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X-Ray Diffraction and X-Ray Flourescence of (9th-10th Century AD) Ancient Bricks of Pengkalan Bujang Temple (Site 21/22) Bujang Valley, Kedah

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Abstract: Candi Pengkalan Bujang site 21/22) is one of the temple sites of Buddha element that used bricks as its main construction material in addition to the use of pillar bases made from granite stones and pillars believed to have been made of wood and palm roof. Relative dating that was made on this site suggests it was built between the 9-10th century AD. The two main structures, namely a structure that had vimana and mandapa areas, as well as stupa structure showed there was evolution in temple construction using bricks after the 10th century AD, in particular in the usage of bricks and its arrangement. This study will focus on the material composition analysis of ancient bricks that were used to create this temple of which its main purpose is to see whether the raw material used to produce the bricks utilised local raw material. The two analysis techniques will be used, namely; the X-Ray Fluorescence Technique (XRF) and the X-Ray Diffraction Technique (XRD) in determining the major and trace element content, as well as mineral content in the ancient bricks. Results of the study show that the major minerals in the ancient bricks of Candi Pengkalan Bujang (site 21/22) are the presence of major minerals, such as quartz, muscovite and microcline while other minerals that exist in small quantities are minerals, such as gibbsite, mullite and kaolinite. Kaolinite mineral content in the PB21/22 (xx-xxii) samples shows that the bricks were fired at a temperature below 550°C. PB21/22 (iii, iv and viii) samples only contained quartz mineral content and this shows that the samples were fired at high temperatures between 850-1000°C. The mineral content and physical observation of the bricks indicate that the open firing technique was used in producing the bricks. The major and trace element content also show that these bricks were produced from the same source and it is suggested that local raw material was used in the production of the bricks. The involvement of the local community in producing the bricks should not be refuted, proving the knowledge transformation of the local community in Bujang Valley had already begun, since the 4th century.

Key words: Candi Pengkalan Bujang (site 21/22), x-ray fluorescence, x-ray diffraction, bricks, Bujang Valley

INTRODUCTION

There are 4 monument sites that are located at the North side of site 19 that were identified by Quaritch (1940) in his survey in the year 1936-1937. The sites are: Site 20-23. In 1974, the Museum and Antiquity Department had conducted excavation work, as well as rebuilding of the temples of these sites, except site 23. Archaeological research results and report by Quaritch-Wales indicated that site 20 had suffered severe damages as a result of agricultural activities.

Site 21 and 22 (Pengkalan Bujang Village) were excavated simultaneously by the Museum and Antiquity Department in 1974. The excavation work had exposed the original plan of both sites where site 21 contained a main structure while site 22 contained 2 main structures. Site 21 and 22 actually existed in pairs. The structures' construction material on the whole used bricks. The main finds at this site were Buddha statue made of terracotta, Buddha statue made of bronze, bricks with inscriptions, scorpion and elephant statues from clay. Based on these finds, the temple represented the teachings of Buddha and based on relative dating, it is assumed the temple was built around the 9-10th century (Abdul Rahman and Yatim, 1990).

In the context of the scientific research conducted at this site, it involved material composition analysis of bricks that were used to build the temple at Candi

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Pengkalan Bujang (site 21/22). The main objective of conducting this research is to determine whether the raw material used to produce the bricks are local raw materials or otherwise. It is generally known that brick's raw material is clay that is available abundantly in Bujang Valley. This is crucial in proving this hypothesis that the Bujang Valley Malay community was already skilled in brick making technology at that time. Analysis was carried out using XRF and XRD technique and several research using the same method was done in Malaysia, especially to determine the compositional composition of artefacts, such as pottery, glass beads, bronze artifacts and also ancient bricks from monuments (Jusoh *et al*., 2012, 2013; Chia, 1997; Abdul Rahman *et al*., 2013; Ramli *et al*., 2011a-c, 2012a, b; Ramli and Abdul Rahman, 2009, 2013; Ramli *et al*., 2001, 2013a-c).

MATERIALS AND METHODS

A total of 23 brick samples were taken from the site of Candi Bukit Pendiat (site 21/22) and then taken to the lab for samples treatment and labelled with the names: PB21/22 (i), PB21/22 (ii), PB21/22 (iii), PB21/22 (iv), PB21/22 (v), PB21/22 (vi), PB21/22 (vii), PB21/22 (viii), PB21/22 (ix), PB21/22 (x), PB21/22 (xi), PB21/22 (xii), PB21/22 (xiii), PB21/22 (xiv), PB21/22 (xv), PB21/22 (xvi), PB21/22 (xvii), PB21/22 (xvii), PB21/22 (xix), PB21/22 (xx), PB21/22 (xxii), PB21/22 (xxiii), PB21/22 (xxiv). Analysis was conducted to determine the mineral, major and trace elements content in the brick samples. Samples weighing 0.4 g were refined and heated up for 1 h 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 1 h 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 2 mm 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, Ti, Al, Fe, Mn, Mg, Ca, Na, K and P_2O_3. Pressed pallet samples were prepared for analysis of trace elements, such as As, Ba, Ce, Co, Cr, Cu, Ga, Hf, La, Nb, Ni, Pb, Rb, Sr, Th, V, Y, Zn and Zr. These samples were prepared by mixing 1.0 g of samples together with 6.0 g of boric acid powder and then pressure of 20 psi was applied by using a hydraulic pressure equipment. The samples of fused pallets and pressed pallets were then analysed using a 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 Diffractometer Siemen).

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 (Ramli *et al*., 2011a, c).

RESULTS AND DISCUSSION

Scientific analysis was conducted with the purpose of proving whether the bricks used to build the temple of site 21/22 used local clay or otherwise. The 2 analysis techniques were used to achieve this purpose, namely; the technique to determine the mineral content and the technique to determine the major and trace element content. Mineral content in the brick samples found in Candi Pengkalan Bujang (site 21/22) indicated the presence of major minerals such as quartz, muscovite and microcline while other minerals that exist in small quantities are minerals, such as gibbsite, mullite and kaolinite (Table 1). Kaolinite mineral content found in PB21/22 (xx), PB21/22 (xxi) and PB21/22 (xxii) samples showed that the bricks were fired at temperatures <550°C. PB21/22 (iii), PB21/22 (iv) and PB21/22 (viii) samples only contained quartz mineral content and this showed that the samples were fired at high temperatures between 850-1000°C. Mullite mineral came about as a result of the pressure of high temperature that was applied on the kaolinite mineral. Clearly, here the open firing technique was performed to produce the bricks at Pengkalan Bujang site (site 21/22) because of the irregular temperature applied on the bricks while the firing work was carried out. This might be due to the way the arrangement of the bricks was done before the firing process was conducted.

The bricks used to build this temple is smaller and has a standard size compared to the previous temples, particularly the temple at site 23 that is also located close to this temple. In the aspect of brick making and temple architecture at this time, it can be said that changes or evolution took place. Temple architecture is also more complex compared to previously and can be seen in the architecture of site 21 (Pengkalan Bujang Village).

The major element content in the brick samples of site 21/22 can be referred to in Table 2. Analysis results showed that the brick samples have high silica and aluminium content. Dry weight percentage for silica is between 61.82-79.07% while for aluminium is between 15.22-30.82%. Iron element content is also high that is between 1.10-6.19%; however the mineral content analysis did not discover geothite mineral in any sample. Dry weight percentage content for potassium is between 0.18-2.22% while the calcium element is between 0.06-1.18%. Even though, there is calcite mineral ($CaCO_3$) in the PB21/22 (xiv) sample, the calcium element content is still at a low level. The titanium and magnesium element content is 0.46-2.61 and 0.15-1.12%, respectively. The graphs for the silica and aluminium dry weight percentage of the brick and clay samples and the titanium and magnesium dry weight percentage of the brick and clay samples were plotted to see the distribution of the brick samples together with the clay samples of which its data were obtained from the earlier analysis. Figure 1 and 2 show that the brick samples of site 21/22 used local raw material. This proves the continuity of brick production to build temples in Bujang Valley which begun in Sungai Mas by the local community since the 4th or 5th century AD (Ramli *et al*., 2012b).

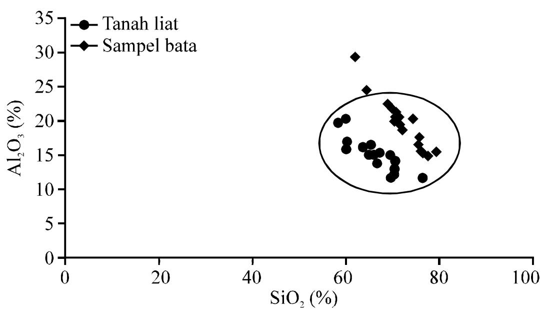

Fig. 1: Dry weight percentage of silica and aluminium in the brick and clay samples

Trace element content contained in the brick samples of site 21/22 can be referred to in Table 3-5.

Table 1: Mineral content in the brick samples of site 21/22 (Candi Pengkalan Bujang)

Samples	Mineral content
PB21/22 (i)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 1M
PB21/22 (ii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $Al_2O_3.3H_2O$ Gibbsite
PB21/22 (iii)	SiO_2 Quartz
PB21/22 (iv)	SiO_2 Quartz
PB21/22 (v)	SiO_2 Quartz $H_2KAl_3Si_3O_{12}$ Muscovite
PB21/22 (vi)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $KAlSi_3O_8$ Microcline
PB21/22 (vii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $KAlSi_3O_8$ Microcline
PB21/22 (viii)	SiO_2 Quartz
PB21/22 (ix)	SiO_2 Quartz $Al_6Si_2O13/3Al_2O_3.2SiO_2$ Mullite
PB22/21 (x)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $KAlSi_3O_8$ Microcline
PB21/22 (xi)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 1M
PB21/22 (xii)	SiO_2 Quartz $KalSi_3O_8$ Microcline
PB21/22 (xiii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 1M
PB21/22 (xiv)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 1M $CaCO_3$ Calcite
PB21/22 (xv)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $KAlSi_3O_8$ Microcline
PB21/22 (xvi)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $KAlSi_3O_8$ Microcline
PB21/22 (xvii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $KAlSi_3O_8$ Microcline
PB21/22 (xviii)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1
PB21/22 (xix)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 1M
PB21/22 (xx)	SiO_2 Quartz $Al_2Si_2O_5$ $(OH)_4$ Kaolinite $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $KAlSi_3O_8$ Microcline
PB21/22 (xxi)	SiO_2 Quartz $KAl_2Si_3AlO_{10}$ $(OH)_2$ Muscovite 2M1 $Al_2Si_2O_5(OH)_4$ KAolinite
PB21/22 (xxii)	SiO_2 Quartz $Al_2Si_2O_5$ $(OH)_4$ Kaolinite KAl_2 Si_3 AlO_{10} $(OH)_2$ Muscovite 2M1 $KalSi_3O_8$ Microcline
PB21/22 (xxiii)	SiO_2 Quartz $K_2O.3Al2O_3.6SiO_2.2H_2O$ Muscovite

Table 2: Major element content in the brick samples of site 21/22

Samples	Dry weight (%) Si	Ti	Al	Fe	Mn	Mg	Ca	Na	K	P_2O_3
PB21/22 (i)	61.82	1.47	30.82	1.10	0.03	0.55	1.18	0.18	0.18	0.02
PB21/22 (ii)	69.13	1.03	23.09	3.47	0.04	0.50	0.82	0.50	0.79	0.01
PB21/22 (iii)	75.77	2.61	18.21	4.23	0.01	0.15	0.06	0.05	0.32	0.01
PB21/22 (iv)	73.95	0.61	21.27	1.63	0.01	0.30	0.06	0.06	2.22	0.01
PB21/22 (v)	71.38	0.62	21.51	3.58	0.02	0.66	0.12	0.13	1.73	0.02
PB21/22 (vi)	71.67	0.72	19.89	5.61	0.01	0.33	0.07	0.08	1.25	0.01
PB21/22 (vii)	70.91	0.63	22.34	3.66	0.02	0.37	0.14	0.13	1.72	0.02
PB21/22 (viii)	79.07	0.57	16.10	2.92	0.01	0.16	0.08	0.06	0.32	0.01
PB21/22 (ix)	75.57	0.56	15.57	3.61	0.03	1.12	0.22	0.16	1.64	0.01
PB22/21 (x)	77.02	0.46	15.22	2.91	0.02	0.56	0.12	0.16	1.82	0.01
PB21/22 (xi)	70.45	0.76	22.39	4.17	0.01	0.49	0.01	0.11	1.57	0.01
PB21/22 (xii)	71.79	0.74	21.12	3.96	0.01	0.39	0.08	0.09	1.35	0.01
PB21/22 (xiii)	71.18	0.76	21.05	4.39	0.01	0.50	0.07	0.09	1.19	0.01
PB21/22 (xiv)	68.73	0.77	23.49	4.34	0.02	0.59	0.13	0.12	1.79	0.02
PB21/22 (xv)	70.65	0.81	21.41	4.82	0.01	0.54	0.08	0.09	1.41	0.01
PB21/22 (xvi)	75.47	0.48	18.25	2.75	0.04	0.37	0.12	0.16	1.64	0.01
PB21/22 (xvii)	72.02	0.62	19.10	5.10	0.01	0.65	0.09	0.08	1.07	0.01
PB21/22 (xviii)	64.19	1.33	26.19	4.98	0.01	0.26	0.08	0.28	2.58	0.01
PB21/22 (xix)	70.14	0.82	20.66	6.19	0.01	0.34	0.11	0.08	1.39	0.01
PB21/22 (xx)	75.93	0.51	15.21	3.34	0.02	1.01	0.18	0.13	1.41	0.02
PB21/22 (xxi)	75.83	0.53	16.26	3.02	0.02	0.61	0.19	0.22	2.20	0.01
PB21/22 (xxii)	75.19	0.53	16.83	3.23	0.03	0.77	0.20	0.20	1.85	0.01
PB21/22 (xxiii)	70.55	0.81	21.56	4.60	0.01	0.60	0.07	0.09	1.42	0.01

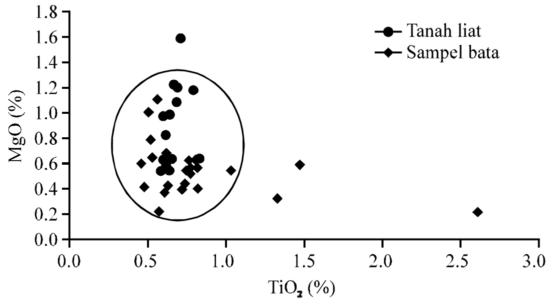

Fig. 2: Dry weight percentage of titanium and magnesium in the brick and clay samples

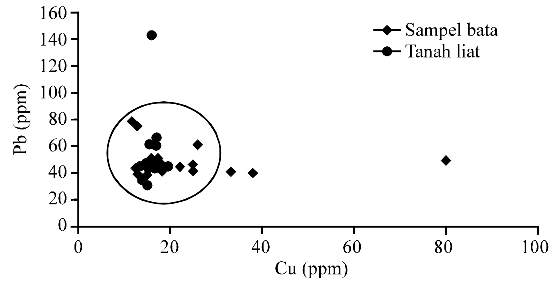

Fig. 3: Concentration of copper and lead elements in the samples of brick (site 21/22) and clay

Table 3: Trace element content in the brick samples of site 21/22 (i-viii)

Elements (ppm)	Sample (PB21/22) i	ii	iii	iv	v	vi	vii	viii
As	17	19	44	15	14	15	17	36
Ba	628	746	657	679	707	767	806	727
Ce	548	548	539	606	588	573	563	599
Co	28	13	14	9	12	12	10	10
Cr	115	67	121	80	86	70	64	99
Cu	15	25	12	17	25	12	15	13
Ga	24	25	20	27	25	15	16	19
Hf	7	7	7	8	7	7	7	7
La	28	29	29	29	29	29	30	30
Nb	28	31	30	32	32	29	30	32
Ni	20	25	22	30	28	26	27	23
Pb	38	47	81	52	44	44	50	76
Rb	101	65	34	33	191	176	178	47
Sr	14	133	2	2	17	32	31	2
U	9	9	9	9	9	9	9	9
Th	29	13	17	21	21	18	17	17
V	122	134	134	91	99	84	81	121
Y	14	2	0	0	27	17	16	0
Zn	49	100	31	53	68	59	71	29
Zr	219	248	231	287	21	181	174	223

Table 4: Trace element content in the brick samples of site 21/22 (ix-xvi)

Elements (ppm)	Sample (PB21/22) ix	x	xi	xii	xiii	xiv	xv	xvi
As	13	17	16	12	15	15	17	15
Ba	712	779	736	714	683	770	789	773
Ce	505	571	632	570	554	620	613	546
Co	14	12	11	11	12	12	13	9
Cr	90	81	97	82	108	104	112	62
Cu	13	16	14	13	27	27	28	15
Ga	22	18	25	19	27	27	28	15
Hf	7	7	7	7	7	7	7	7
La	29	29	29	29	29	30	30	29
Nb	28	29	33	32	28	33	33	31
Ni	30	32	28	27	26	27	26	28
Pb	37	47	46	37	42	46	46	45
Rb	166	165	81	127	115	136	134	183
Sr	48	38	10	38	15	12	14	34
U	9	9	9	9	9	9	9	9
Th	27	22	26	21	32	29	32	18
V	94	89	119	87	115	114	122	79
Y	31	23	6	17	15	18	17	20
Zn	83	84	63	93	69	63	62	25
Zr	206	221	289	188	221	215	222	182

Concentrations of trace elements beyond 100 ppm were recorded in elements, such as barium, cerium, rubidium, vanadium and zircon. Barium element concentration that was recorded is between 628-1146 ppm. Cerium element recorded readings between 457-632 ppm while rubidium element was between 33-242 ppm. Zircon element recorded concentration between 174-289 ppm. Other elements recorded readings <100 ppm where copper element recorded concentration reading of between 12-80 ppm while lead element was between 37-81 ppm. The graph of copper and lead element concentration for brick samples of site 21/22 and clay samples in Bujang Valley was plotted to see whether the distribution of the elements is similar or different (Fig. 3). Readings showed that concentration of these elements are the same between the brick samples of site 21/22 and the clay samples in Bujang Valley. This result is consistent with the result recorded for the major elements.

Table 5: Trace element content in the brick samples of site 21/22 (xvii-xxiii)

Elements (ppm)	Sample (PB21/22) xvii	xviii	xix	xx	xxi	xxii	xxiii
As	18	16	14	26	20	21	17
Ba	695	1146	749	743	708	784	747
Ce	500	457	572	600	584	544	579
Co	16	13	13	10	12	12	13
Cr	117	131	107	122	135	100	113
Cu	33	38	17	26	18	80	22
Ga	25	32	29	37	35	29	28
Hf	7	7	8	8	7	7	7
La	29	29	29	29	29	30	29
Nb	28	31	32	32	32	30	31
Ni	23	38	30	34	35	28	27
Pb	41	41	46	63	50	52	46
Rb	119	242	138	135	96	181	149
Sr	11	96	15	18	21	14	14
U	9	9	9	11	9	9	9
Th	30	31	33	43	44	27	34
V	120	160	117	135	133	112	123
Y	13	91	22	25	27	30	22
Zn	71	61	68	99	84	76	75
Zr	236	230	229	266	283	252	231

CONCLUSION

Findings of the archaeological and scientific studies conducted on the Candi Pengkalan Bujang (site 21/22) have uncovered several strong evidences that there were involvements of the local community in the process of development and prosperity of Bujang Valley, as one of the settlements with an entrepot and a community that accepted the influence of Hindu-Buddha. The tools found at the layer of the proto-historic culture indicated the influence and role played by the local community at the time. The stone tools were used to pound the hematite that were often used in religious ceremonies. Scientific analysis has proven that the bricks used to build the temple at site 21/22 had used local raw material. This showed that the local community had already mastered the knowledge and technology of brick making and temple architecture in Bujang Valley. This technology was already mastered, since the 5th or 6th century AD and it begun in the Sungai Mas area that was the centre of all science and technology knowledge development in Malaysia with products, such as earthenware, bricks, iron tools and monochrome glass beads or more known as Indo-Pacific beads.

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