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Determining the mineralogical properties of Gua Sagu ancient pottery using X-Ray Diffraction and portable Raman spectrometer

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Abstract: This paper highlights the mineralogical properties of earthenware pottery shards excavated from Gua Sagu archaeological site in Kuantan, Pahang. Six earthenware pottery shards from different trenches and spits were selected for mineralogical analysis. For provenance study, soil samples collected from the site during the 1990-1991 excavations and clay samples obtained during the 2022 fieldwork from Sungai Batu, the nearest river to Gua Sagu were included in this study. Selected pottery samples, as well as the clay and soil samples, were analysed by means of X-Ray Diffraction (XRD) and portable Raman Spectrometry (pRS). The analyses reveal that the mineral compositions of the Gua Sagu pottery consist mainly of quartz, hematite muscovite, albite, microcline and kaolin. The same minerals were identified in clay samples from Sungai Batu while gypsum and hydroxyapatite were observed in soil samples from Gua Sagu. It can be concluded that the Gua Sagu pottery was composed of high-grade clay, and was sand-tempered and fired in an uneven atmosphere at low temperatures presumably around 650 °C or less. However, the Gua Sagu pottery was not made by using clay sourced from Sungai Batu. The pottery may have been a trade item brought in from elsewhere to be used for cooking or storage. A lesson from this study is that the portable Raman spectrometer is a fast and easy-to-use device for the spectroscopic characterisation of ancient pottery.

Keywords: Pottery, mineralogy, manufacturing, firing temperature, Gua Sagu

INTRODUCTION

Archaeological research in Pahang beginning in the late 1920s has uncovered several prehistoric sites containing earthenware pottery. These sites include Gua Tok Long in Gunung Senyum, Gua Kecil in Raub, Gua Sagu and Gua Tenggek in Kuantan, Bukit Komel, Bukit Karim, Jeram Koi and Nyong in Sungai Tembeling, and Gua Angin and Kota Tongkat in Jerantut, Pahang (Linehan, 1928, 1930; Evans, 1931; Dunn, 1964; Theseira, 1976; Adi, 1983, 1985, 1987, 1989, 2007; Suresh *et al*., 2020a&b). Although information on pottery from these sites is scarce, previous studies have shown that they are archaeologically significant and comparable to other prehistoric pottery found in Peninsular Malaysia. To date, only a few archaeological sites in Pahang have been chronometrically dated. Among them were Gua Kecil which had produced an early date of 4,800 +/- 800 B.P. while Gua Tok Long was dated by the thermoluminescence to 1,500 and 500 B.P. (Dunn, 1964, 1966; Mahat *et al*., 1998). Other sites, such as Bukit Komel, Bukit Karim, Jeram Koi and Nyong of Sungai Tembeling are yet to be dated (Suresh *et al*., 2020a).

The archaeological site of Gua Sagu, located in the Bukit Sagu massif of Kuantan, is considered one of Pahang's most important pottery sites (Figure 1). The site was jointly excavated by Universiti Sains Malaysia and the Department of Museums and Antiquities in 1990 and 1991 (Zuraina *et al*., 1998). Earlier the cave was visited by M.W.F. Tweedie somewhere in 1935 and a brief report on the archaeological work conducted at the site was published in British Raffles Museums in 1937. The archaeological assemblage from the 1935 excavation includes pottery shards and lithic artefacts in the form of flaked discoid tools, scrapers, and a broken ground implement (Tweedie, 1937). Details on the physical features and technology of earthenware pottery and other artifacts excavated from Gua Sagu in 1935 are unavailable.

Earthenware pottery recovered during the 1990-1991 excavations has been examined by Zuraina *et al.* (1998). However, only the physical characteristics of the pottery were described. The pottery was decorated chiefly with cord-marked and incised designs. Also several fragments of plain earthenware were discovered. There was no mention of pottery form and shape because the shards were small in size. A few broken pieces of flat-bottomed vessels with no ring base were encountered (Zuraina *et al*., 1998). Variations in the height and texture of the rim shards further indicate the presence of restricted vessels, which were suitable for

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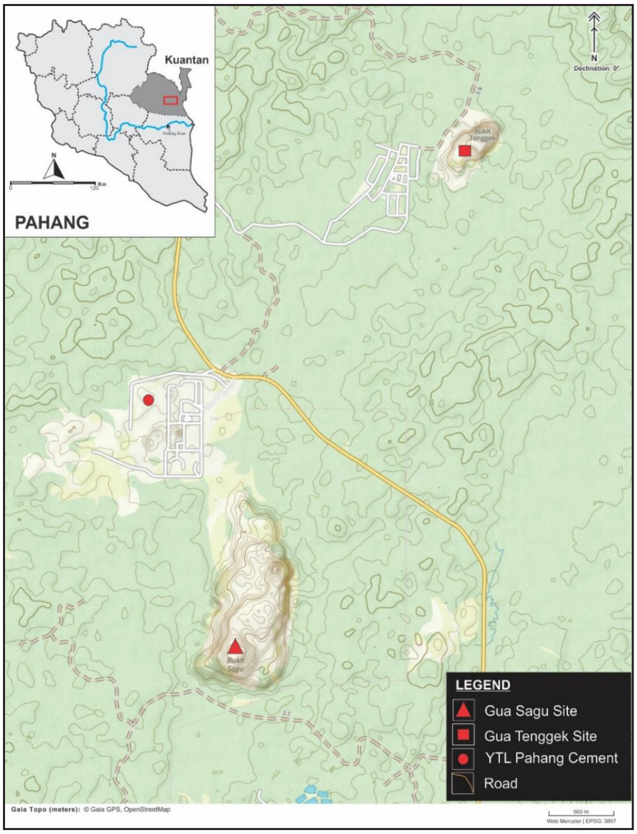

Figure 1: The location of Bukit Sagu dan Bukit Tenggek archaeological sites in Kuantan, Pahang.

Figure 2: The condition of the Bukit Sagu limestone hill in 2022. (Source: Photo courtesy of CGAR, USM).

storing food and water. Based on colour analysis, Zuraina *et al*. (1998) suggested that the Gua Sagu pottery was fired in an uneven firing atmosphere. Petrography of the Gua Sagu pottery was examined by Chia (1997). However, the mineralogical properties of the Gua Sagu pottery were largely unstudied. Recent visit to the site by a research team from the Centre for Global Archaeological Research (CGAR), Universiti Sains Malaysia (USM) found that future archaeological investigation at Gua Sagu will not be possible because a private firm is quarrying the Bukit Sagu limestone complex for cement manufacturing (Figure 2).

MATERIALS AND METHODS

For almost three decades, the Gua Sagu artefacts were housed at the Centre for Global Archaeological Research (CGAR), Universiti Sains Malaysia (USM), Pulau Pinang. Recently, the artefacts, including pottery specimens, were returned to Muzium Sultan Abu Bakar (MSAB) in Pekan, Pahang. Before being returned to the museum, the Gua Sagu pottery was re-examined, and several samples were selected for compositional analysis, the results of which are presented in this study.

Six (6) pottery shards, two each from trenches Z3, Z4 and Z5, were selected for mineralogical analysis. The samples were labelled S1, S2, S3, S4, S5 and S6. Pottery shards from other trenches (A4, A5, A6, X3 and Y3) were not included in this study because the results of the reclassification analysis showed that each of these trenches contained less than 8 shards, except for A5 which had 44 shards. Furthermore, permission was only granted by CGAR of USM to conduct compositional studies on pottery shards from trench Z3, Z4 and Z5. The two techniques used were X-Ray Diffraction (XRD) and Portable Raman Spectroscopy (pRS). S1, S3 and S5 were submitted for XRD analysis while S2, S4 and S6 were submitted for pRS analysis (Table 1).

X-ray Powder Diffraction (XRD) is used for the identification of crystalline phases (Sarhaddi-Dadian *et al*., 2015; Zeinab, 2018). In archaeology, this technique is used to identify components of lithics, metals, pigments and ceramics (Quinn & Benzonelli, 2018). It is also useful in characterizing parameters involved in the production of ceramics, such as firing temperatures (Papakosta *et al*., 2020; Moon *et al*., 2021).

To the best of our knowledge, in Malaysia, this is the first time that Portable Raman Spectoscopy (pRS) has been used to investigate the mineralogical phase of prehistoric pottery. pRS serves as a complement to XRD to identify the major minerals in archaeological objects. Recent studies have demonstrated that pRS can be a powerful

Table 1: List of Gua Sagu pottery samples selected for XRD and pRS analyses.

Sample	Trench	Spit	Type of analysis	Source
S1	Z3	4	XRD	CGAR, USM
S2	Z3	4	pRS	
S3	Z4	3	XRD	
S4	Z4	3	pRS	
S5	Z5	2	XRD	
S6	Z5	2	pRS	

Note: CGAR (Centre for Global Archaeological Research); USM (Universiti Sains Malaysia); XRD (X-Ray Diffraction); pRS (Portable Raman Spectroscopy) and effective tool for compositional analysis, imaging procedure and stratigraphy investigation (Rinaudo *et al.,* 2010; Jehlička *et al.,* 2017; Bersani *et al.,* 2019; Chiriu *et al.,* 2020). Moreover, this method grants short measurement times without any sample preparation. In pottery studies, pRS has a great potential in the analysis of coarse-grain and low-fired ceramics (Medeghini *et al.,* 2014; Petriglieri *et al.,* 2021). The characterisation of the mineralogical composition through this non-destructive technique also allows exploring the technology of ancient pottery. Therefore, the application of pRS in this study is believed to provide new information principally on the manufacturing process and firing condition of Gua Sagu pottery.

To determine pottery source and origin, a geological survey was done where soil samples from the cave (collected during the 1990-1991 fieldwork) and clay samples from two different areas of Sungai Batu, the nearest river to Gua Sagu (collected during the 2022 fieldwork) were tested using XRD and pRS. The soil sample was labelled GSa 95/YZX/S1 while clay samples were labelled C1 and C2. The results of the analyses were compared to Gua Sagu pottery composition to check if there were any similarities or differences. The outcome of the study is also believed to provide some information on the geological setting of the site during ancient times.

For XRD analysis, the samples were pulverized to <50µm grain size using a motorized grinding machine and was further grounded manually to a finer grain size of 20µm using an agate mortar. A Bruker D8 Advance (Germany) X-Ray Diffractometer was used to obtain diffractograms. The experimental parameters are as follows; Source of X-Rays: Kα, $\lambda = 1.54060$ Å, scanning range = 10° - 70° (2θ), scanning speed: 0.02° 2Θ/sec. A Portable Raman Analyzer Bravo by Bruker (dual beam configuration) was utilised for pRS analysis. The sample (pottery shard) was strapped to the aperture of the device using tape and analysed using pre-set parameters. The spectra obtained was analysed between 300 cm^{-1} and 1200 cm^{-1} fit for inorganic material. Three spots were selected for each potshard which include the core (cross-section) and the inner and outer surfaces (flat and stable). The spectrum was analysed using the OPUS 8.5 application by Bruker. Finally, the peaks and wavenumber of the spectra were compared to the open-source Raman spectra, X-ray diffraction and chemistry database for minerals chiefly RRUF™ developed by The University of Arizona. All these analyses were done at USM's Centre for Global Archaeological Research's Earth Material Characterisation Laboratory. No physical study of the pottery shards was carried out because the morphologies of Gua Sagu pottery have been studied and reported by Zuraina Majid and her team between 1992 and 1998 (Zuraina *et al*., 1998). However, a brief reclassification analysis was conducted to check on pottery shapes and forms.

Table 2: Mineral contents of earthenware pottery and soil from Gua Sagu.

Sample	Mineral content
S1	Quartz (SiO_2), Muscovite ($KAl_2Si_3AlO_{10}(OH)_2$), Albite ($NaAlSi_3O_8$), Microcline ($KAlSi_3O_8$)
S3	Quartz (SiO_2), Albite ($NaAlSi_3O_8$), Microcline ($KAlSi_3O_8$)
S5	Quartz (SiO_2), Kaolinite ($Al_2Si_2O_5(OH)_4$), Albite ($NaAlSi_3O_8$)
GSa 95/ YZX/S1	Gypsum ($CaSO_4 \cdot 2H_2O$), Hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$), Muscovite ($KAl_2Si_3AlO_{10}(OH)_2$), Microcline ($KAlSi_3O_8$)

RESULTS AND DISCUSSION

XRD analysis reveals that the Gua Sagu pottery shards consist mainly of quartz, albite and microcline (Table 2; Figure 3-5). Muscovite was detected in S1 while kaolin was found in S5 (Table 2). According to previous studies by Chia (1997, 2003) and Asyaari (2002), clay, silica, feldspar and mica minerals are commonly observed in prehistoric

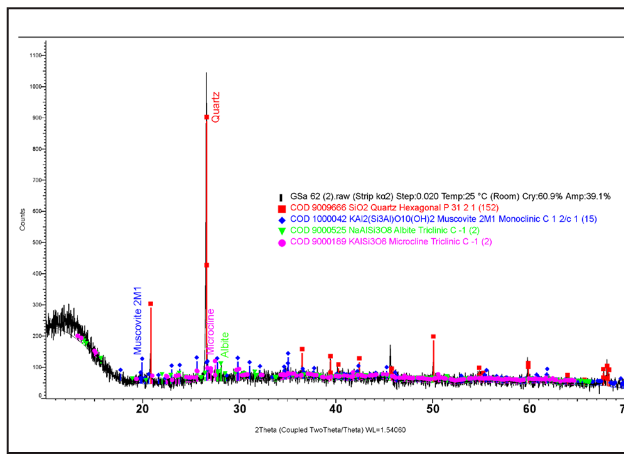

Figure 3: XRD spectrum of S1.

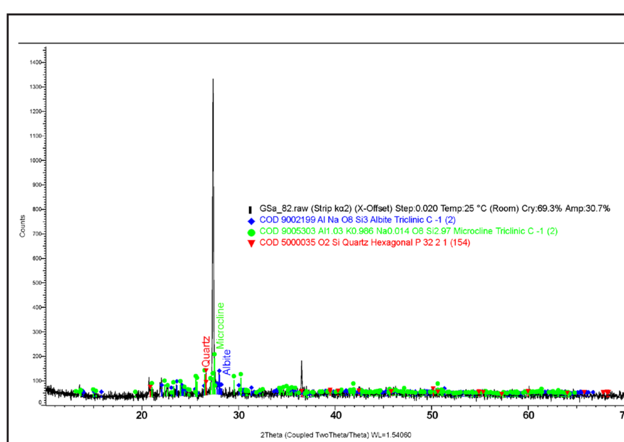

Figure 4: XRD spectrum of S3.

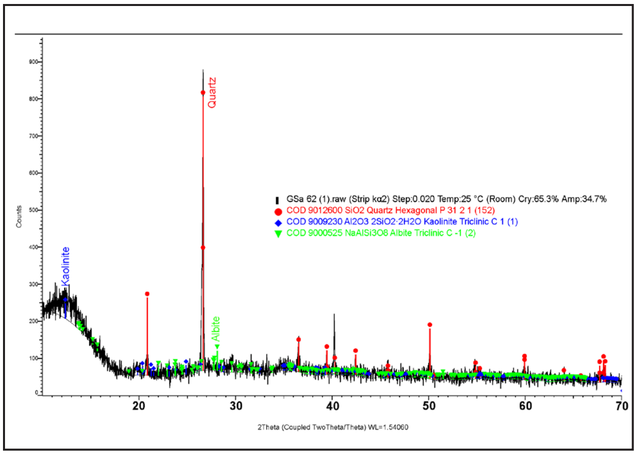

Figure 5: XRD spectrum of S5.

pottery of Peninsular Malaysia. In addition, ethnographic studies have unveiled that local clay used for traditional pottery-making in Malaysia is naturally rich in kaolin, quartz and other clay minerals, similar to that found in prehistoric pottery (Suresh, 2011, 2017). The presence of gypsum and hydroxyapatite, particularly in the Gua Sagu soil sample, showed that the cave soil had been mixed up with minerals like chalk, most likely from the cave walls, and food remains in the form of bones and shells (Table 2). Gypsum ($CaSO_4 \cdot 2H_2O$) is a sulfate mineral composed of hydrated calcium sulfate and commonly found in limestone caves and rockshelters. Also found in the Gua Sagu soil is hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$), a calcium phosphate biomaterial, is indicative of the presence of bone and tooth material. No human remains were found during USM's excavation at Gua Sagu in 1990 and 1991. Since the soil from Gua Sagu has been contaminated, it is an inappropriate candidate for a comparative study of pottery.

pRS analysis of the Gua Sagu pottery shards identifies minerals which are identical to those observed by XRD. In addition, several other minerals were identified by pRS, for instance, hematite and kaolin in S2, S4 and S6. Bulk-sample XRD powder diffraction is not suitable for identifying and differentiating clay minerals such as kaolinite, smectite, chlorite and micas due to ambiguous and overlapping peaks, and weak crystallinity, and is incapable of identifying amorphous phases (Kumari & Mohan, 2021). For this reason, pRS was used to determine the presence of clay and other amorphous minerals.

Based on the results of XRD and pRS analyses, this study confirms that the Gua Sagu pottery was made by using high-quality clay. The presence of mica (muscovite) and feldspar minerals (albite and microcline) indicate the use of good clay in the production of Gua Sagu pottery (Nelson, 1984). The presence of quartz indicates that the clay used in the production of Gua Sagu pottery is rich in sand, which may have been added to the clay as a temper. Recent studies on prehistoric pottery from other sites in Pahang, for instance those excavated in Ulu Tembeling, revealed the use of sand temper (Esnita *et al*., 2024). Therefore, it is very likely that sand was used as inclusion in the manufacturing of Gua Sagu pottery with the purpose of improving the workability and quality of the pottery (Suresh, 2011, 2017). Thin-section petrography reveals the presence of sand sized quartz, primarily rounded to sub-rounded, indicating the sand was not pounded before being added to the pottery (Centre for Global Archaeological Research, 2023). Sand could have been procured from nearby riverbank and sieved before being mixed to the clay, which is common practice among traditional pottery-making communities in Malaysia and Southeast Asia (Suresh, 2011, 2017), indicating that those who were involved in the production of Gua Sagu pottery must have been skilled artisans as they knew where to source raw materials to make pottery. They were also probably good at clay preparation and were cognizant of the clay-to-sand ratio and the use of other additives, in making pottery.

A few samples (S2, S4, S5 and S6) contain traces of kaolin. This strongly suggests that the Gua Sagu pottery was fired at low temperatures, possibly between 450°C and 650°C. This is because at a firing temperature of 650°C or above most clay minerals in the pottery will be transformed into other amorphous or crystalline phases (Velraj *et al*., 2010; Suresh & Nasha, 2023). When fired, at high temperature, kaolin loses weight due to the release of water and transforms into metakoalin (Hansen, 2024). In addition, mica (in this case, muscovite, which is detected in some samples) disappears on firing at around 600 – 700 °C (Zuliskandar *et al*., 2011). These low firing temperatures indicate that the Gua Sagu pottery was subjected to open-firing. The pinkish grey to grey surface colour of several Gua Sagu pottery shards indicates that they were fired in an uneven atmosphere. Fuels used for pottery firing may have consisted of dried firewood, split bamboo and coconut fronds, and such resources as might have been gathered from Bukit Sagu or its surrounding areas. At present time, however, these resources are not present because a major part of Bukit Sagu and its neighbouring areas have massively been cleared for palm oil cultivation.

In terms of production locality, the pottery could have been produced at Gua Sagu or elsewhere. Pottery found at Gua Sagu could have been made by potting communities living near Bukit Sagu like Gua Tenggek or Sungai Tembeling in Jerantut, Pahang. There is also evidence of pottery trading and exchange between the interior and coastal communities during the Neolithic Period (Suresh, 2011, 2017). A similar case has also been reported in the Nenggeri Valley of Kelantan where pottery was traded into the Ulu Kelantan region during ancient times (Shafiq *et al*., 2021). Patterns of decoration are not very helpful for determining provenance because, in the case of the Gua Sagu pottery fragments, they were limited to several simple designs consisting of cord-marked and incised patterns, made using carved wooden paddles and sharp pointed tools. Based on the results of reclassification and morphological analysis of pottery shapes and forms, the Gua Sagu pottery was used for practical purposes, for instance, for storing water and food. In addition, the pottery may have been used for cooking since soot marks have been identified (Zuraina *et al*., 1998). All this implies that the Gua Sagu pottery vessels were utilitarian, consisting mainly of containers and cooking pots used for food preparation and water storage.

Analysis conducted on clay samples obtained from Sungai Batu and the results revealed the presence of quartz, hematite, kaolin, muscovite, albite and microcline (Table 3; Figure 6). This suggests that clay from Sungai Batu is suitable for the manufacture of pottery. However, photomicrographs of clay samples from Sungai Batu revealed the existence of quartz sand grains with sharp, angular edges and abrasions, whereas quartz grains in Gua Sagu pottery were rounded to sub-rounded in shape (Centre for Global Archaeological Research, 2023). To further verify this, 2D and 3D microscopy (Ahmad Syahir *et al*., 2023) verified that quartz sand grain morphology and grain size in the Sungai Batu clay were significantly different from that observed in the Gua Sagu pottery fragments, clearly indicating that the Sungai Batu clay was not employed to manufacture Gua Sagu pottery. The limited quantity of pottery shards and the lack of evidence of pottery-making activity in the cave also suggests that pottery was not made at Gua Sagu. At the time, the site was most likely used as a temporary or seasonal campsite by the hunting and gathering groups. In addition, the discovery of a considerable amount of faunal remains in an archaeological context and hydroxyapatite in the cave soil support the idea that the site was used for short-term habitation.

Table 3: Results of the Raman analyses of soil and clay samples from Gua Sagu.

Portable RAMAN Analysis			
Sample	**Sampling Area (GPS coordinate given)**	**Wavenumber (cm^{-1})**	**Mineral Identification**
Clay sample (C1)	(3°58'18.0"N 103°09'05.7"E)	401, 464, 510, 810	Quartz SiO_2
		587, 702, 748, 903	Muscovite $KAl_2(ALSi_3)O_{10}(OH)_2$
		335, 410, 432, 464, 510, 748, 793, 914	Kaolinite $Al_2Si_2O_5(OH)_4$
		401, 1110, 1124	Microcline $KAlSi_3O_8$
		401, 410, [overlap], 478, 633, 645, 761, 810, 979	Albite $NaAlSi_3O_8$
		410, 498, 614	Hematite FeO_2
Clay sample (C2)	(3°58'29.5"N 103°08'47.1"E)	402, 464, 509	Quartz SiO_2
		414, 588, 698, 748, 900	Muscovite $KAl_2(ALSi_3)O_{10}(OH)_2$
		396, 433, 464, 638, 748, 794	Kaolinite $Al_2Si_2O_5(OH)_4$
		402, 519, 1109, 1120	Microcline $KAlSi_3O_8$
		328, 596, V407, 414, 504, 571, 630, 648, 760, 811, 974	Albite $NaAlSi_3O_8$
		410, 498, 614	Hematite FeO_2
Soil sample (GSa 95/ YZX/S1)	(3°58'37.0"N 103°08'25.9"E)	403, 465, 508, 808	Quartz SiO_2
		410, 498, 614	Hematite FeO_2
		589, 702, 747 900	Muscovite $KAl_2(ALSi_3)O_{10}(OH)_2$
		330, 403, 508, 645, 762, 977	Albite $NaAlSi_3O_8$
		317, 498, 620, 1007, 1144	Gypsum $CaSO_4.2H_2O$
		448,580, 589, 606, 852, 1030, 1042	Hydroxylapatite $Ca_{10}(PO_4)_6(OH)_2$

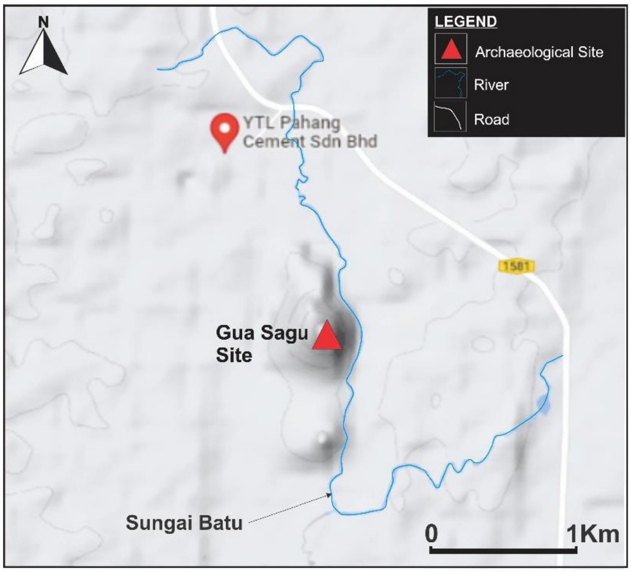

Figure 6: Clay samples obtained from Sungai Batu, the nearest river to Bukit Sagu limestone massif in Kuantan.

CONCLUSION AND RECOMMENDATIONS

Despite that only six pottery samples were analysed, the combination of XRD and pRS techniques have contributed new information on the manufacturing process and firing conditions of archaeological earthenware pottery excavated from Gua Sagu. Also, the study had shed some light on the origin of Gua Sagu pottery. This information is very essential because the findings can be used to check their connections with other prehistoric pottery found in and outside the state of Pahang, mainly in terms of production and technology. The portable Raman spectrometer has also proved to be a fast and easy-to-use device for spectroscopic characterisation of ancient pottery, particularly for detecting the presence of clay minerals with small particle size. Unlike XRD, the Raman spectrometer is useful in identifying amorphous materials and provides information on profile depth. The current study revealed that the Gua Sagu pottery was skilfully crafted utilising high-quality clay, sand-tempered but fired unevenly. The firing temperature ranged between 450°C to 650°C. Tools used for pottery manufacturing may consisted of carved wooden paddles, pebble stones and sharp pointed implements for decoration. In the case of Gua Sagu, the pottery was not manufactured on site, but was brought in as a trade item to be used for cooking and storing. This is in line with previous researchers' assumption on Gua Sagu pottery which could have been bartered with other communities living near the site (Zuraina *et al*., 1998).

In its current state, the Gua Sagu site offers very little potential for future archaeological work. The site has been badly disturbed due to human activities since the late 1990s. In addition, palm oil cultivation and quarrying activities in the area had affected the flow, sedimentation and ecosystem of the nearby rivers like Sungai Batu and Sungai Ramen. Compositional analysis of soil and clay from Bukit Sagu would not yield relevant data because they are no longer in-situ and are highly contaminated. As of now, further systematic sampling and scientific analysis of Gua Sagu pottery may be conducted to investigate its source and origin. Future research should use more advanced analytical methods such as X-ray fluorescence, scanning electron microscopy, inductively coupled plasma mass spectrometry, thermogravimetric and thin-section petrography analyses to yield more promising and convincing results on the technology, production and provenance of Gua Sagu pottery. A more detailed and comprehensive study on pottery specimens from nearby archaeological sites like Gua Tenggek in Kuantan and Sungai Tembeling in Jerantut should also be considered as this will provide valuable information and contribute to our understanding of Gua Sagu's ancient pottery-making and its relationship with other pottery sites in Pahang.

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AUTHORS CONTRIBUTION

All authors except for NRK were involved in the fieldwork at Bukit Sagu (Kuantan, Pahang) in 2022. At USM, ASZ, AFJ and SSKM carried out scientific analysis of clay, soil and pottery samples while SN, NRK and NKT prepared and edited the original draft of the manuscript. All authors have read and agreed to the published version of the article.

CONFLICT OF INTEREST

The authors have no conflict of interest to declare that are relevant to the contents of this article.

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