gone to other few safe places including Sri Lanka, Thailand and Vietnam where they and their skills are moving all around the surrounding area.

The theory of the beads as indicator for economic distribution, technology and skill mastery transfer was proven by the scientific study which shown that the glass bead manufacturing skills was spreading from Arikamedu, India in 250 B.C. to 250 A.D, followed by Mantai, Sri Lanka from 1st or 2nd A.D. to 10th A.D, Klong Thom, South Thailand from 2nd to 6th A.D, Pulau Kalumpang or Kuala Selinsing in 2nd A.D. (Ramli et al. 2009; Ramli et al. 2012, Ramli et al. 2016)) and Oc-eo or Go Oc Eo, Vietnam from 2nd to 7th A.D. The movement of the Indo-Pacific glass bead manufacturer continued with Sungai Mas, Kedah, Malaysia from 6th to 11th A.D., Sating Pra, South Thailand in 7th A.D. to 10th A.D. Indo-Pacific beads manufacturer in South Thailand moved again to Takua Pa, in 9th to 10th A.D. (Francis, 2002).

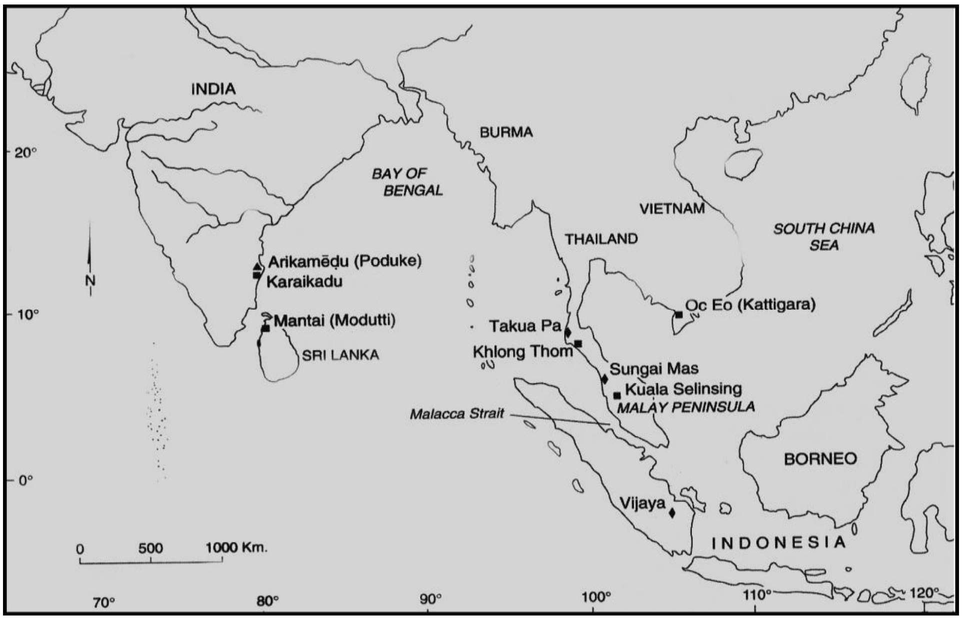

Figure 1: The location of Indo-Pacific beads manufacturer (Francis 2002)

The international connection existed in Pulau Kalumpang and the status as one of the important supplier ports at that time is not something absurd when the area was populated by the experienced and skilled civilization (see Figure 2). It is shown by the findings of the deep-sea fish bones, semi-precious stone beads such as carnelian, agate, amethyst, quartz as well as pottery and earthenware amongst the artifacts. Semi-precious stone beads were believed to be originally from India (Evans 1932, Wales 1940) meanwhile the ceramic found in Pulau Kalumpang was Persian ceramic and glasses made in Middle East (Nik Hassan Shuhaimi & Abdul Latif, 1988) and stone pottery from Yueh Dynasty (Evans, 1932).

Archaeological research and expedition in Pulau Kalumpang started and reported since 1928 by Evans (1932) regarding the discoveries of various artifacts such as glass beads, earthenware, human bones and jewelries. Amongst artifact found was abundance of assorted colors and shape of glass beads. Glass beads that found and collected at Pulau Kalumpang are known as Indo-Pacific glass bead which means, small drawn monochrome glass bead (Francis 1992) and made in first in India, and subsequently later in Sri Lanka, Malaysia, Vietnam and Thailand. More studies were conducted in 1936, 1956 and 1963 (Nik Hassan Shuhaimi & Abdul Latib, 1988). In 1987, one more excavation was made until 1989 and continued in 2008 (Mohd Aziz Ngah, 2008). In the latest excavation a culture strata and an absolute dating result has been obtained and indicated that the archaeological sites existed since 2nd A.D. (Ramli et al., 2009; Ramli et al., 2012)

Pulau Kalumpang is located in Kuala Gula, Bagan Serai, Perak. It is the first archaeological site where the 1800-year-old prehistorical remains are found in mangrove forest unlike other archeological site where the prehistoric were found in the caves. This fact is proven by the fact that there are a non-swamp plant existed in the area (Nik Hassan Shuhaimi & Abdul Latib 1988).

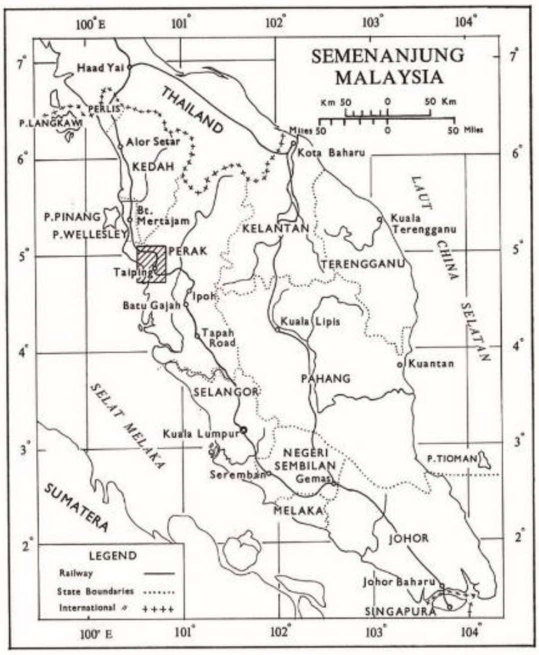

Figure 2: Location of the study area

Lies in the Quartener area, Pulau Kalumpang consists of Holocene estuarine marine sediments from Gula Formation. Gula Formation is divided into five members, Bagan Datoh, Teluk Intan, Port Weld, Parit Buntar and Matang Gelugor. Lithologies for this formation are silt, mud, sand and gravel. The formation is a result of littoral, estuary and shallow sea deposited (Kamaludin,1990).

2. MATERIALS AND METHODS

The beads went through cleansing process where it was soaked with distilled water and left to dry (see Fig.3). The amount of the beads is recorded accordingly to spit. The beads are categorized by colors and shape since Indo-Pacific beads went through similar manufacturing process. Beads width, length and perforation length is recorded using calipers. All the beads are named by their respective spit and later change to shape and color for easy identification. After cleaning, recording and the physical characterization was completed, the samples with different shape and colors are selected to be analyzed with field-emission scanning electron microscope facility owned by Malaysia Nuclear Agency located in Bangi, Selangor, Malaysia.

Carl Zeiss Gemini SEM 500 has been used for the FESEM-EDS analysis. This equipment was chosen because of the abilities to get the data and mapping info with fresh sample without coating process. Hence, it would not damage the sample and the sample can be retained for future used or preservation purposes. The analysis was conducted with Smart SEM for image acquiring using 10 kV accelerating volt and 8.5mm working distance. The image processing was run with SmartTiff. The sample size taken is 115μm x 86.3 μm with back-scattered electron. The result generated with Aztech Energy gave individual element and oxidized element weight and percentage as well as mapping images. For easy comparison process, the result included in this article is percentage oxidized element. Mappings of the element were in resolution size 1024 x 768 pixels.

Figure 3. Glass Beads Sample from Pulau Kelumpang

3. RESULT AND DISCUSSION

3.1 FESEM-EDS Analysis - General Observation

Seventeen samples were sent and it was chosen based on the size, shape and color. The composition analyses are shown in Table 1 for major element and Table 2 for trace element for each sample.

The sample name was given accordingly to these two characters, color and shape for easy recognition, where the name is combination of color and the shape of the sample. The alphabet in the bracket represent the shape of the glass beads where (s) is sphere, (c) is cylinder and (d) is for disc type.

Seventeen elements have been found from all seventeen samples where yellow (c) consists most of the element for both major and traces with thirteen of seventeen found. Orange brick (c) contains the entire major element and yellow (c) contains most of the trace element (four of seven observed). Blue (c) and translucent white (c) are the samples with least element, where they only have four major elements and no trace element all.

From general observation, it can be seen that all samples contain silica and aluminum in the element analysis result. Except for light orange (c) and white (d) which consist of high calcium content (79.90% and 69.90% respectively) all other samples contains high silica between 52.40% and 78.10% as well as high aluminum which amounted 12.10% to 20.60%. Aluminum percentage for those two samples with high calcium content is 3.10% for light orange (c) and 7.0% for white (d).

15 samples exhibit high sodium content, which is 0.3% to 19.20% compared to potassium reading, ranging from 0.7% to 2.40% with no detection of potassium content in light orange (c) and white (d).

The other two samples, black (s) and dark brown (s) show higher potassium reading, 5.50% and 1.20% with low sodium content which is 3.50% for black (s) and no sodium detected for dark brown (s).

Apart from light orange (c) and white (d) that contain high calcium and three other samples, blue (s), blue (c) and Translucent white (c) which show no detection of calcium, other 12 samples consist of calcium ranging from 1.0% to 12.90%.

Six samples show no detection of phosphorus, black (c), dark brown (s), translucent yellow (s), blue (c), white (s) and translucent white (c) while other samples show reading from 0.60% to 3.60%. Magnesium appear in most all of samples with reading from 1.0% to 4.90% except for seven samples, translucent yellow (s), blue (s), blue (c), green (c), white (s), white (d) and translucent white (c).

Iron and lead respectively appear in 4 samples. Both orange brick (c) and yellow (c) contains both element with iron content 3.70% and 0.60% and lead 1.30% and 1.80%. Iron also appear in black (s) with 6.60% and white (d) with 3.10%, meanwhile lead appear in both orange brick (s) and yellow (s) with 1.60% and 4.50% each. Orange brick (c) is the only sample with copper amounted 3.90%

For trace element, out of seventeen samples only five samples that do not consist of chlorine. The samples are dark brown (s), blue (s), blue (c), white (d) and translucent white (c) meanwhile sulfur appear in five samples, black (s), orang brick (s), light orange (c), yellow (c) and green (c). Titanium appears in four samples, black (s), dark brown (s), orange brick (c) and yellow (c). There are two samples with molybdenum, blue (s) with 1.60% and 1.40% in white (d). Manganese only can be detected in white (d) with 1.30% and Osmium in yellow (c) with 0.70%.

3.2 FESEM-EDS Analysis - Colors and Element

Pulau Kalumpang glass bead can be divided in to seven major color group which is black, dark brown, orange, yellow, blue, green and white. The glass beads are also divided by in to three shapes which are sphere, cylinder and disc. There are eight samples with sphere shape and eight samples with cylinder shape. The only glass bead with disc shape is white (d).

The differences can be seen if analysis of element composition for each glass bead with same color but different shape is compared side by side. The first in the list is black (s) in sphere shape and black (c) in cylinder shape. Both samples contain same 6 major elements out of 10 observed (silica, aluminum, potassium, sodium, calcium and magnesium) and both consists trace of chlorine in their trace element. However, while all the elements are almost similar in quantity it is clear to see that black (s) exhibited small differences for potassium and sodium which is 5.50% and 3.50% respectively while black (c) showed wide gap between those two elements with 1.80% for potassium and 19.20% for sodium. The difference between these two glass beads with same color but different in shape is the existence of extra elements in black (s) namely phosphorus and iron for major element and sulfur and titanium in trace element.

Next, glass beads with same color but different shape are dark brown (s) and dark brown (c). Both of the glass beads are sharing the same 5 elements out 10 major elements with no similarities in trace element. The major elements are silica, aluminum, potassium, calcium and magnesium. The differences for this glass bead is clear where dark brown (c) contains sodium with quite high value, 16.50% and 0.60% of phosphorus. For trace elements, dark brown (s) only contains 0.70% titanium while dark brown (c) only detected trace of chlorine.

Orange brick (s) and orange brick (c) both consist same seven major elements out of ten observed with almost similar numbers. While both contain trace of chlorine in trace element, orange brick (s) have 1.0% of sulfur and orange brick (c) contains 1.10% of titanium. The additional major elements for orange brick (c) are iron and copper both 3.70% and 3.90% respectively. For light orange, even though it falls under the same group with orange brick in term of color, and share six out of 10 major elements and the amount for phosphorus and magnesium is similar, the amount for the other four elements is totally different. Percentage of silica, aluminum, sodium and calcium is for 7.0%, 3.10%, 1.80% and 79.90% while the reading for the same element for orange brick (s) and orange brick (c) is 54.50% and 52.40%, 20.60% and 17.10%, 10.20% and 7.40% as well as 4.50% and 5.50% respectively. All three samples detected traces of chlorine; there are sulfur content in orange brick (s) with 1.0% and light orange with 1.20%.

For glass beads in yellow in color, there are three types of beads that can be jointly discussed. This is accordingly to the existence of a glass beads that share the same color and shape but possess translucent characterization where it allows part of the light to go through the substance. These three types of glass beads, yellow (s), yellow (c) and translucent yellow (s) consists of five same major elements (silica, aluminum, potassium, sodium and calcium) and all them detected trace of chlorine for trace element. Even though three of the glass beads contains sodium, it can be seen that that yellow (s) sodium content is lower, 4.70% compared to yellow (c) with 10% and translucent yellow (s) with 11.60%. From ten major elements, yellow (c) did not contain copper;

yellow (s) did not contain copper and iron; while translucent yellow (s) did not contain copper, iron, lead, magnesium and phosphorus. Trace elements shows that only yellow (s) contains sulfur, titanium and osmium. From this analysis, it shows that the elements responsible for opacity of the glass beads are among these three elements, lead, magnesium and phosphorus.

Blue (c) and blue (s) have same four major elements they are similar in percentage value (silica, aluminum, potassium and sodium). There are no traces of chlorine detected in both glass beads. The differences can be seen in the existence of phosphorus with 1.0% and molybdenum with 1.60%. For sample blue (s).

Glass beads in green (s) and green (c) consists four same major elements out of ten, which is silica, aluminum, potassium, sodium, calcium and phosphorus with similar percentage amount for both samples. Meanwhile, for green (s) and green (c) trace elements contains trace of chlorine. Both samples compositions were differentiate by the presence of magnesium (2.10%) in green (s) and sulfur (1.10%) in green (c).

Table 1: Content of major elements in Indo-Pacific beads in Pulau Kalumpang

Sample / Ox	Si	Al	K	Na	Ca	P	Mg	Fe	Pb	Cu
Black (S)	54.60	12.10	5.50	3.50	12.90	0.90	1.0	6.60	-	-
Black (C)	58.60	16.10	1.80	19.20	1.90	-	1.20	-	-	-
Dark Brown (S)	78.10	9.20	1.20	-	1.70	-	1.10	-	-	-
Dark Brown (C)	64.70	13.50	1.20	16.50	1.60	0.60	1.20	-	-	-
Orange Brick (S)	54.50	20.60	1.40	10.20	4.50	3.60	2.30	-	1.60	-
Orange Brick (C)	52.40	17.10	2.40	7.40	5.50	2.30	1.30	3.70	1.30	3.90
Light Orange (C)	7.00	3.10	-	1.80	79.90	1.20	4.90	-	-	-
Yellow (S)	66.70	13.40	1.70	4.70	5.90	1.80	1.00	-	4.50	-
Yellow (C)	60.00	15.80	1.50	10.00	4.30	2.30	1.10	0.60	1.80	-
Translucent Yellow (S)	71.80	14.30	0.70	11.60	1.0	-	-	-	-	-
Blue (S)	70.50	13.10	0.80	13.10	-	1.00	-	-	-	-
Blue (C)	74.90	14.40	1.10	9.60	-	-	-	-	-	-
Green (S)	63.00	20.50	1.20	5.10	3.90	3.60	2.10	-	-	-
Green (C)	72.40	16.20	1.10	3.20	4.20	1.30	-	-	-	-
White (S)	71.20	12.90	1.10	11.90	2.50	-	-	-	-	-
White (D)	13.60	7.00	-	0.30	69.90	2.30	-	3.10	-	-
Translucent White (C)	70.40	13.60	1.10	14.90	-	-	-	-	-	-

**(S)=sphere, (C)=cylinder, (D)=disc*

The glass beads in white is in the same group as yellow glass beads where it was divided into three; white (s) and white (d) which is opaque samples and translucent white (c) which have translucent characterization and allows part of the light to go through the substance. The compositions were totally different except for the three main component in major elements namely silica, aluminum and sodium. Even that so, the values for these three samples are different where while white (s) and translucent white (s) have similar percentage values (71.20% 70.40% for silica, 12.90% and 13.60% for aluminum and 11.90% and 14.90% for sodium respectively) white (d) have low silica (13.60%), low aluminum (7.0%) and even lower sodium (0.30%). Both white (s) and translucent white (s) contain 1.10% potassium. White (s) and white (d) exhibit the presence of calcium but the percentage content for white (s) is 2.50% and white (d) is 69.90%. There are presence of phosphorus (2.30%) and iron (3.10%) as well in white (d). Translucent white (s) contains no trace elements, white (s) only contain trace of chlorine and white (d) only contain molybdenum and manganese, 1.40% and 1.30% respectively.

Table 2: Content of trace elements in Indo-Pacific beads in Pulau Kalumpang

Sample / Ox	S	Ti	Mo	Mn	Os	Cl
Black (S)	1.0	1.10	-	-	-	Trace
Black (C)	-	-	-	-	-	Trace
Dark Brown (S)	-	0.70	-	-	-	-
Dark Brown (C)	-	-	-	-	-	Trace
Orange Brick (S)	1.0	-	-	-	-	Trace

Orange Brick (C)	-	1.10	-	-	-	Trace
Light Orange (C)	1.20	-	-	-	-	Trace
Yellow (S)	-	-	-	-	-	Trace
Yellow (C)	0.80	0.30	-	-	0.70	Trace
Translucent Yellow (S)	-	-	-	-	-	Trace
Blue (S)	-	-	1.60	-	-	-
Blue (C)	-	-	-	-	-	-
Green (S)	-	-	-	-	-	Trace
Green (C)	1.10	-	-	-	-	Trace
White (S)	-	-	-	-	-	Trace
White (D)	-	-	1.40	1.30	-	-
Translucent White (C)	-	-	-	-	-	-

**(S) =sphere, (C) =cylinder, (D) =disc*

Since there are two translucent glass beads; yellow and white, the comparisons shows that there are presence of calcium and trace of chlorine in translucent yellow (s) while both the samples consists the same other major elements. These might implied that the yellow hue in that sample originally from either chlorine or calcium.

From element observation, it can be seen that there are two samples with no physical character similarities but share similar composition. The samples are light orange (c) and white (d). Physically, light orange (c) is a light milky orange in color almost likely to be peachy tones with cylinder shape. Meanwhile, white (d) is an opaque white with disc shape. Both of the samples have high calcium content, 79.90% for light orange and 69.90% for white (d). The samples are containing low aluminum and phosphorus, 3.10% and 7.0% as well as 1.20% and 2.30% respectively. Both of samples content do not consist of potassium and only have low ration of sodium, light orange with 1.80% and white (d) 0.30%. the differences between these two is while light orang (c) have magnesium, sulfur and trace of chlorine, white (d) consist of iron, molybdenum and manganese. It is likely that they may share same basic mix before other substance added and became two different colors.

3.3 Mapping

EDS mapping is a technique where the software, AztechEnergy combined all the data and derived an elemental analysis in map where the element detected were colored in different colors. This method provide complete picture of composition for better observation.

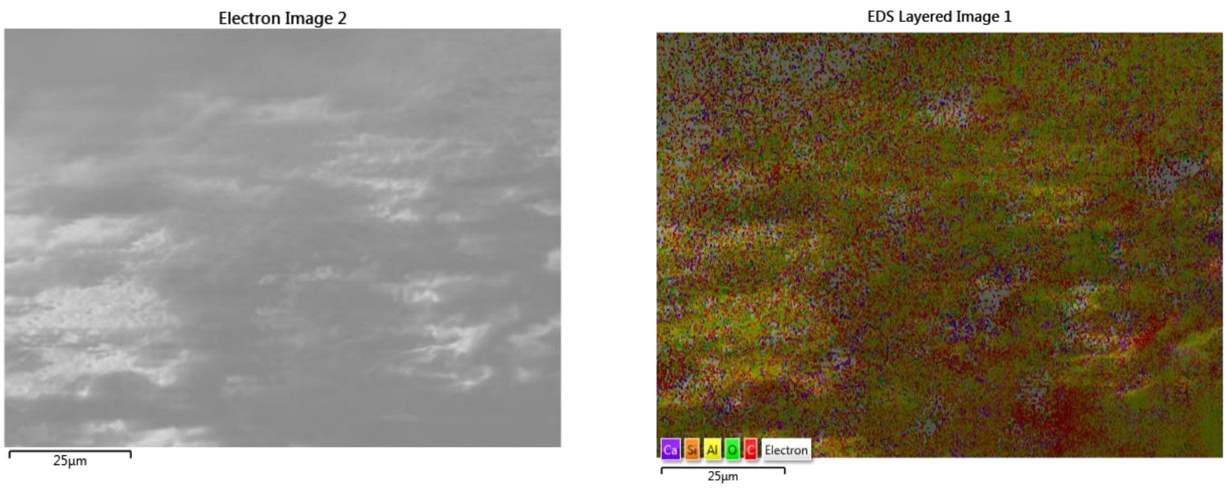

Figure 4: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Black (sphere)

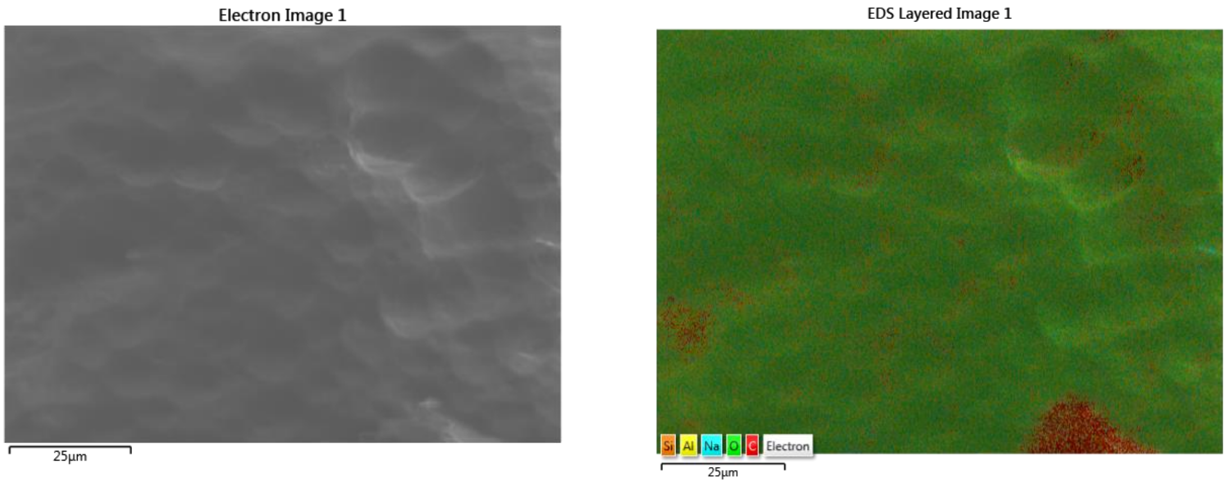

Figure 5: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Black (cylinder)

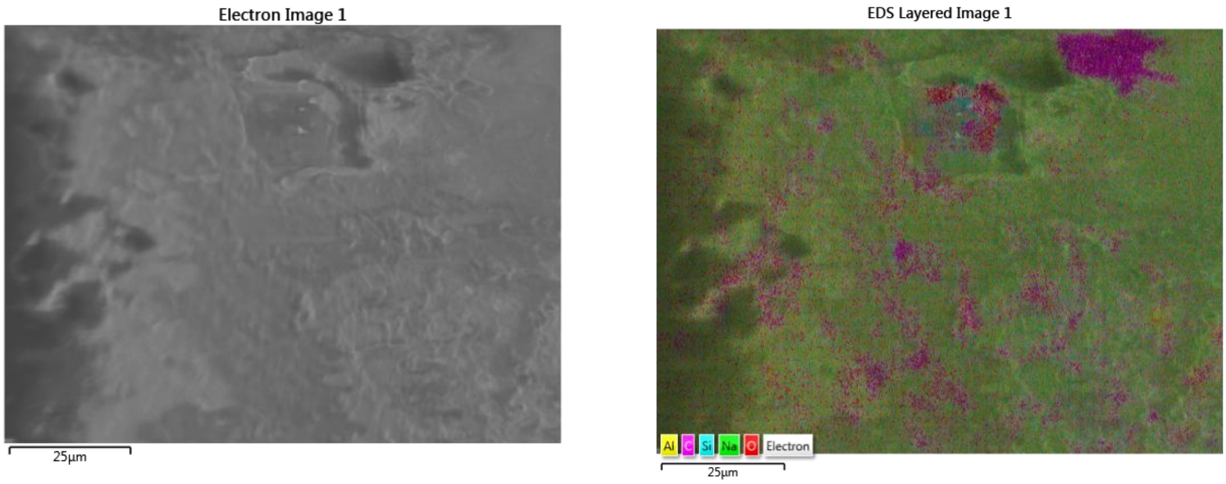

Figure 6: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Dark Brown (sphere)

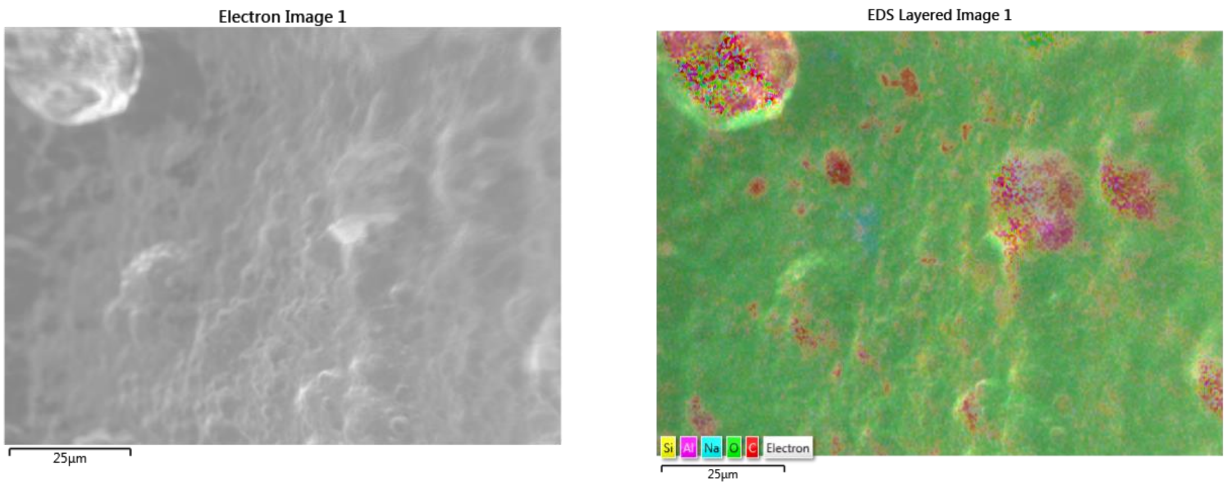

Figure 7: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Dark Brown (cylinder)

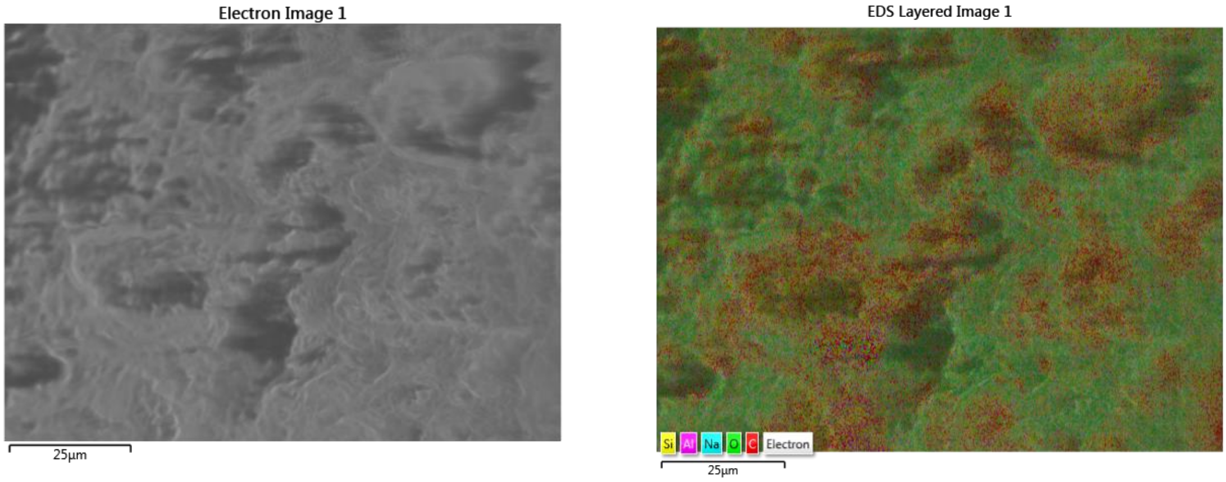

Figure 8: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Orange Brick (sphere)

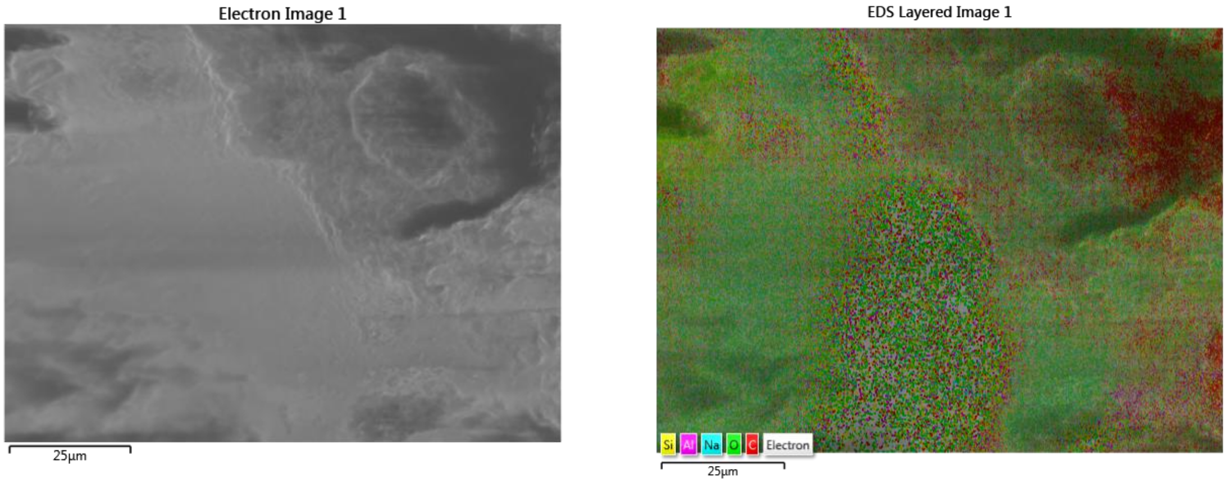

Figure 9: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Orange Brick (cylinder)

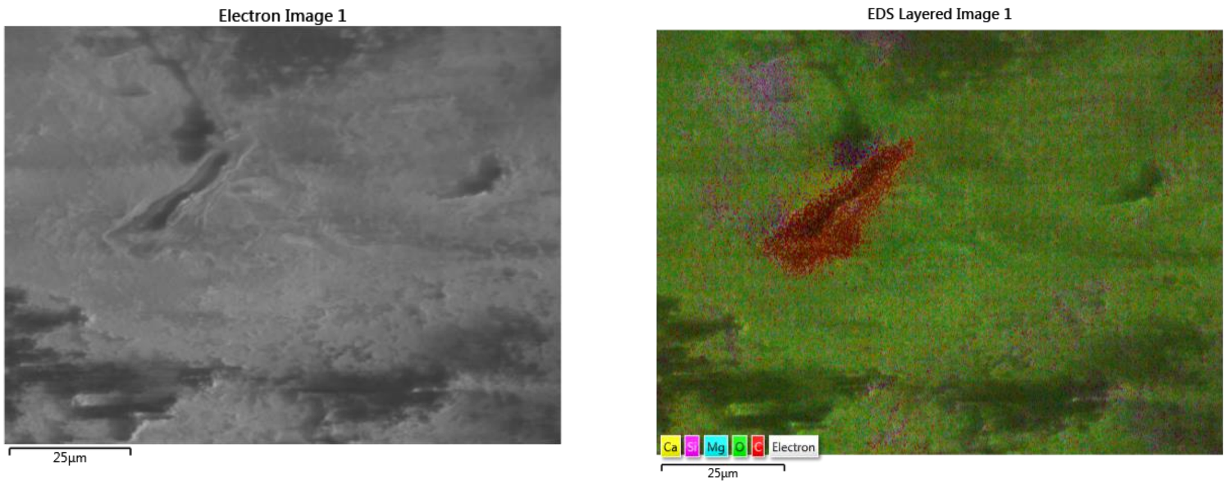

Figure 10: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Light Orange (cylinder)

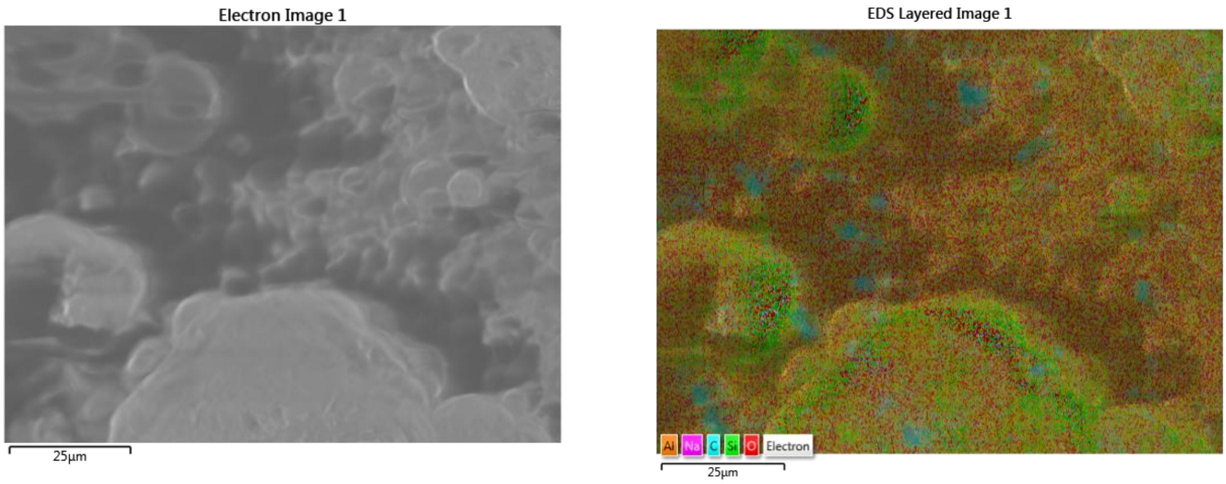

Figure 11: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Yellow (sphere)

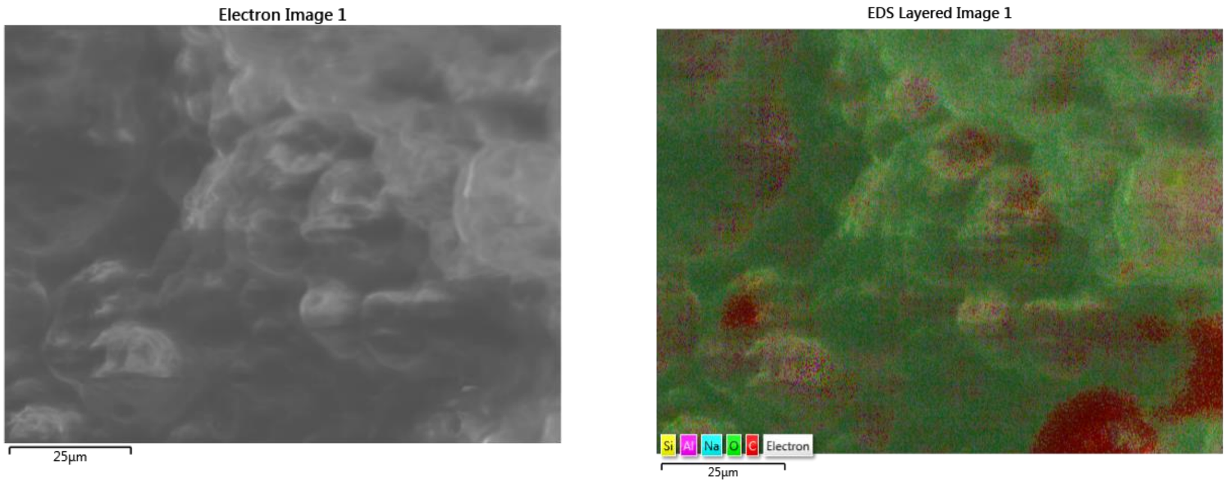

Figure 12: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Yellow (cylinder)

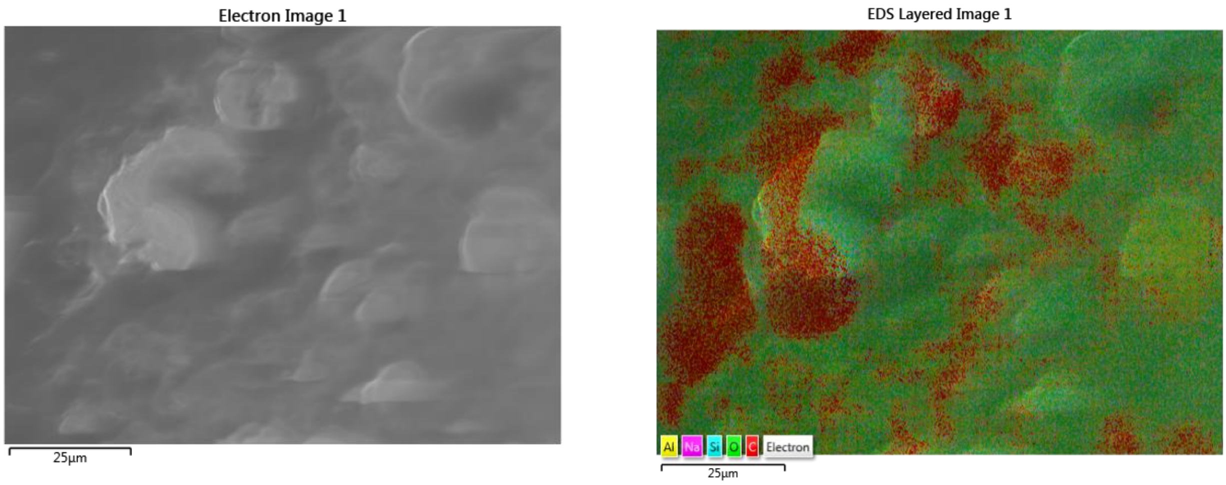

Figure 13: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Translucent Yellow (sphere)

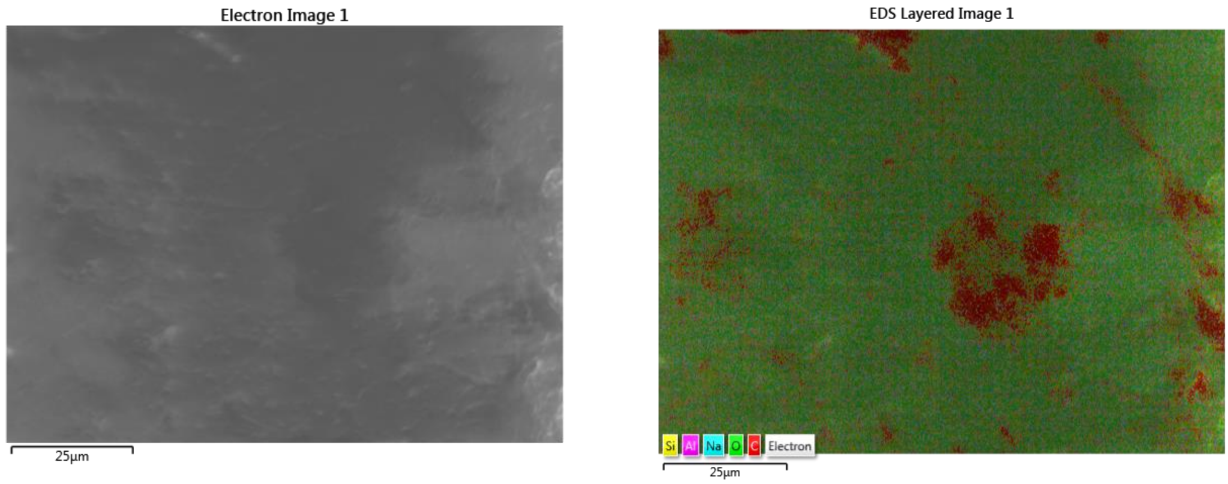

Figure 14: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Blue (sphere)

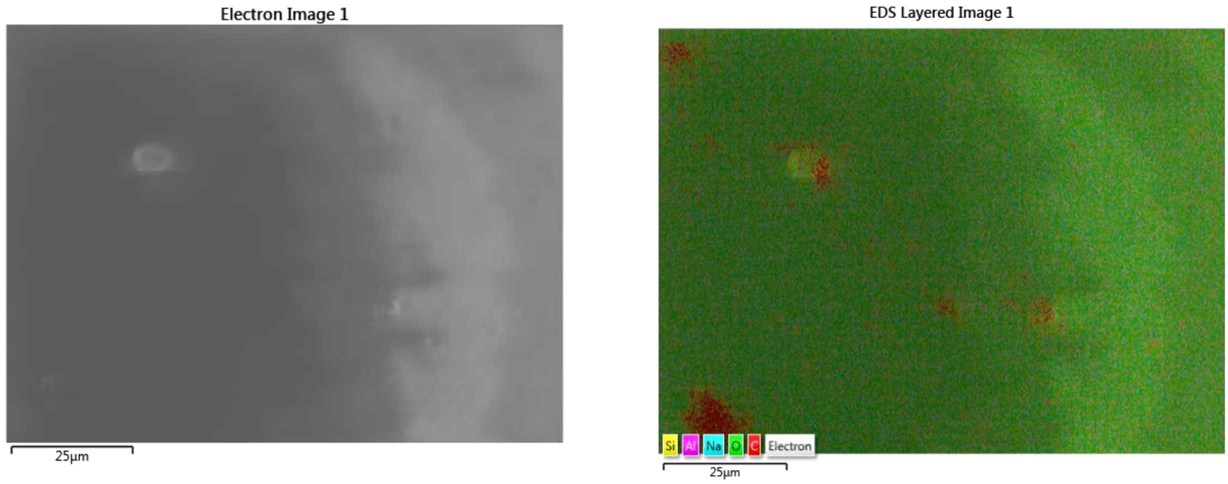

Figure 15: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Blue (cylinder)

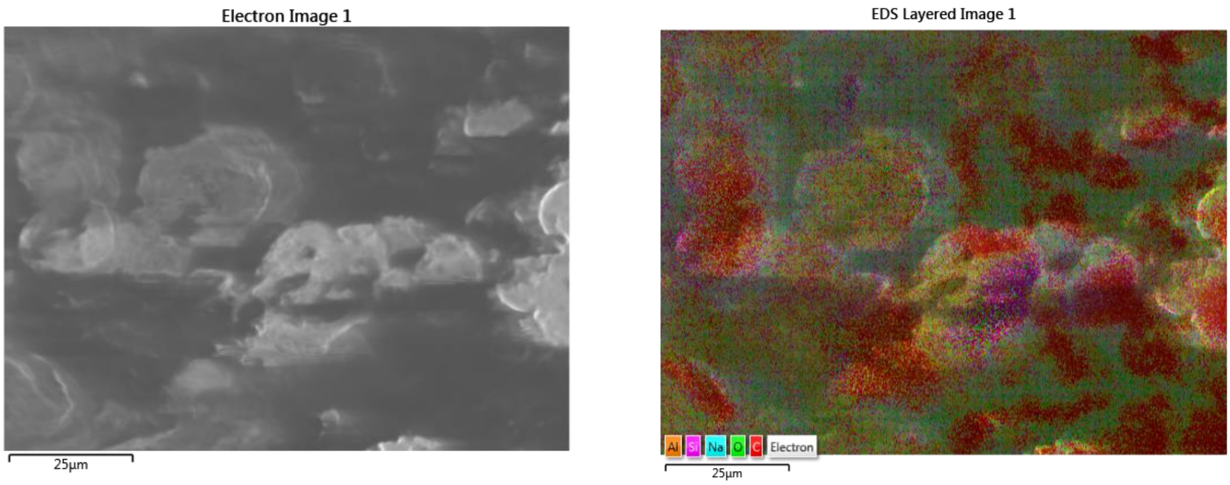

Figure 16: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Green (sphere)

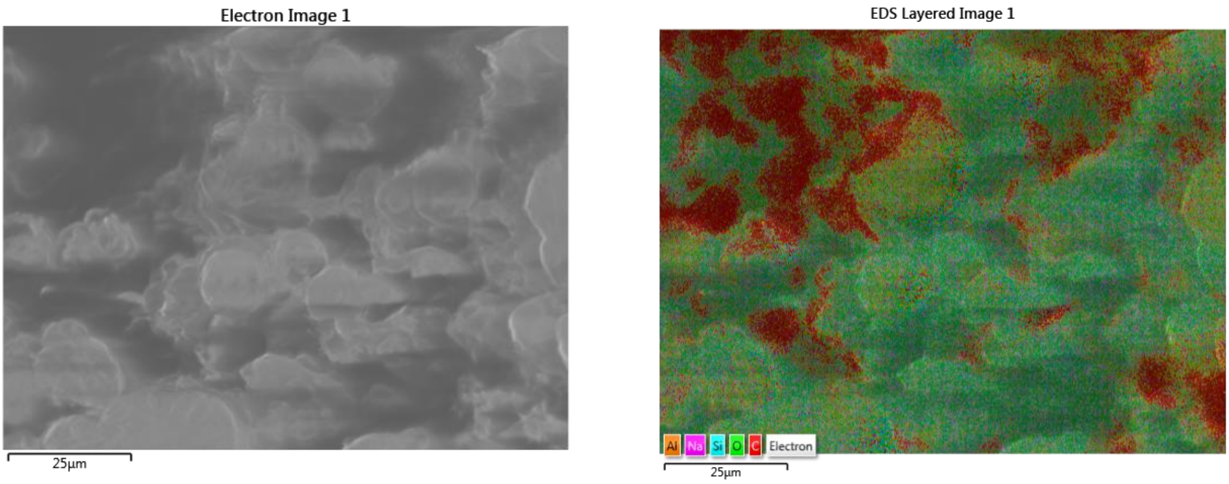

Figure 17: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Green (cylinder)

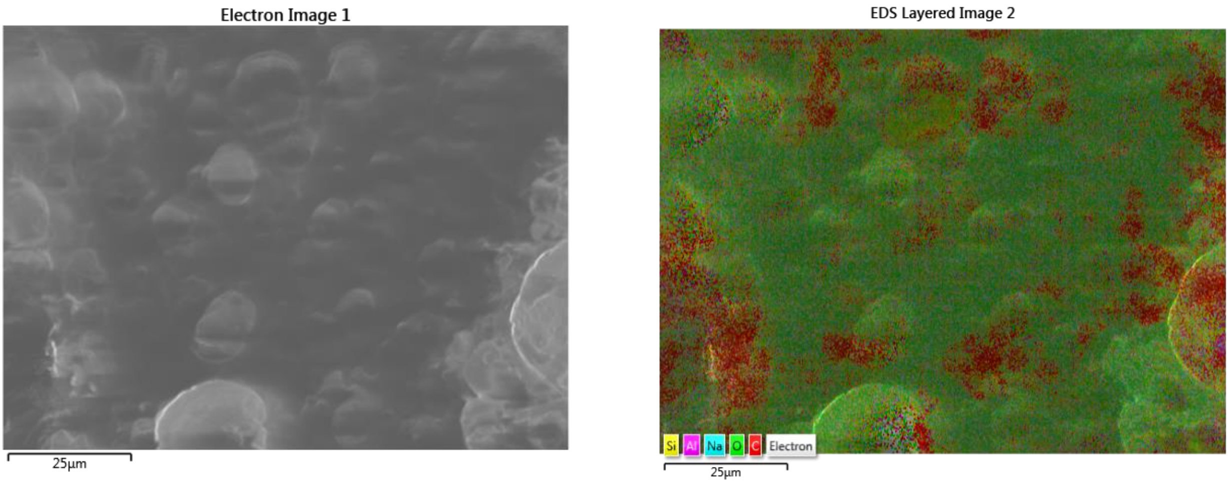

Figure 18: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: White (sphere)

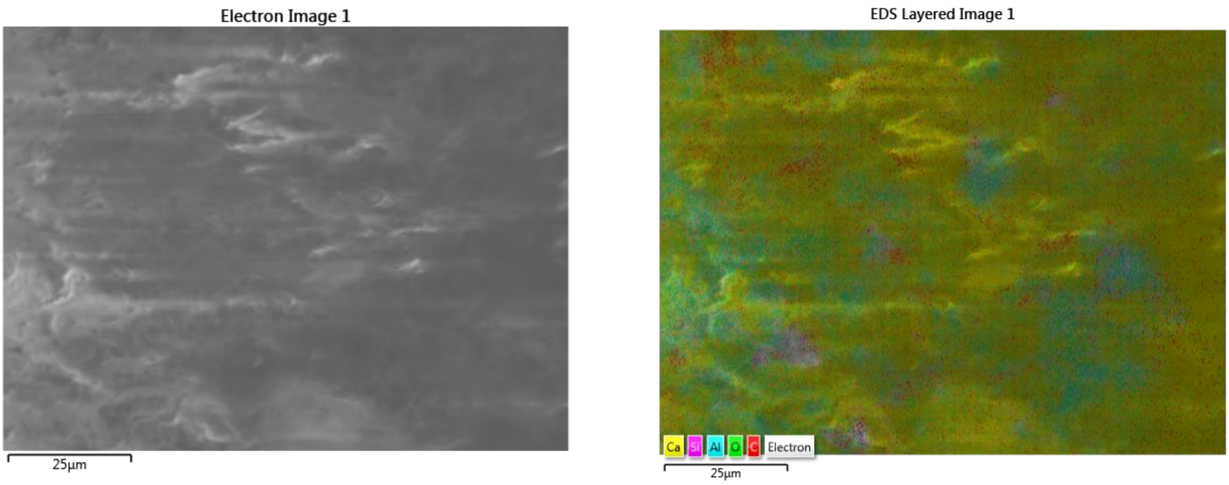

Figure 19: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: White (disc)

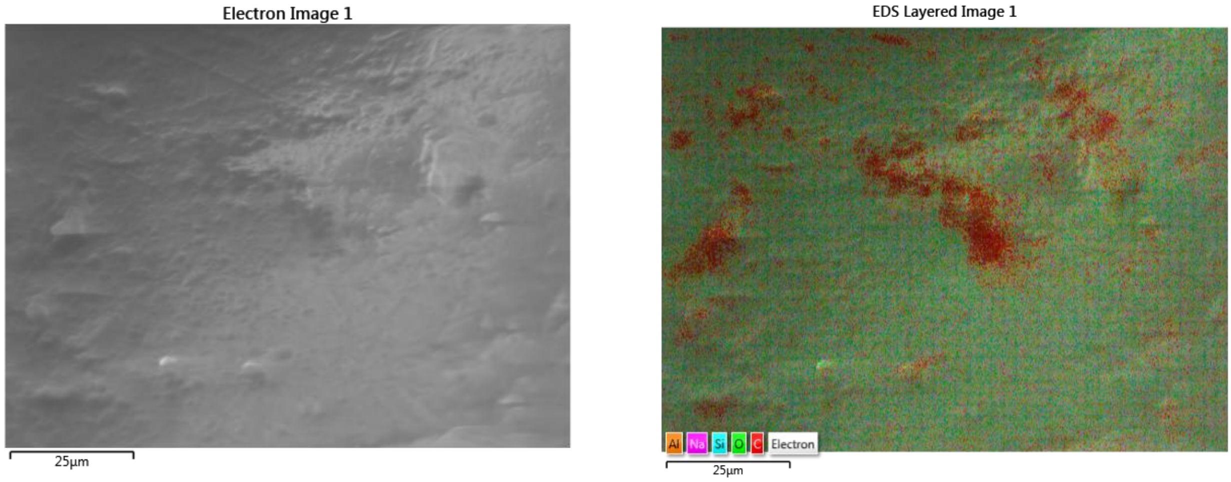

Figure 20: Electron image and EDS layered image of Pulau Kalumpang glass beads sample: Translucent White (cylinder)

4. CONCLUSION

Field-emission scanning electron microscope element analysis was chosen to analyze the glass sample from Pulau Kalumpang archeological site for its ability to conduct non-destructive analysis to study the composition of the glass beads and to provide mapping analysis where all elements and its distribution can be observed. This method is an excellent method for samples preservation especially archeological materials, where it is limited and valuable.

From the EDS analysis run to seventeen samples with different colors and shapes, it shows that there are two important contents that made the glass beads, silica and aluminum. These are the main elements for all glass beads found. Out of seventeen samples, fifteen samples contain between 52.0% to 78.0% for silica and 9.0% to 20% for aluminum. The other two samples content are high in calcium (69.0% to 79.0%) and low silica and aluminum.

There are also elements that appear in most of the samples with moderately high ratio. The elements are sodium, potassium, calcium, phosphorus, lead and magnesium with the value from 1.0% to 20%. And the trace elements are chlorine (appear in twelve samples), copper, titanium, sulfur, molybdenum and osmium.

Field-emission scanning electron microscope element analysis on the glass beads from Pulau Kalumpang show that the glass beads is high in silica, aluminum and sodium hence fulfilling the criteria of beads made from South East Asia. The glass beads consists of low lead content, no barium content, no cobalt-manganese-potash combination for blue glass beads and this eliminates the probabilities that the beads are from China, Europe or India.

Trace elements comparison with the nearest Indo-Pacific glass beads production center, Sg Mas, Kedah shows different composition which proposed that the glass beads from Pulau Kalumpang are made locally and and the raw materials from broken glass were originated from Middle East.

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