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XRD AND XRF ANALYSIS OF THE ANCIENT BRICKS FROM CANDI KAMPUNG BARU, KEDAH

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Abstract

XRD and XRF analysis on the ancient bricks were conducted to identify whether the raw materials to produce the bricks originate from the site. Candi Kampung Baru is recorded as one of the temple that used bricks as the main construction material. This site is suggested to be built between the 8th to 10th centuries AD based on the size of the bricks and imported ceramics associated with the structure. The position of this *candi* that is facing to the north is a major indication that the *candi* in Kampung Baru Site is one of the many Buddhist *candi*s that have been found in Bujang Valley. This study used X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) analysis techniques to identify the composition of the major and trace elements and minerals found in the bricks. The results of the analysis on the ancient brick of Candi Kampung Baru revealed that the mineral content contained in the brick samples consists of quartz, muscovite, microcline and sanidine. Minerals found suggest that open burning technique was used during the brick making process with the local raw clay sources were used to produce the bricks. The results therefore implied that the participation of the local communities in Old Kedah Kingdom contributed to the prosperity of this region.

Keyword: x-ray fluorescence (XRF), x-ray diffraction (XRD), candi Kampung Baru, Bujang Valley, old Kedah kingdom

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INTRODUCTION

The Kampung Baru site was discovered during the Muda River exploration work which was conducted in 2010 until 2011 by a group of researchers from The National University of Malaysia (Ramli & Nik Abdul Rahman, 2012). This site is located on the bank of Muda River in Kampung Baru, Kota Kuala Muda, Kedah and the distance between this site and the Muda River is about 100 metres and the archaeological excavations that have been carried out on this site have also found various types of artefacts such as earthenware, glass, metal and other ceramics such as c'hing-pai and celadon (Mohd Nasir, Ramli, & Hassan, 2017).

The study of the ancient brick from the *candi*'s found in Kedah has formerly been carried out by scholars. However, there is a contradiction between scholars and researchers about the Old Kedah *candi*'s builders and the sources of building materials besides each one of them has their own arguments and evidence. Similarly, the source of raw materials used to build a temple at the Kampung Baru Site is still unknown, therefore, the scientific analysis of the ancient brick at Kampung Baru Candi is necessary to identify the composition of the brick. XRF and XRD analysis have also provided new data on the origin and technology of prehistoric pottery manufacturing in Malaysia (Treloar, 1978; Chia, 1997), glass beads (Ramli, Nik Abdul Rahman, & Samian, 2011; Ramli, Abdul Rahman, Hussin, Sayed Hasan, & Mohamed Dali, 2017), bronze drum (Jusoh, Sauman, Abdul Rahman, & Ramli, 2012), as well as ancient bricks (Ramli, 2012; Ramli, Nik Abdul Rahman, Jusoh, & Hussein, 2012; Ramli et al., 2013; Sabtu, Mahat, Mohd Amin, Price, & Bradley, 2015). Whereas Yahaya, Hussein, Ramli, and Zakaria (2005) conducted a physical analysis on the bricks found in Kuala Muda, Kedah. The study focused on the brick pressure force tests and also by looking at the physical size of the brick. Even closer study has also been conducted at the Panggung Drama Building, Kuala Lumpur which combines the analysis of the content and physical composition of the building materials (Hussein, Ramli, & Yahaya, 2004).

The Kampung Baru site located near Kampung Sungai Mas site was the centre of government and the port of Old Kedah. The study on the Kampung Sungai Mas site was initiated in 1979 by Jane Allen and Jan Wisseman Christe in collaboration with the Bujang Valley Archaeological Museum and the Department of Geography, University of Malaya to conduct research on "Trade transportation and tributaries: exchange, agriculture and settlement distribution in early historic-period Kedah, Malaysia". Subsequently in 1980 a team of researchers from Universiti Kebangsaan Malaysia led by Nik Hassan Shuhaimi Nik Abd. Rahman has conducted an archaeological survey at the Kampung Sungai Mas Site (Nik Abdul Rahman & Mohd Yatim, 1992). In 1981, Nik Hassan Suhaimi and his team conduct the first trial excavation at the Site 33 (Abdullah, 2013). Drawing to this activity, in 1985, another site identified as Kampung Sungai Mas (Site 32/34) was selected for the 2nd Intra-ASEAN Archaeological

Excavation and Conservation project of Bujang Valley, Kedah, Malaysia in 19858. Discovery of foreign ceramics, building materials, glass, pottery, beads, and stone have become significant findings at that time.

Compositional analysis of the ancient brick used in the construction of the candi in Kampung Baru is carried out in order to determine the mineral content besides the major and trace elements contained in the brick samples. Data obtained from the subsequent brick analysis will be compared with the composition data of clay material around the Bujang Valley area, Kedah. 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, because there are scholars who think that the candis found in Old Kedah was built by the Indian traders (Jacq-Hergoualc'h, 1992; Treloar, 1978; Wheatley, 1964). Besides that, the bricks that were found at this site also have different sizes and shapes (refer figure 1 until 4 below).

Figure 1: Brick 5 (top view)

Figure 2: Brick 5 (side view)

Figure 3: Brick 28 (top view)

Figure 4: Brick 28 (side view)

METHODOLOGY

A total of 20 brick samples were taken from the Candi Kampung Baru Site and then taken to the lab for cleaning and labelled with the names KB 1, KB 2, KB 3, KB 4, KB 5, KB 6, KB 7, KB 8, KB 9, KB 10, KB 11, KB 12, KB 13, KB 14,

KB 15, KB 16, KB 17, KB 18, KB 19, and KB 20. The 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 105o 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 1100o 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 32mm. The samples were 1:10 dilution. Samples in the formed 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, Cr, Cu, Ga, Ni, Pb, Rb, Sr, 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 the pressure of 20 psi was applied by using hydraulic pressure equipment. The samples of the fused pallet and pressed pallets were then analysed by using the Philips PW1480 equipment. Samples in the form of very fine powder were put into the pellets (sample holder) and then analysed by using the X-ray Diffraction instrument (D500 Diffract meter SIEMEN).

A scatter plot diagram of MgO versus TiO2 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. The applicability of the analytical methods for the multi-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 materials, 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 results of the analysis showed that the mineral content contained in the brick samples consists of quartz, muscovite, microcline, sanidine. Besides, there was no kaolinite mineral in the brick sample but based on the physical appearance it was found that the brick did not have complete oxidation (refer Table 1). This showed that open burning techniques were used during the bricks making process. The burning temperature of the brick samples from this site was believed to be between 600°C to 800°C.

The major element contents contained in the ancient brick samples of Kampung Baru Candi in detail can be referred to Table 2. The analysis showed that the ancient brick samples contained percentage of dry weight quartz elements between 70.00% to 83.66%. The percentage of dry weight of titanium was between 0.07% to 0.96%. The iron element contained a dry weight percentage of

1.81% to 3.68%. The percentage of dry weight of aluminium was between 13.95% and 22.54%. The manganese element contained a dry weight percentage of 0.01% to 0.04% while the calcium elements contained a dry weight percentage of 0.05% to 0.34%. The percentage of dry weight of magnesium and sodium was between 0.18% to 0.99% and 0.07% to 0.42%. Potassium and phosphorus elements contained dry weight percentage ranging from 0.46% to 2.15% and 0.11% to 0.85%.

Elements such as silica, aluminium, and iron are elements containing a high percentage of dry weight for brick samples at Kampung Baru Candi. The percentage graph of the dry weight of SiO2 and Al2O3 (refer Figure 5) and also the percentage graph of the dry weight of MgO and TiO2 (refer Figure 6) for brick samples from Candi Kampung Baru and clay in Bujang Valley have been plotted to observe the results of the comparison between the samples of brick and clay based on its primary source. Based on the graphs, the composition of the major elements of the brick sample of Kampung Baru Candi was found to be similar to the composition of the major elements of clay in the Bujang Valley. These raw materials were obtained from the Sungai Muda basin, the Bujang River basin and the surrounding Mukim Merbok and Mukim Bujang. In fact, this study also found that the raw materials used at this site were obtained from various sources compared to the raw material used to produce bricks candi in Kampung Sungai Mas (Site 32/34) that only used raw materials found in Muda River and Bujang River.

Table 1: Minerals content in bricks sample from Candi Kampung Baru

Site	Sample	Mineral Content
Kg. Baru, Kota Kuala Muda, Kedah	KB 1	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M $K_2O.Al_2O_{3.6}SiO_2$ Microline
	KB 2	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	KB 3	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M $KAlSi_3O_8$ Microcline
	KB 4	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	KB 5	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M $KAlSi_3O_8$ Microcline
	KB 6	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M $K_2O.Al_2O_{3.6}SiO_2$ Microline
	KB 7	SiO_2 Quartz $KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M $KAlSi_3O_8$ Microcline
	KB 8	SiO_2 Quartz

	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
KB 9	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	$KAlSi_3O_8$ Microcline
KB 10	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	$KAlSi_3O_8$ Microcline
KB 11	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	$K_{0.47}Na_{0.43}Ca_{0.10}Al_{1.1}Si_{2.9}O_8$ Sanidine
KB 12	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
KB 13	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	$KAlSi_3O_8$ Microcline
KB 14	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
KB 15	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
KB 16	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	$KAlSi_3O_8$ Microcline
KB 17	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
KB 18	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
KB 19	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M
	$KAlSi_3O_8$ Microcline
KB 20	SiO_2 Quartz
	$KAl_2Si_3AlO_{10}(OH)_2$ Muscovite 1M

Table 2: Dry Weight (%) of major elements in Candi Kampung Baru Brick Samples

Sample	**Dry Weight (%)**									
	Si	**Ti**	**Fe**	**Al**	**Mn**	**Ca**	**Mg**	**Na**	**K**	**P_2O_3**
KB 1	70.00	0.49	3.21	13.95	0.02	0.14	0.44	0.19	1.40	0.22
KB 2	72.83	0.93	3.37	21.47	0.01	0.05	0.20	0.10	0.73	0.15
KB 3	77.96	0.42	3.05	12.98	0.03	0.25	0.90	0.42	1.96	0.45
KB 4	72.97	0.96	2.85	21.76	0.01	0.04	0.22	0.09	0.72	0.12
KB 5	76.81	0.52	3.07	14.14	0.04	0.30	0.98	0.19	1.62	0.28
KB 6	73.86	0.53	3.27	16.62	0.02	0.30	0.99	0.31	2.12	0.85
KB 7	75.08	0.54	3.68	16.06	0.02	0.30	0.84	0.25	1.96	0.32
KB 8	75.62	0.70	1.81	20.33	0.01	0.03	0.18	0.07	0.58	0.13
KB 9	76.05	0.46	2.77	14.72	0.02	0.29	0.80	0.28	1.30	0.38
KB 10	76.59	0.49	3.04	14.43	0.02	0.25	0.97	0.27	1.73	0.33
KB 11	76.58	0.49	2.92	15.07	0.03	0.24	0.67	0.32	1.93	0.61

KB 12	73.88	0.80	2.49	21.41	0.01	0.09	0.22	0.11	0.68	0.14
KB 13	74.94	0.51	3.31	15.14	0.04	0.34	0.86	0.51	2.15	0.44
KB 14	83.66	0.07	1.93	21.20	0.01	0.04	0.28	0.07	0.46	0.11
KB 15	73.27	0.68	3.50	18.97	0.01	0.10	0.59	0.20	0.83	0.34
KB 16	71.40	0.93	3.59	22.51	0.02	0.09	0.21	0.11	0.80	0.28
KB 17	73.80	0.95	2.82	20.48	0.01	0.07	0.26	0.10	0.58	0.19
KB 18	71.72	0.91	3.40	22.54	0.01	0.05	0.21	0.08	0.74	0.13
KB 19	76.75	0.51	3.71	14.5	0.02	0.24	01.01	0.24	1.62	0.29
KB 20	77.22	0.62	1.72	18.65	0.01	0.03	0.20	0.07	0.62	0.14

The content of trace elements (Table 3) showed the content of more than 100 ppm for the elements such as barium, cerium, chromium, vanadium, and zircon. Other elements were quite low concentration namely less than 100 ppm. The barium element content was between 638 ppm to 835 ppm while the cerium was between 468 ppm to 625 ppm. The content of rubidium and chromium elements was between 56 ppm to 205 ppm and 67 ppm to 111 ppm while vanadium and zircon elements had a concentration between 79 ppm to 140 ppm and 165 ppm to 430 ppm. Figure 7 is a graph plotted to see the distribution of copper elements against the lead to brick samples at Kampung Baru Temple where the concentration of the two elements was between 14 ppm to 20 ppm and 41 ppm and 57 ppm. The result showed that the raw material used to make the brick is a local raw material.

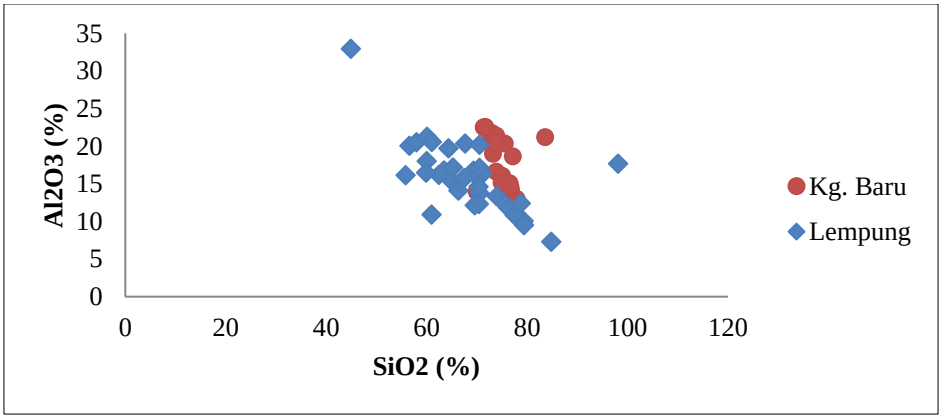

Figure 5: Scatter plot of SiO_2 and Al_2O_3

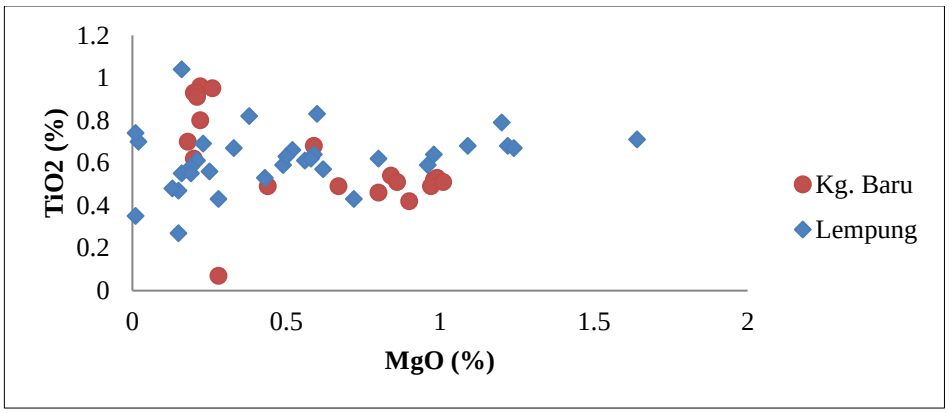

Figure 6: Scatter plot of MgO and TiO2

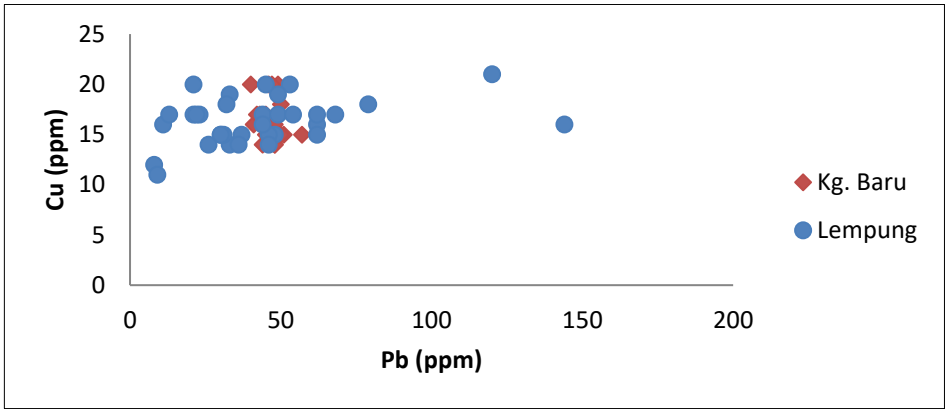

Figure 7: Scatter plot of SiO2 and Al2O3

Table 3: Content of trace elements (ppm) in bricks sample from Candi Kampung Baru

Sample	ppm													
	As	**Ba**	**Ce**	**Cr**	**Cu**	**Ga**	**Ni**	**Pb**	**Rb**	**Sr**	**Th**	**V**	**Zn**	**Zr**
KB 1	19	747	574	81	20	17	46	49	132	31	23	88	76	170
KB 2	16	696	584	100	14	27	26	48	66	9	24	125	45	332
KB 3	18	835	468	67	17	15	32	45	187	62	19	79	81	220
KB 4	17	710	600	100	15	26	27	51	59	11	28	134	48	417
KB 5	15	721	553	77	16	18	31	41	173	53	23	89	90	241
KB 6	17	818	566	81	20	19	39	46	192	53	24	89	105	278
KB 7	17	755	608	85	17	20	34	43	184	58	24	94	98	173
KB 8	10	706	569	74	15	24	28	46	56	8	13	105	45	349
KB 9	17	747	538	72	15	17	29	45	195	58	20	82	83	165
KB 10	14	768	599	80	17	18	29	42	171	44	22	88	64	189
KB 11	18	801	509	71	20	18	31	47	195	51	24	84	96	202
KB 12	15	749	586	93	18	26	30	50	66	11	23	112	50	281

KB 13	18	745	535	76	14	18	34	44	205	59	25	87	78	269
KB 14	9	727	617	79	16	23	35	47	57	8	12	103	42	315
KB 15	18	689	590	88	16	24	30	48	88	26	19	106	76	279
KB 16	24	664	502	109	15	28	29	57	66	10	27	131	65	430
KB 17	15	750	614	94	14	26	28	48	60	10	27	123	42	382
KB 18	15	781	625	111	15	28	29	50	60	9	24	140	53	348
KB 19	16	638	469	86	20	20	36	40	159	52	26	89	94	199
KB 20	11	761	559	76	15	22	29	47	58	5	12	97	42	320

CONCLUSION

The study on the material composition of the ancient bricks of Candi Kampung Baru shows that the bricks have an almost similar material composition as the clay in Bujang Valley, Kedah which is based on major and trace elements which are similar to compositional of clay samples. The mineral content present in the ancient brick samples consists of quartz, muscovite, and microcline. The results of the study also found that the open burning technique was used in the production of the candi's bricks because some of the bricks have an indication of low firing burning process. 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 materials were obtained from the Bujang River and Muda River Basin in Kedah. Therefore, local community was directly involved in brick making industry where the industry was the main supplier for the construction of several structure or monument made from bricks located in Old Kedah Kingdom.

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