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ELEMENTAL ANALYSIS OF CERAMIC ARTEFACTS FROM TANJUNG SIMPANG MENGAYAU SHIPWRECK IN SABAH

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Abstract: This article discusses the scientific analysis of ceramic artefacts recovered from a shipwreck at Tanjung Simpang Mengayau in Kudat, Sabah. The main objective is to determine the chemical composition of these artefacts to identify the materials and technology used in the production of the ceramics. Understanding the ceramic materials and technology will assist archaeologists and conservators to find the best methods for long-term and sustainable preservation of the ceramics heritage in Sabah. In total, 16 ceramic jar and celadon bowl sherds recovered from the Tanjung Simpang Mengayau shipwreck were analysed using X-ray diffraction (XRD) and X-ray fluorescence (XRF). The results revealed the presence of quartz and mullite minerals in the jars while quartz, mullite and cristobalite minerals were found in the celadon bowls, suggesting that both the jars and celadon bowls were made of kaolinite clay with added sand and limestone, but fired at different temperatures of 1,345°C and 1,470°C, respectively. Data gathered from this study is important not only for finding out and comparing the ceramic materials and production methods, but also to identify the possible origins of the Tanjung Simpang Mengayau ceramics.

Keywords: Scientific analysis, Tanjung Simpang Mengayau, sustainable preservation, ceramics, technology.

Introduction

Underwater archaeological excavations undertaken at the Tanjung Simpang Mengayau shipwreck site, off the coast of Kudat, Sabah, from 2003 to 2016 had contributed new knowledge and data on the maritime archaeology and history of Borneo and Southeast Asia (Figure 1). Although the shipwreck site of Tanjung Simpang Mengayau had been disturbed and looted by local fishermen and treasure hunters since the 1980s, the Sabah Museum Department, in collaboration with the Department of Museums and Antiquities Malaysia and Universiti Malaysia Sabah has managed to rescue and recover thousands of historical artefacts and objects from 2003 to 2006. The Tanjung Simpang Mengayau wreck is the oldest in waters of northern Borneo, dating back to the 10th century CE Song Dynasty in China (Baszley Bee *et al*., 2004; Flecker, 2012; Azman & Abdullah, 2015; Baszley Bee *et al.*, 2018). This important shipwreck laden with ancient cargoes confirmed the significance of northern Borneo as one of the main maritime routes or gateways to Brunei, Santubong, Sumatra, Java and Peninsular Malaysia (Figure 2). This has been suggested by previous studies by scholars on the context of ceramics from shipwreck sites in Southeast Asia (Craig, 2015; Orillaneda, 2016). This shipwreck site is located approximately 200 metres from the shoreline of Tanjung Simpang Mengayau, which has a narrow channel and sand banks that may be altered by strong currents during stormy weather, especially during the monsoon. This is probably the reason the ship ran aground and sank as opposed to pirate attack or fire on board (Baszley Bee *et al.*, 2007; Baszley Bee & Bala, 2009). The artefacts recovered from the Tanjung Simpang Mengayau shipwreck consisted of different types of ceramics, from jars to jarlets, celadon bowls, plates and earthenware, besides bronze drums and copper tools of various shapes. The ship's hull is scattered, with its wooden planks broken due to looting and scavenging over the centuries (Baszley Bee *et al.,* 2004; Baszley Bee *et al*., 2018).

The main objectives of this study are to determine the elemental compositions of ceramic artefacts from the Tanjung Simpang Mengayau shipwreck in Kudat, Sabah and to determine the materials and technology used in the production of the ceramics, in particular the clay type, paste materials, and firing temperatures used to produce the ceramics. The understanding of the ceramic materials and technology will assist archaeologists and conservators in preservation of Sabah's and Malaysia's ceramic cultural heritage.

The Ceramic Samples

In total, 16 ceramic sherds were analysed (Table 1). These samples were selected from the Tanjung Simpang Mengayau ceramic sherds collection at Universiti Malaysia Sabah in Kota Kinabalu, Sabah, which were recovered from underwater excavations by the Sabah Museum Department, Universiti Malaysia Sabah and Department of Museums and Antiquities Malaysia in 2004 (Baszley Bee & Bala, 2009). The samples were selected from two main ceramic types, namely jars and celadon bowls, which comprised the main artefacts found in the Tanjung Simpang Mengayau ceramic assemblage (Figure 3).

Method of Study

The ceramic samples were analysed using the X-ray diffraction (XRD) and X-ray fluorescence (XRF) spectrometers at the Centre for Global Archaeological Research (CGAR), Universiti Sains Malaysia (USM), Penang. Both these X-ray methods were chosen because they were minimally destructive and relatively fast and accurate to determine the selected range of elements and minerals within required detection limits. The XRF method could detect elements such as Si, Al, Fe, Ca, K, Na, Ti, Mg and several trace elements, which were among the most useful for characterising ceramics in Southeast Asia (Weidong *et al.,* 2005; Wenpeng *et al.,* 2019; Pei Shi *et al.,* 2020).

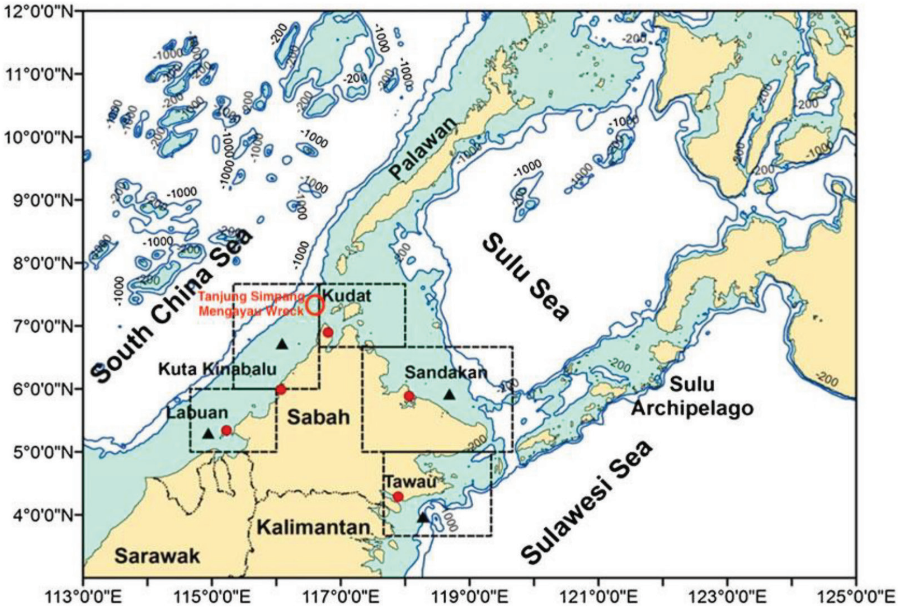

Figure 1: Location of Tanjung Simpang Mengayau shipwreck, off Kudat, Sabah, in red circle
Source: Alaa Abdul-Hadi *et al.* (2012)

Figure 2: Maritime trade network and routes in Southeast Asia ca. 10-13 CE
Source: Baszley Bee *et al.* (2018)

Table 1: List of ceramic sherds from Tanjung Simpang Mengayau shipwreck site in Kudat, Sabah

Sample Name	Type	Weight (g)	Colour
TSM/J/1	Jar	70.7	Dark reddish brown (5YR 3/4)
TSM/J/2	Jar	117.6	Light gray (2.5YR 7/2)
TSM/J/3	Jar	144.7	Light olive gray (2.5Y 6/2)
TSM/J/4	Jar	150.8	Olive gray (5Y 4/2)
TSM/J/5	Jar	369.5	Light olive gray (5Y 6/2)
TSM/B/6	Bowl	21.0	Light olive brown (2.5Y 5/4)
TSM/B/7	Bowl	56.2	Pale brown (10YR 7/3)
TSM/B/8	Bowl	138.9	Light yellowish brown (2.5Y 6/4)
TSM/B/9	Bowl	158.8	Pale olive (5Y 6/3)
TSM/B10	Bowl	96.9	Light olive gray (5Y 6/2)
TSM/B/11	Bowl	161.6	Pale olive (5Y 6/4)
TSM/B/12	Bowl	111.4	Light olive gray (5Y 6/2)
TSM/B/13	Bowl	134.6	Light gray (5Y 7/2)
TSM/B/14	Bowl	105.8	Pink (7.5Y 7/3)
TSM/B/15	Bowl	126.8	Pale olive (5Y 6/3)
TSM/B/16	Bowl	143.4	Pale yellow (2.5Y 8/2)


TSM/J/1


TSM/J/2


TSM/J/3


TSM/J/4


TSM/J/5


TSM/B/6


TSM/B/7


TSM/B/8

Figure 3: Pictures of ceramic samples from the shipwreck

The XRD method, on the other hand, could detect minerals like Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$) and Cristobalite ($SiO_2$) which were useful in identifying the types of clay and firing temperatures employed to produce the ceramics. The XRD analysis required only a small piece of sample, measuring about 10 mm long, which was cut out using a Dremel diamond blade (Model 4000 4/34). The core of this piece was pulverized using a Retch PM100 milling machine to obtain a very fine and homogenized sample at between 20 and 30 µm in particle grain size. The powdered sample was then dried in a Memmert oven (UFB500) at 105°C for two hours. After cooling to room temperature, it was analysed using the Bruker D8 Advance XRD spectrometer with scanning parameters of 10-70° (2 theta), 0.02 s step size and continuous scanning mode with Copper (Cu) X-ray Tube (1.5406Å). The diffractogram produced peaks with d-spacing value at certain points with intensity counts, respectively. The peaks of the diffractogram were identified using the DIFFRAC.Eva Version 3.0 software by referring to a Crystallography Open Database (COD).

XRF analysis was performed using the Panalytical Axios Max Spectrometer. Powdered samples were melted into fused beads and pressed into pellets using boric acid to determine the trace elements. To make the fused beads, 0.5 g of each sample was mixed with 5.0 g of flux (lithium tetraborate) and smelted in the Fluxana fusion machine (Fluxana Vulcan) at 1,200ºC until it turned into a glass pellet. To produce the pressed pellet sample, 1.5 g of the powdered glass was embedded with 5.5 g of Boric acid in a mold and the mixture was compressed into a pellet with a plastic rod. The XRF analysis parameters were set at 60 mA and 60 kV at maximum power using Rhodium (Rh) X-ray tubes. The analysis was conducted in a vacuum chamber and all samples were exposed to X-ray for approximately 30 minutes. The resulting elemental spectrum (keV) was analyzed using the SuperQ Panalytical software. All data were presented in Table 2 for XRD analysis, Table 3

Table 2: Minerals identified in ceramic samples from Tanjung Simpang Mengayau shipwreck using XRD analysis

Sample Name	Minerals
TSM/J/1	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Microcline ($Al_{1.03}K_{0.98}Na_{0.02}O_8Si_{2.97}$), Gypsum ($CaSO_4.2H_2O$)
TSM/J/2	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Gypsum ($CaSO_4.2H_2O$)
TSM/J/3	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$)
TSM/J/4	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Gypsum ($CaSO_4.2H_2O$)
TSM/J/5	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$)
TSM/B/6	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$)
TSM/B/7	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Gypsum ($CaSO_4.2H_2O$)
TSM/B/8	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Cristobalite ($SiO_2$), Wollastonite 2M ($Ca_3Si_3O_9$)
TSM/B/9	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Cristobalite ($SiO_2$)
TSM/B/10	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Cristobalite ($SiO_2$), Sillimanite ($Al_2SiO_5$), Maghemite ($Fe3O_4$), Wuestite (FeO)
TSM/B/11	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Sillimanite ($Al_2SiO5$)
TSM/B/12	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$)
TSM/B/13	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Cristobalite ($SiO_2$)
TSM/B/14	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Gypsum ($CaSO_4.2H_2O$)
TSM/B/15	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Cristobalite ($SiO_2$)
TSM/B/16	Quartz (SiO_2), Mullite ($Al_{2.28}Si_{0.72}O_{4.86}$), Gypsum ($CaSO_4.2H_2O$), Muscovite 2M1 ($KAl_3O_2Si_3O_6H_2O_4$)

Table 3: Major oxide elements with dry weight (%) value in ceramic artefacts identified using XRF analysis

Sample	Dry Weight (%)									
	$Si0_2$	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_3
TSM/J/1	48.29	0.55	17.59	2.96	0.07	1.28	0.26	0.46	2.10	0.03
TSM/J/2	48.98	0.53	17.18	2.86	0.08	1.78	0.23	0.43	2.17	0.04
TSM/J/3	50.38	0.49	14.74	1.79	0.04	0.37	0.23	0.48	2.59	0.02
TSM/J/4	49.28	0.49	16.96	2.66	0.07	1.27	0.24	0.47	2.15	0.03
TSM/J/5	49.29	0.48	17.06	2.36	0.05	0.83	0.24	0.41	2.11	0.02
TSM/B/6	47.55	0.34	17.56	3.04	0.15	0.86	0.21	0.22	2.59	0.05
TSM/B/7	48.31	0.15	16.69	1.81	0.10	2.16	0.54	0.16	2.24	0.06
TSM/B/8	49.63	0.17	17.31	2.10	0.06	1.69	0.09	0.23	1.56	0.05
TSM/B/9	49.70	0.31	18.24	2.44	0.08	1.08	0.09	0.18	1.45	0.04
TSM/B/10	46.01	0.42	20.87	3.47	0.15	1.51	0.34	0.18	1.69	0.07
TSM/B/11	50.38	0.10	15.58	1.51	0.05	0.84	0.15	0.13	2.55	0.02
TSM/B/12	49.23	0.09	17.32	1.87	0.04	0.43	0.43	0.12	3.31	0.04
TSM/B/13	50.62	0.14	15.44	1.50	0.17	0.45	1.17	0.13	2.47	0.11
TSM/B/14	45.77	0.14	19.18	1.75	0.08	4.57	0.60	0.15	1.96	0.07
TSM/B/15	51.88	0.13	14.76	1.68	0.10	0.33	0.57	0.07	2.11	0.09
TSM/B/16	47.98	0.11	15.96	1.34	0.17	3.01	1.15	0.15	2.64	0.15

for XRF major elements (percentage in oxides) and Table 4 for XRF trace elements (in ppm).

Results and Discussion

The results of the XRD mineralogical analysis are presented in Table 2, the results of the XRF elemental analysis are provided in Tables 3 (for major elements) and 4.

From the XRD results, all the Tanjung Simpang Mengayau ceramic samples contained sand (quartz, SiO_2) and alumina (mullite, $Al_{2.28}Si_{0.72}O_{4.86}$). Other minerals present were those formed under high temperature such as cristobalite (SiO_2), a calcium-based mineral, gypsum ($CaSO_{4.2}H_2O$) and feldspar ($K_{0.98}Na_{0.02}Al_{1.03}Si_{2.972}O_8$). All could be found in clayey soil as described by Samsudin (2011). TSM/B/10 was the only sample that contained sillimanite (Al_2SiO_5), maghemite (Fe_3O_4) and wuestite (FeO) which were metallic minerals. Mullite and cristobalite, on the other hand were high-temperature minerals. Mullite was formed upon firing aluminosilicate in temperatures of up to 1,345ºC and cristobalite was formed at 1,470ºC (Worrall, 1982).

Ceramic commonly contained clayey minerals such as kaolinite (Li *et al*., 2016) but all XRD diffractograms did not show the presence of any clayey minerals. This was most likely because all clayey minerals, in particular kaolinite had been transformed under high temperatures to another called meta-kaolin. Meta-kaolin is amorphous, which would not show any peak in the XRD diffractogram (Zuliskandar *et al*., 2012). Mullite minerals were present in all ceramic samples. They were common constituents in ceramics or refractory bodies, where they would be formed when silicate (quartz) and alumina minerals (kaolinite) were combined and heated to 1,345ºC. The presence of cristobalite with mullite (alumina) which were found in the celadon bowl samples only, indicated the change of quartz mineral to cristobalite due to high firing temperature of 1,470ºC (Worral, 1982). This suggested that the celadon bowls had been subjected to firing at

Table 4: Trace elements (ppm) in ceramic samples identified using XRF analysis

Element (ppm)	Dry Weight (ppm)											
	TSM/J/ 1	**TSM/J/ 2**	**TSM/J/ 3**	**TSM/J/ 4**	**TSM/J/ 5**	**TSM/B/ 6**	**TSM/B/ 7**	**TSM/B/ 8**	**TSM/B/ 9**	**TSM/B/ 10**	**TSM/B/ 11**	**TSM/B/ 12**
Ba	241	128	254	219	186	bdl	bdl	bdl	bdl	bdl	1351	bdl
Ce	373	248	bdl	bdl	bdl	590	bdl	bdl	477	311	bdl	255
Cl	944	859	205	1013	454	1964	3360	4204	2416	2837	1298	302
Cr	bdl	53	58	bdl	bdl	bdl	bdl	bdl	118	492	bdl	bdl
Cu	363	58	674	44	118	126	148	1690	138	234	896	84
Co	bdl	51	1111	bdl	bdl	bdl	bdl	1094	bdl	bdl	bdl	52
F	bdl	bdl	bdl	bdl	bdl	bdl	bdl	15	bdl	bdl	bdl	bdl
Ga	bdl	27	42	18	19	240	101	191	116	106	121	148
Nb	32	12	27	17	24	160	274	339	418	282	217	27
Nd	bdl	bdl	bdl	bdl	bdl	341	721	194	bdl	bdl	341	bdl
Ni	44	46	bdl	66	37	bdl	bdl	89	bdl	bdl	bdl	bdl
Pb	147	76	58	58	bdl	822	607	4345	691	388	1423	bdl
Rb	156	127	152	129	125	1010	922	1107	979	1012	1055	176
S	1219	1139	800	1008	647	2116	1268	1019	1534	1588	1078	287
Sr	78	56	90	72	62	270	454	148	120	227	400	62
Th	41	51	bdl	39	21	203	245	bdl	380	296	350	55
Tl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
W	4354	4923	6281	2918	2691	2759	1920	3178	2405	2649	3806	4089
Y	47	32	57	35	36	723	753	824	425	268	858	112
Zn	142	80	90	85	69	515	142	193	262	121	243	109
Zr	216	109	251	121	119	1178	984	1089	1213	1237	1277	163

bdl: Below detection limit

Element (ppm)	Dry Weight (ppm)			
	TSM/B/13	TSM/B/14	TSM/B/15	TSM/B/16
Ba	bdl	bdl	bdl	bdl
Ce	209	146	218	213
Cl	99	263	98	bdl
Cr	bdl	bdl	bdl	bdl
Cu	bdl	233	bdl	100
Co	282	224	275	bdl
F	bdl	bdl	bdl	bdl
Ga	79	29	218	32
Nb	bdl	18	31	26
Nd	bdl	bdl	bdl	bdl
Ni	52	bdl	bdl	bdl
Pb	84	93	69	65
Rb	217	123	174	210
S	52	1177	46	595
Sr	98	42	88	112
Th	34	bdl	45	45
Tl	bdl	bdl	21	bdl
W	7765	2405	7629	1974
Y	133	49	92	49
Zn	34	77	70	50
Zr	100	61	117	71

bdl: Below detection limit

much higher temperatures than the jars (Tong *et al.,* 2019).

Silicate minerals such as sillimanite minerals found in TSM/B/10 and TSM/B/11 suggested that these minerals were added to the ceramic paste. Some of them had turned into mullite after being fired at above 1,400ºC. Although the main raw material was clay which had high alumina content similar to kaolin, its use on its own was unlikely and it would be usually mixed with other raw materials such as quartz, feldspar and calcareous minerals (limestone or soil impregnated with lime). Calcareous mineral like gypsum found in the ceramic samples were added as a fluxing agent to increase the melting temperature during the burning process (Yap *et al*., 1994). Based on the minerals found in the ceramic samples, it could be proposed that the ceramics from Tanjung Simpang Mengayau were produced using kaolinite and sand as tempers before the ceramics were dried and baked in a kiln at very high temperatures.

The XRF results showed silica (SiO_2) as the highest dry weight percentage in all samples, ranging from 47.98% to 51.88%. The dry weight of alumina (Al_2O_3) was the second highest, ranging from 14.76% to 20.87%, followed by iron oxide (Fe_20_3) with a range of 1.34% to 3.04%. Potassium oxide (K_2O) recorded a range of 1.45% to 2.59%, magnesium oxide (MgO) from 0.33% to 4.57%, calcium oxide (CaO) from 0.23% to 1.17% and titanium oxide (TiO_2) from 0.09% to 0.55%. Small amounts of phosphate oxide (P_2O_3), manganese oxide (MnO) and sodium oxide (Na_2O) were recorded at less than 0.5%.

In total, 21 trace elements were detected in the ceramic samples with tungsten (W) recording the highest concentration followed by sulfur (S), chlorine (Cl), zircon (Zr), zinc (Zn) and lead (Pb). Other elements showed very low concentrations while some elements registered below detection limits.

Both the jar and celadon bowl samples showed rather similar major and trace element composition and were, therefore, not distinctive. Previous studies had shown similarities in the compositions of ceramic artefacts from the Tanjung Simpang Mengayau shipwreck. For instance, Xu *et al.* (2019) found that the main content of ceramics from the Song dynasty found in the Java Sea Shipwreck consisted of silica, alumina and feldspar by using XRF. Weidong *et al.* (2005) found that ancient Chinese ceramics contained quartz, mullite and cristobalite minerals as a manifestation of burning at a high temperature in a kiln similar to the Tanjung Simpang Mengayau samples.

Summary and Conclusion

In summary, this study had determined the elemental compositions and identified the technology used in the production of ceramics recovered from the Tanjung Simpang Mengayau shipwreck in Kudat, Sabah. The chemical characteristics of jars and celadon bowls were found to be rather similar and indistinctive, consisting of silica (SiO_2), alumina (Al_2O_3), iron oxide (Fe_2O_3), potassium oxide (K_2O), magnesium oxide (MgO) and calcium oxide (CaO) with trace elements comprising tungsten (W), sulfur (S), chlorine (Cl), zircon (Zr), zinc (Zn) and lead (Pb), strontium (Sr), yttrium (Y), rubidium (Rb), copper (Cu) and niobium (Nb).

In terms of clay types, the chemical analysis indicated that both jars and celadon bowls were made using kaolin (kaolinite) with the addition of crushed sand (quartz) and flux (CaO). The presence of high temperature minerals, in particular mullite and cristobalite, indicated that the jars and celadon bowls were fired at high temperatures of 1,345ºC and 1,470ºC, respectively. The findings of this study could assist museum and heritage conservators in understanding the best methods to preserve and conserve the Tanjung Simpang Mengayau archaeological ceramics. Artefacts with high content of contaminants such as sodium chloride (salt) and calcium carbonate (barnacles) from the sea, and inclusions of silicon oxide (sand) during production would require different methods and levels of conservation. In addition, further research should be undertaken on the Tanjung Simpang Mengayau ceramics, especially with more samples such as plates and ornaments to not only analyse its components, but also the chemical used to glaze the ceramic. Other commonly used methods such as the thin section petrographic study to determine the minerals and inclusions, as well as thermoluminescence dating (TL) to determine the age and period of manufacture should be included in future studies. Information gathered from these studies would be important so that determine the significance of Tanjung Simpang Mengayau shipwreck site in the course of history that might currently promote sustainable economic interests, besides protecting the country's heritage.

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