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X-Ray Diffraction and X-Ray Flourescence of Ancient Bricks of *Candi bukit pendiat* (Site 17), Bujang Valley, Kedah

[1]Zuliskandar Ramli, [1]Nik Hassan Shuhaimi Nik Abd. Rahman,
[1]Abdul Latif Samian and [2]Mohd Ambar Yarmo
[1]Institute of the Malay World and Civilisation (ATMA)
[2]School of Chemical Sciences and Food Technology, Faculty of Science and Technology,
the National University of Malaysia, 43600, Selangor

Abstract: *Candi bukit pendiat* (Site 17) is one of the temple sites that used bricks as the main construction material and based on Global Positioning System, Candi *bukit pendiat* (Site 17) located at N5 41 43.4 E100 25 21.3. Apart from bricks, laterite stones were also used as the basis of the construction's structure which is octagonal in shape that is a stupa. Based on relative dating of this site, it is proposed that it was built between the 7 to 9th centuries AD. At this site, bricks containing rice husks were also found. These rice husks were used as strengthening material or one of the rituals when building temples. This combine evidence shows that the Old Kedah Malay community had already practised the paddy cultivation system since the 7th century AD or earlier. This study will focus on material composition analysis of ancient bricks that were used to construct this temple of which the main purpose is to see whether the raw materials used to produce those bricks utilised local raw materials or not. This is because the usage of local raw materials was associated with brick making technology that was already mastered by the local community. Two analysis techniques will be used namely the X-Ray Fluorescence (XRF) and the X-Ray Diffraction Technique (XRD) in determining the content of the major and trace elements as well as the mineral content in the ancient bricks. The findings show that the major minerals contained in the ancient bricks of *Candi bukit pendiat* (Site 17) are quartz, muscovite and microline while the other minerals that exist are kaolinite. The mineral content and physical observation of the bricks indicate that the open burning technique was used to produce these bricks because of the presence of kaolinite in one of the samples (BP17 (xv)). The kaolinite content shows that the samples were baked at a temperature less than 550˚C. The content of the major and trace elements also shows that these bricks were produced from the same source and it is proposed that local raw materials were used in the production of the bricks and the nearest source that could be detected is at the Sungai Bujang basin. The involvement of the local community in producing the bricks should not be refuted and this proved that the knowledge transformation of the local community had already started since the 4th century AD based on archaeological context in Bujang Valley especially inscriptions finding.

Keywords: Bricks, bujang valley, *Candi bukit pendiat*, material composition, X-ray fluorescence, X-ray diffraction

INTRODUCTION

Candi Bukit Pendiat (Site 17), Bujang Valley, Kedah is one of the temple sites that used bricks as the main construction material. Based on Global Positioning System, Candi Bukit Pendiat (Site 17) located at N5 41 43.4 E100 25 21.3. Apart from the bricks, laterite stones were also used as the basic structure in the construction of the eight-shaped structure which is predicted as a stupa. This site was at first excavated by Quaritch-Wales in the year 1936 until 1937 (Quaritch, 1940). Then in the year 1976, Adi Taha from the Museum Department conducted re-excavations with the purpose of exposing the entire site. Based on relative dating of bricks size and structure of the stupa, it is believed that this site was built between the 7th to the 9th century AD (Quaritch, 1940; Nik Hassan and Othman, 1992). Among the important findings that represented the existence of local elements is the discovery of earthenware that were used as reliquary. When the exploration activities were carried out to obtain samples for material composition analysis, bricks containing rice husks were found where the husks functioned as a strengthening material for the bricks. These finding shows that the society at that time were already skilled at rice planting activities and this characteristic is the basic feature of the Malay civilisation in the Malay Archipelago (Coedés, 1968).

On this study, the objective is to determine whether bricks taken from Candi Bukit Pendiat (Site 17) are

Corresponding Author: Zuliskandar Ramli, Institute of the Malay World and Civilisation (ATMA), The National University of Malaysia, 43600, Selangor

locally made or not. It is important to carry out material composition analysis of the ancient bricks of this site because the analysis can determine the raw materials used to produce the bricks whether it locally made or not. For example, compositional analysis on ancient bricks of Candi Sungai Mas (Site 32/34), showed that the raw materials used were local raw materials and these raw materials were obtained from the Muda River and Bujang River basin (Ramli *et al*., 2012a) Data obtained from the subsequent brick analysis will be compared with the composition data of clay material around the Bujang Valley area, Kedah (Zuliskandar *et al*., 2002). Compositional analysis on artefact from Bujang Valley and other part of Malay Peninsula has been carried out before especially on monochrome drawn beads from Sungai Mas (Rahman *et al*., 2008; Ramli *et al*., 2011a, 2012b; Zuliskandar and Nik Hassan Shuhaimi, 2009) and pottery from prehistoric site (Chia, 1997; Ramli *et al*., 2011b; Zuliskandar *et al*., 2001) Analysis on monochrome drawn glass beads from Sungai Kedah showed that Sungai Mas produced their own monochrome glass beads from 6^{th} century AD until 13^{th} century AD (Ramli *et al*., 2011b; Zuliskandar and Nik Hassan Shuhaimi, 2009).

MATERIALS AND METHODS

A total of 17 brick samples were taken from the site of *Candi bukit pendiat* (Site 17) and then taken to the lab for cleaning and labelled with the names BP17 (i), BP17 (ii), BP17 (iii), BP17 (iv), BP17 (v), BP17 (vi), BP17 (vii), BP17 (viii), BP17 (ix), BP17 (x), BP17 (xi), BP17 (xii), BP17 (xiii), BP17 (xiv), BP17 (xv), BP17 (xvi), BP17 (xvii). Analysis was conducted to determine the mineral content in the ancient brick samples. Samples weighing 0.4g were refined and heated up for one hour at a temperature of 105°C and mixed until homogenous with the flux powder of a type of Spectroflux 110 (product of Johnson and Mathey). These mixtures were baked for one hour in a furnace with a temperature of 1100°C. The homogenous molten was moulded in a container and cooled gradually into pieces of fused glass with a thickness of 2mm and a diameter of 32 mm. The samples were of 1:10 dilution. Samples in the form of fused glass were prepared for analysis of major elements such as Si, Na, K,Ca, Fe, Al, Ti, Mn and Mg. Pressed pallet samples were prepared for analysis of trace elements such as As, Ba, Ce, Co, Cr, Cu, Ga, La, Nb, Ni, Pb, Rb, Th, V, Zn and Zr.

These samples were prepared by mixing 1.0g of samples together with 6.0g of boric acid powder and then pressure of 20 psi was applied by using hydraulic pressure equipment. The samples of fused pallets and pressed pallets were then analysed using Philips PW 1480 equipment. Samples in the form of very fine powder were put into the pellets (sample holder) and then analysed using the X-ray Diffraction instrument ((D500 Diffract meter SIEMEN).

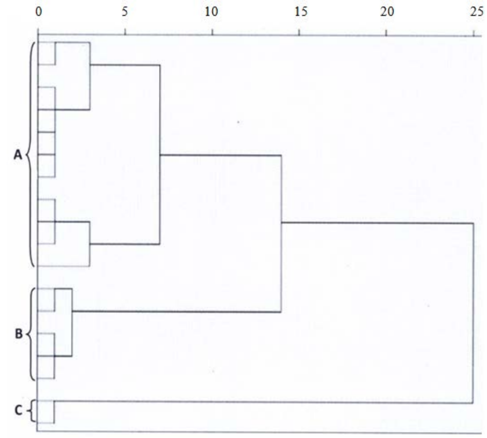


Fig. 1: Hierarchical agglomerative clustering of the bricks samples (TiO_2 and MgO) from *Candi bukit pendiat* (Site 17) using the ward's method

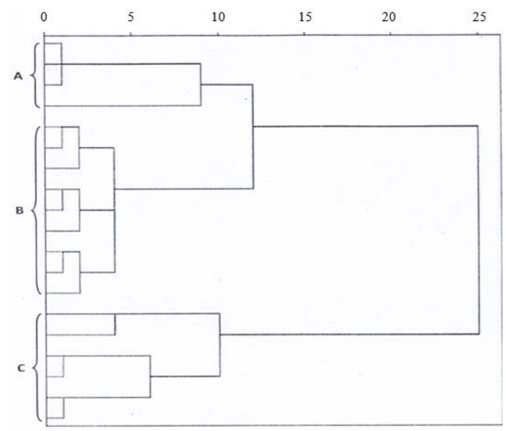


Fig. 2: Hierarchical agglomerative clustering of the bricks samples (lead and cooper) from *Candi bukit pendiat* (Site 17) using the ward's method

A scatter plot diagram of MgO versus TiO_2 and lead versus copper was then performed to demonstrate the differences among the group and analysed using Microsoft Excel software. The main purpose is to see the distribution of the samples in the group and subsequently to compare with the clay elements. Hierarchical Cluster Analysis (HCA) was applied to the chemical data from the four components, Titanium (TiO_2) and Magnesia (MgO), copper and leads, of all 17 brick samples in order to verify the presence of compositional groups of brick fragments differentiated by their probable major element sources. The measurement of distance used in the assignment rule was based on Ward's Linkage and Squared Euclidean Distance algorithm. The results are presented in the form of a dendogram (Fig. 1 and 2) showing in the graphical form the distance between the glass samples on the basis of their TiO_2 and MgO concentrations and lead and copper concentration (Johnson and Wirchen, 1992; Munita *et al*., 2000; Santos *et al*., 2009). The applicability of the analytical methods for the multi

Table 1: Major Elements concentration of certified references material, 315 fire brick

Element	This work (%)	Certified values (%)
SiO_2	49.36	51.20
TiO_2	1.28	1.23
Al_2O_3	40.09	42.40
Fe_2O_3	2.70	3.01
MnO	0.01	0.02
MgO	0.55	0.57
CaO	0.30	0.34
Na_2O	0.10	0.13
K_2O	0.54	0.52
P_2O_5	0.05	0.11

Table 2: Trace elements concentration of certified reference material, SY-2

Element	This work (ppm)	Certified values (ppm)
Ba	427	460
Co	11	11
Cr	4	12
Ni	3	5
Pb	42	80
Rb	213	220
Sr	261	275
V	50	52
Zn	245	250
Zr	255	280
Ga	26	28
La	22	88
Nb	9	23
Th	377	380
Ce	153	210
Y	146	130

elemental analysis by XRF of the glass beads is evaluated by the analysis of certified reference material, 315 Fire Brick (Calibration: G-FBVac28 mm) for major elements and certified reference material, SY-2 (Calibration: Trace Element P-20) for trace elements. The CRM was also used as the quality control material of the analytical procedure.

RESULTS AND DISCUSSION

The result of the Certified Standard Material (CRM) analysis as compared to the certified concentration of the elemental content in CRM is shown in Table 1 and 2. The results showed good agreement as compared to the certified value. Figure 1 shows the cluster of the samples based on TiO_2 and MgO component and the separation in three groups (A, B and C) with statistically significant of 7 of maximal linkage distance (Fig. 1). Figure 2 shows the cluster of the samples based on lead and copper component and separation in three groups (A, B and C) with statically significant of 10 of maximal linkage distance (Fig. 2). Its shows that Fig. 1 is more significant than Fig. 2 based on their compositional similarities.

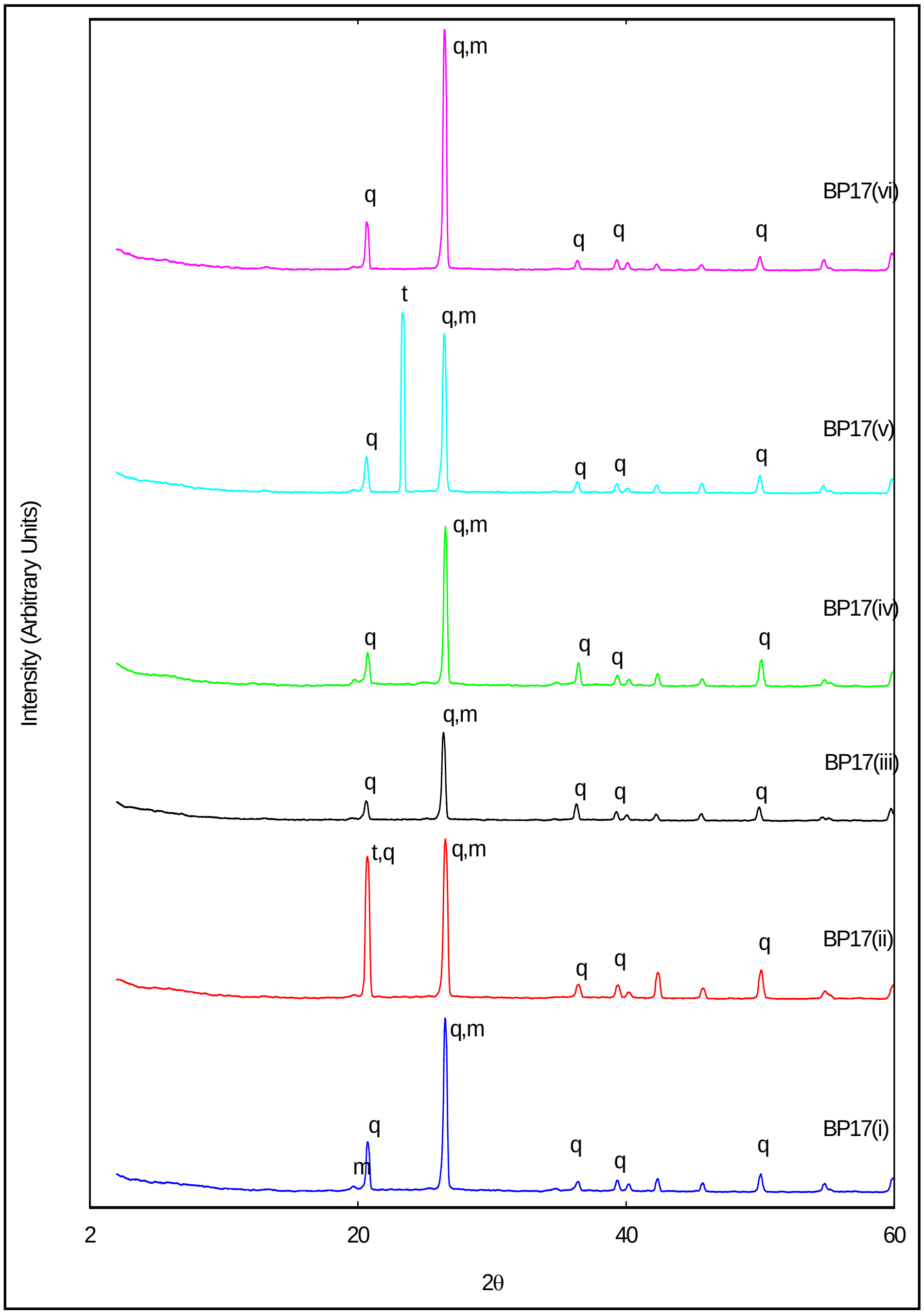
The mineral content contained in the ancient brick samples of Candi Bukit Pendiat (Site 17) can be referred to in detail in Table 3. Analysis shows that the

Table 3: Mineral content in the ancient brick samples of *Candi bukit pendiat* (site 17)

Location	Sample	Mineral content
Candi bukit pendiat (site 17)	BP17 (i)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (ii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (iii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (iv)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (v)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (vi)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (vii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 2M1
	BP17 (viii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 2M1
	BP17 (ix)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (x)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (xi)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M $KAlSi_3O_8$ Microcline
	BP17 (xii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (xiii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (xiv)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (xv)	SiO_2 Quartz $Al_2Si_2O_5(OH)_4$ Kaolinite 1Md
	BP17 (xvi)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (xvii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	BP17 (xviii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M

Table 4: Major element content of the ancient bricks of *Candi bukit pendiat (*site 17)

Sample	Dry weight (%)									
	Si	Ti	Fe	Al	Mn	Ca	Mg	Na	K	P_2O_3
BP 17 (i)	72.30	0.74	1.74	23.59	0.01	0.05	0.26	0.07	0.68	0.25
BP 17 (ii)	77.59	0.63	2.21	17.52	0.02	0.06	0.17	0.05	0.33	0.18
BP 17 (iii)	77.43	0.63	3.50	17.17	0.01	0.04	0.16	0.05	0.34	0.19
BP 17 (iv)	73.30	0.72	1.62	22.41	0.02	0.04	0.28	0.07	0.68	0.21
BP 17 (v)	76.01	0.70	2.90	19.44	0.01	0.04	0.15	0.05	0.29	0.14
BP 17 (vi)	78.32	0.60	2.14	17.40	0.01	0.04	0.16	0.05	0.32	0.20
BP 17 (vii)	76.13	0.64	3.53	18.32	0.03	0.04	0.21	0.06	0.37	0.19
BP 17 (viii)	77.70	0.61	2.88	17.24	0.01	0.04	0.17	0.06	0.33	0.18
BP 17 (ix)	73.57	0.73	1.63	22.67	0.01	0.04	0.19	0.06	0.62	0.22
BP 17 (x)	76.71	0.63	3.19	17.74	0.02	0.04	0.22	0.06	0.42	0.17
BP 17 (xi)	75.24	0.70	4.06	18.98	0.01	0.04	0.18	0.05	0.35	0.17
BP 17 (xii)	78.89	0.58	2.24	19.70	0.01	0.03	0.14	0.06	0.32	0.17
BP 17 (xiii)	77.47	0.61	2.71	17.79	0.01	0.04	0.16	0.07	0.28	0.17
BP 17 (xiv)	75.97	0.62	0.34	19.15	0.02	0.03	0.21	0.06	0.29	0.15
BP 17 (xv)	78.90	0.59	1.98	16.74	0.01	0.04	0.14	0.06	0.33	0.17
BP 17 (xvi)	76.88	0.71	3.10	18.39	0.03	0.03	0.16	0.05	0.34	0.20
BP 17 (xvii)	75.36	0.67	3.80	19.02	0.01	0.04	0.20	0.05	0.35	0.22
BP 17 (xviii)	75.03	0.71	3.55	19.65	0.01	0.04	0.19	0.05	0.35	0.17

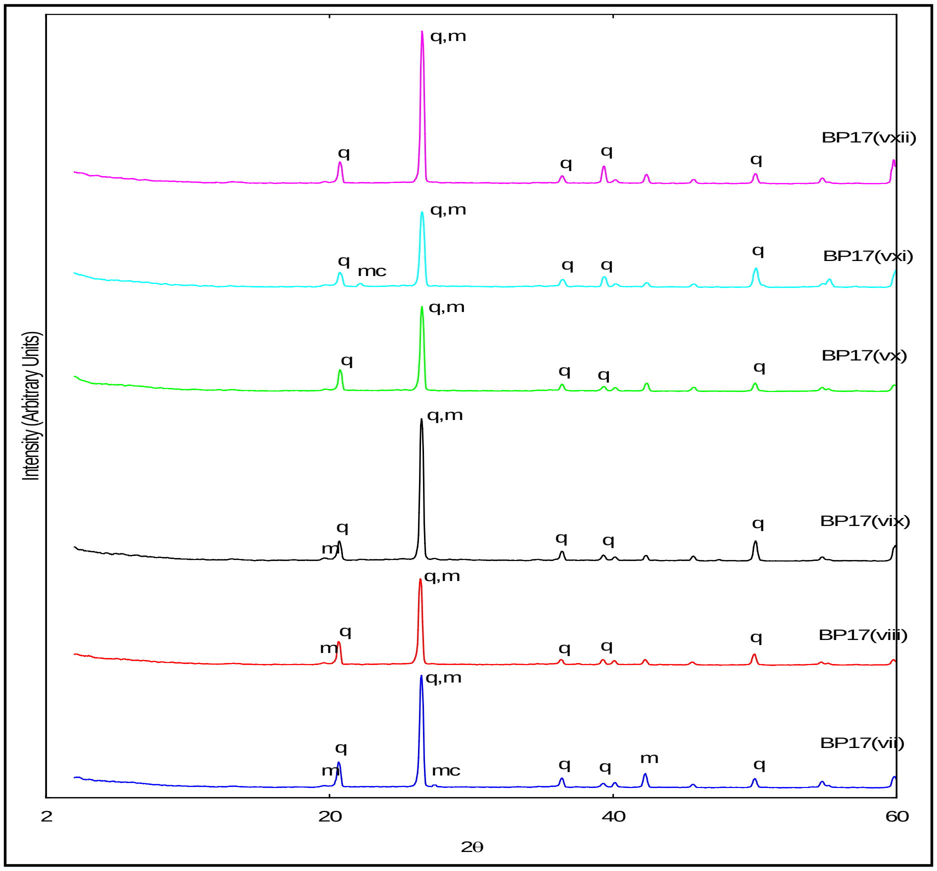


Legend: q = quartz, m = muscovite, o = orthoclase

Fig. 3: The XRD diffraction pattern of *Candi bukit pendiat* bricks (Site 17)

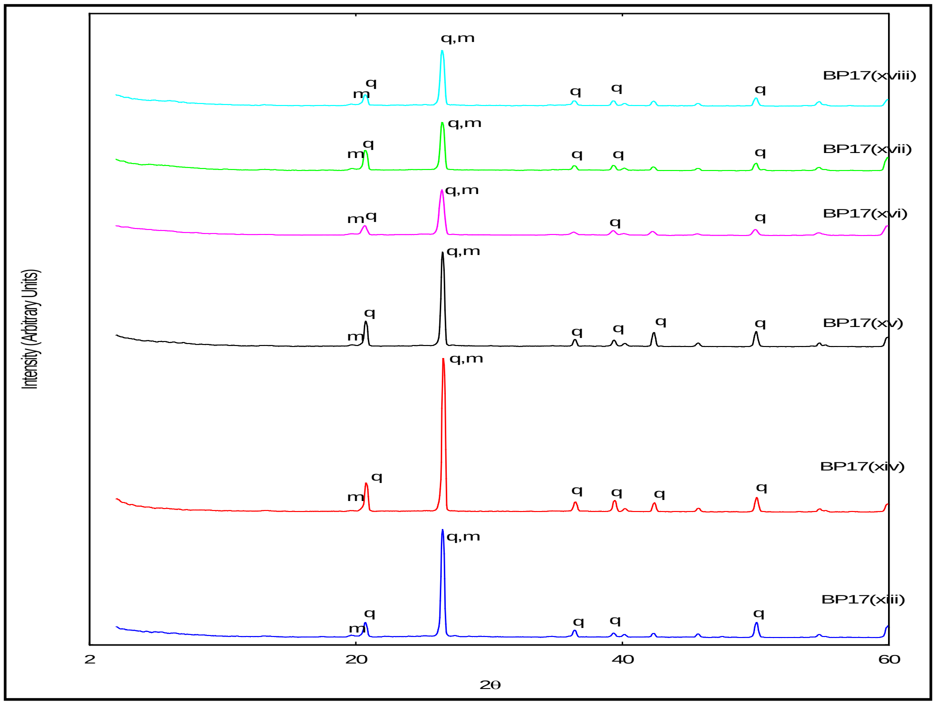
mineral content in the brick samples of *Candi bukit pendiat* (Site 17) consists of quartz, muscovite, microline and kaolinite. The kaolinite mineral shows that some of the bricks were not baked at sufficient temperature namely less than 550˚C. This shows that the open burning technique was used in the production of these temple bricks. Physical observation of the bricks shows that incomplete burning occurred. The XRD patterns of Site 17 brick samples can be referred to in Fig. 3, 4 and 5.

The content of the major elements in the ancient brick samples of *Candi bukit pendiat* (Site 17) can be referred to in detail in Table 4. The analysis shows that the bricks have between 72.30% to 78.90% of dry weight percentage for silica element. The dry weight percentage of titanium element is between 0.58% to 0.74%. Iron element has dry weight percentage of between 0.34% to 4.06%. Dry weight percentage for aluminium element is between 16.74% to 23.59%. Manganese element contains dry weight percentage of



Legend: q= quartz, m= muscovite, o= orthoclase

Fig. 4: The XRD diffraction pattern of candi bukit pendiat bricks (Site 17)



Legend: q = quartz, m = muscovite, o = orthoclase

Fig. 5: The XRD diffraction pattern of candi bukit pendiat bricks (Site 17)

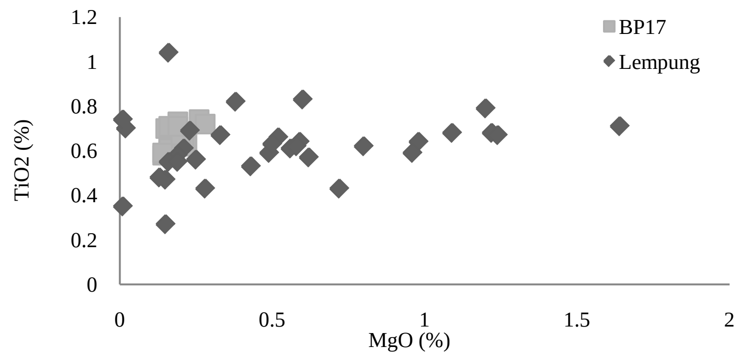


Fig. 6: Percentage of dry weight (%) of MgO and TiO_2 elements for the brick samples of *Candi bukit pendiat* (Site 17) and the clay of bujang valley

Table 5: Trace element content of the ancient bricks of *Candi bukit pendiat* (site 17)

Sample	As	Ba	Ce	Co	Cr	Cu	Ga	La	Nb	Ni	Pb	Rb	Th	V	Zn	Zr
	Trace Elements (μg/g)															
BP17 (i)	17	756	577	7	81	20	30	30	35	30	55	89	20	109	33	304
BP17 (ii)	27	690	549	8	89	12	22	29	31	25	66	44	19	114	27	301
BP17 (iii)	46	704	593	11	109	13	21	29	36	22	89	48	16	131	24	239
BP17 (iv)	16	756	633	7	82	20	29	30	36	33	55	86	20	108	34	285
BP17 (v)	35	707	576	9	111	14	23	29	34	24	76	41	23	128	25	268
BP17 (vi)	26	704	604	8	88	15	21	29	34	25	66	41	19	112	27	286
BP17 (vii)	44	680	571	11	118	14	21	29	33	23	86	46	18	128	28	247
BP17 (viii)	40	683	590	9	98	14	20	29	34	25	81	42	17	18	25	262
BP17 (ix)	16	735	608	7	79	19	29	30	33	32	54	74	20	108	33	301
BP17 (x)	39	679	583	10	109	13	22	29	33	25	79	51	20	125	26	266
BP17 (xi)	51	692	573	12	128	13	23	29	33	24	94	47	20	141	24	258
BP17 (xii)	32	684	550	8	90	13	20	29	29	25	73	39	16	127	25	265
BP17 (xiii)	35	656	554	9	92	14	21	29	28	23	75	38	23	117	26	300
BP17 (xiv)	27	668	536	8	89	15	22	29	28	26	65	32	17	110	27	295
BP17 (xv)	24	726	586	8	80	14	20	30	34	26	63	40	15	107	27	264
BP17 (xvi)	37	696	587	9	95	14	23	29	35	24	77	44	23	20	25	268
BP17 (xvii)	46	705	590	11	121	13	23	29	34	23	87	47	19	136	25	254
BP17 (xviii)	43	676	577	11	115	13	23	29	32	27	83	47	23	135	26	276

between 0.01% to 0.03% while calcium element contains dry weight percentage of 0.03% to 0.06%. The dry weight percentage for magnesium element and sodium element are between 0.14 to 0.28 and 0.05 to 0.07% respectively. The elements of potassium and phosphorus contain dry weight percentage of between 0.28 to 0.68 and 0.17 to 0.25% respectively. Elements such as silica, aluminium and iron are elements which contain the highest percentage of dry weight for the brick samples of Site 17, *Candi bukit pendiat*. The graph of dry weight percentage for the elements MgO and TiO_2 (Fig. 6) of the brick samples in *Candi bukit pendiat* (Site 17) and clay of Bujang Valley were plotted to see the results of the comparison between the brick and clay samples based on their major elements. Based on the SiO_2 and Al_2O_3 dry weight percentage graph, there is little difference between the brick and clay composition. The MgO and TiO_2 dry weight percentage shows that the brick samples of this site have similarities with the clay samples of Bujang Valley, Kedah in the composition of their major elements. This analysis shows that the raw materials of this site were obtained from the Sungai Bujang basin, Kedah.

The content of trace elements (Table 5) shows content of more than 100 ppm for the elements such as barium, cerium, chromium, vanadium and zircon. Other elements are of quite low concentrations namely less than 100 ppm. The barium element content is between 656 ppm to 756 ppm while the cerium element is between 536 ppm to 633 ppm. The chromium and vanadium element content are between 79 to 128 and 18 to 141 ppm, respectively while the zircon element shows concentration of content between 239 ppm to 304 ppm. Figure 7 shows the graph plotted to see the distribution of copper against lead for the brick samples of *Candi bukit pendiat* (Site 17) where the concentration content of copper and lead were between 12 to 20 and 54 to 94 ppm, respectively. Based on the graph, it was found that the composition of trace elements of lead and copper for the brick samples of Site 17 has differences with the composition of lead and copper elements of the clay in Bujang Valley, Kedah. Samples of some of the bricks at this site were also almost similar with the clay composition in the area of Mukim Bujang and the area in the Terus River basin and Bujang River basin. Composition analysis has

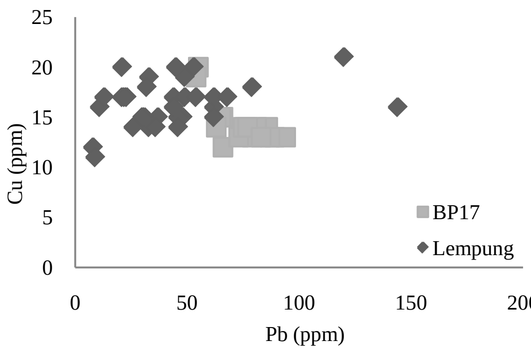


Fig. 7: Concentration of lead and copper in the brick samples of *Candi bukit pendiat* and the clay of bujang valley, kedah

proven that the raw materials used to produce the bricks at this site were local raw materials.

CONCLUSION

The study on material composition of the ancient bricks of *Candi bukit pendiat* (Site 17) shows that the bricks have almost similar material composition as the clay in Bujang Valley, Kedah which is based on major and trace elements which is similar to compositional of clay samples. The mineral content present in the ancient brick samples consists of quartz, muscovite, microline and kaolinite. The kaolinite mineral shows that some of the bricks were not baked at sufficient temperature namely less than 550°C. This shows that the open burning technique was used in the production of the temple's bricks because some of the bricks have an indication of low firing burning. The dry weight percentage graph of silica and aluminium and magnesium and titanium as well as the lead and copper concentration graph indicate that the raw materials used to produce the ancient bricks are local raw materials and these raw materials were obtained from the Sungai Bujang and Sungai Terus basin, Bujang Valley, Kedah.

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