



## Investigations of fine-paste ware production and exchange in maritime Southeast Asia by electron microscopy and synchrotron X-ray absorption

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Received 18 November 2018; Received in revised form 5 May 2019; Accepted 26 July 2019

### Abstract

The combination of synchrotron X-ray absorption and electron microscopy provided information on the production and exchange of fine-paste ware (FPW), dated to around the 11<sup>th</sup> to 14<sup>th</sup> centuries in peninsular Thailand and maritime Southeast Asia. White FPW sherds from Kota Cina in north Sumatra, a well-known trading city in Indonesia, have similar clay composition to those from Kok Moh on Satingphra Spit in peninsular Thailand. Also, homogeneous texture indicating high firing temperatures was found in the samples from Kota Cina as well as in those from Phra Mahathat Temple and Suan Luang Temple in Nakhon Si Thammarat Province in Thailand. However, the split Mn K-edge X-ray absorption near-edge structure (XANES) peaks are exclusively observed in the samples from Nakhon Si Thammarat. The distinctly large compositions of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and carbon, respectively confirmed by Fe K-edge extended X-ray absorption fine structure (EXAFS) and energy dispersive spectroscopy (EDS) spectra, suggest that Nakhon Si Thammarat probably had at least a FPW production site in which the ash glazing process may have been implemented. The evidence of such process is not found in the samples from other sites in maritime Southeast Asia in this analysis.

**Keywords:** Maritime Southeast Asia, fine-paste ware, SEM, Fe K-edge EXAFS, Mn K-edge XANES

### I. Introduction

Southeast Asia has a long history of pottery making. The first earthenware in this region was probably made at least in the Neolithic Period, around 4000 years ago [1]. However, these earthenware of various cultures, sometime created with complex forms, motifs and decorations, were not traded widely. They were usually made and used locally. The exception is one type of earthenware called fine-paste ware (hereafter FPW) in which its distributions suggest that it was traded in a large area in maritime Southeast Asia, from peninsular Thailand to north Sumatra, Java, and southern Philippines since the 10<sup>th</sup> century, but the peak of its trade was probably from the 11<sup>th</sup> to 14<sup>th</sup> centuries [2]. FPW arguably was the only unglazed earthenware in Southeast Asia that was peerlessly traded in such a great geographical extent.

FPW was the trade goods in itself, which was transported to a far corner of Southeast Asia because of its own value, not because of the goods it contained inside. It was not used as container of goods in shipment as well. Other earthenware could travel far too but it is because they were containers of other goods. Manguin [2] suggests that FPW was originally an Indian influence. Various forms of FPW category were found in Oc Eo, an important trading city in the Mekong Delta in the early centuries A.D. and in Palembang, the probable capital city of Sriwijaya, in south Sumatra in the 7<sup>th</sup> to 8<sup>th</sup> centuries A.D.

FPW also withstood the storm of Chinese glazed ceramics flooded into Southeast Asia from the 9<sup>th</sup> century onward, whereas many other types of earthenware were replaced by them. Local earthenware usually could not compete with the shininess, sturdiness and delicate forms and decorations of Chinese glazed ceramics. However, FPW survived and even prospered only in the form of *kendi*, which was an ewer or globular ves-

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sel with a spout for pouring water out as shown in the inset of Fig. 1. This form might have been continued to be used because it had a role in Hindu-Buddhist ritual libations and its unglazed body of almost pure clay was considered exquisite and could keep drinking water cool in the warm climate of Southeast Asia. FPW was wheel-made from fine-grained clay, usually without visible tempers, sometime applied with slips, and always fired in an oxidizing atmosphere [2]. Its exceptionally fine body revealed the high-level craftsmanship and time-consuming delicate process, which possibly made it a kind of prestige goods in this region. Fragments and complete FPW kendis were found in the excavations in peninsular Thailand. In Nakhon Si Thammarat Province, they were usually found in association with the Chinese ceramics from the Southern Sung to early Yuan Dynasties, which can be dated to the 13<sup>th</sup> to 14<sup>th</sup> centuries [3,4].



**Figure 1.** North Sumatra, in present-day Indonesia, is where samples were acquired in Kota Cina (KTC). Sathingphra with samples from Kok Moh (KOM) and Nakhon Si Thammarat province with samples from Phra Mahathat temple (PT7) and Suan Lang temple (SL5) are on the east coast of peninsular Thailand. An example of *kendi* is shown in the inset

Ancient ceramics around the world have been increasingly studied by scientific instruments and methods [5–7]. However, there are only a few scientific reports on the FPW from maritime Southeast Asia. Miksic and Yap [8] analysed composition of FPW sherds by using X-ray fluorescence (XRF) spectroscopy and suggested that there were at least 2 production centres with other probable FPW production sites in Southeast Asia. One production centre was located in east Java in present-day Indonesia and the other production centre was on Sathingphra Spit in present-day Songkhla province of Thailand. Kok Moh (KOM) in Sathingphra has been the only archaeological site in which remains of the FPW kilns were discovered in peninsular Thailand. Recent analysis reveals that the area of modern-day Nakhon Si Thammarat city, also on the east coast of peninsular Thailand around 100 km north of Sating-

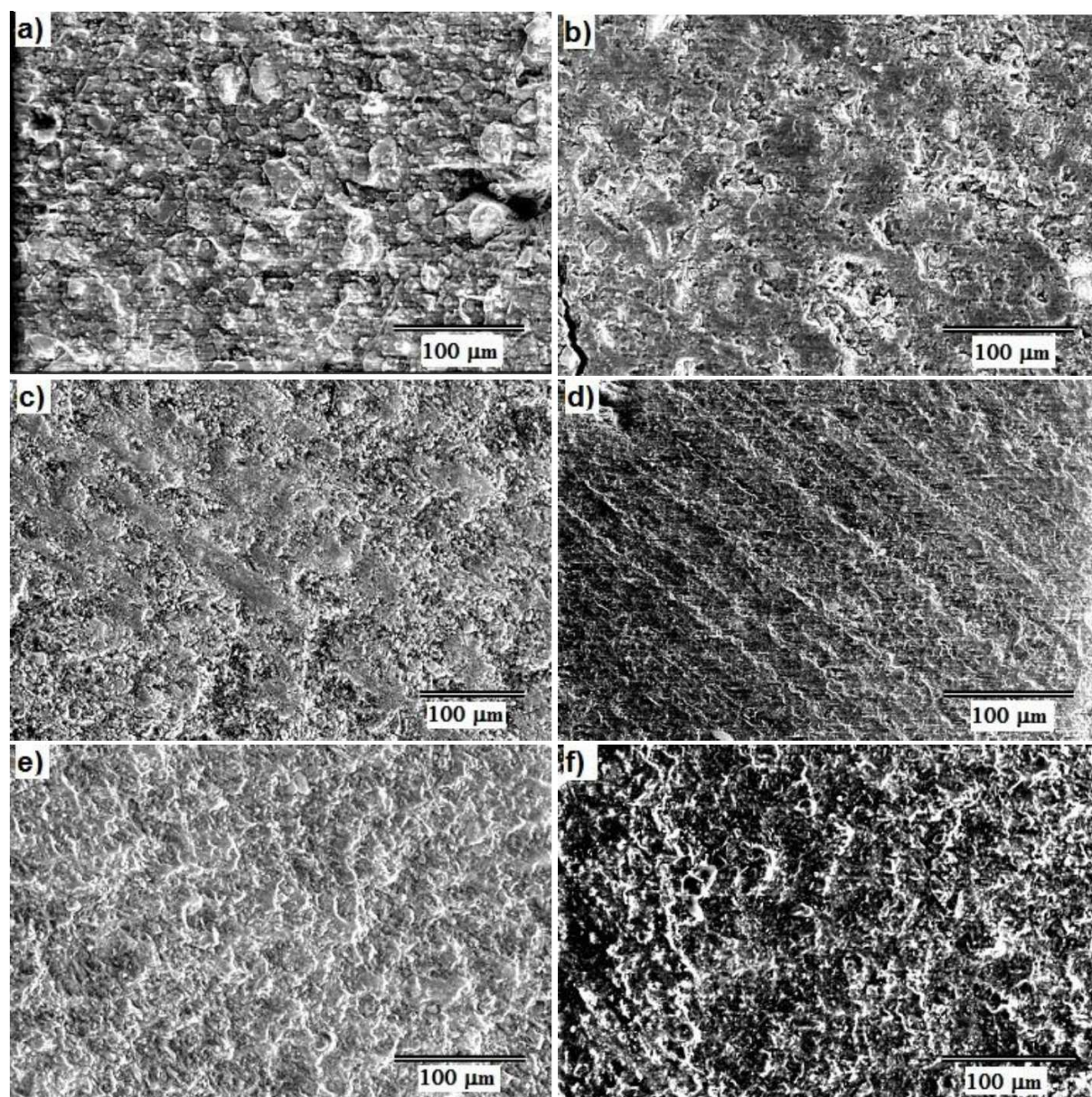
phra as illustrated in Fig. 1, may have been another production area of FPW in this region, although no kiln has been identified in the area yet. The area of modern-day Nakhon Si Thammarat city was greatly populated in 13<sup>th</sup> century A.D., as it became the capital of Nakhon Si Thammarat Kingdom, although this area already had the evidence of ancient communities from at least the 6<sup>th</sup> to 12<sup>th</sup> centuries A.D. [3].

According to XRF and petrographic analyses by Ueda *et al.* in 2017 [9], FPW was classified into the red-slipped and white FPW. The authors suggested that, whereas east Java mainly produced the former, the white FPW mainly composed of kaolin had a production centre in KOM as well as other possible sites in peninsular Thailand. In our previous work [10], XRF and synchrotron Fe K-edge X-ray absorption near-edge structure (XANES) were used to compare FPW kendis from four archaeological sites in peninsular Thailand with those from Kota Cina (KTC) in north Sumatra to establish their trade links in maritime Southeast Asia. KTC was an important trading port from the late 11<sup>th</sup> to the late 13<sup>th</sup> century whose name means a fortified Chinese settlement. The similarities based on clay components of  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\alpha\text{-Fe}_2\text{O}_3$  and  $\gamma\text{-Fe}_2\text{O}_3$  as well as trace elements provided another evidence of a trade route between KOM and KTC. In addition, the uniquely large contribution of  $\alpha\text{-Fe}_2\text{O}_3$  shown by XANES spectra supported the hypothesis that the area of modern-day Nakhon Si Thammarat city was once a production place of white FPW in maritime Southeast Asia [9].

This article aims to compare microscopic texture as well as elemental composition of FPW sherds in order to investigate their production process in maritime Southeast Asia. The linkage between archaeological sites is analysed by the synchrotron X-ray absorption. It can complement the XRF commonly used in archaeometry [5], due to its sensitivity to structures of different phases [6]. Extended X-ray absorption fine structure (EXAFS) was successfully used in identifications of decorated Japanese and Sicilian ceramics, respectively based on Fe and Cu species in their coating [11,12]. In addition to the aforementioned Fe K-edge XANES, there were studies on other elements including Mn, Cu, Cr, As and S species in ancient ceramics and pigments [6,12–14] and Mn K-edge XANES is also used in this study.

## II. Materials and methods

Nine FPW sherds from four different archaeological sites, namely Kota Cina (KTC), Kok Moh (KOM), Phra Mahathat temple (PT7) and Suan Luang temple (SL5) were included in this study and were previously used in the analysis by Ueda *et al.* [9]. The KTC and KOM samples were collected in the surface surveys at the sites and provided to us by Prof. John Miksic. Kota Cina was a trading port in northeastern Sumatra, Indonesia, in the 11<sup>th</sup>–13<sup>th</sup> centuries. Kok Moh is a kiln site producing the white FPW in the Pa-O village on the Sathingphra spit in Songkhla Province, peninsular Thailand. It was



**Figure 2.** Cross-sectional SEM micrographs of FPW shreds: a) KOM\_001, b) SL5\_003, c) KTC\_002, d) PT7\_014, e) PT7\_019, and f) PT7\_020

excavated and hypothesized that its production of FPW was concentrated in the 11<sup>th</sup>–12<sup>th</sup> centuries [9]. The PT7 and SL5 samples were excavated from two temples in Nakhon Si Thammarat province by Wannasarn Noon-suk in 2009 [4]. Phra Mahathat and Suan Luang were the religious sites which may have been originated in the 6<sup>th</sup>–7<sup>th</sup> centuries. Today they are Theravada Buddhist monasteries located on the Gulf of Siam around 100 km north of Kok Moh. The PT7 and SL5 samples were similarly found in the excavation associated with the Chinese ceramics from the Southern Sung to early Yuan Dynasties, which can be dated to the 13<sup>th</sup>–14<sup>th</sup> centuries. Therefore, it can be preliminarily assumed that these two samples can be dated approximately to the 13<sup>th</sup>–14<sup>th</sup> centuries. All samples mentioned above were impregnated with epoxy before they were cut to expose their cross-section.

Scanning electron microscope (SEM; FEI Quanta 450 FEG) operating at 15 kV was used to visualize the texture of cross sectional surface. Composition of clay was characterized by energy dispersive spectroscopy (EDS) probe attached to the SEM. X-ray absorption spectroscopy (XAS) was performed at Beamline 5.2 of Synchrotron Light Research Institute (SLRI), Nakhon Ratchasima, Thailand. Measurements were in the fluorescent mode using 1.2 GeV electrons with a maximum beam current of 150 mA. To obtain Mn K-edge XANES spectra from the XAS experiment, a double-crystal Ge(220) monochromator with an energy resolution ( $\Delta E/E$ ) of  $2 \times 10^{-4}$  was used to scan the energy of the synchrotron X-ray from 6500 to 6700 eV with an energy step of 0.2 eV. For Fe K-edge EXAFS spectra, the scanned energies were –200, –20, 30 and 800 eV with energy steps of 5, 0.2 and 0.01 eV and collecting

times of 3, 3 and 3 s, respectively. The data processing included a pre-edge, post-edge background subtraction and normalization. The normalized XANES data were analysed in the pre-edge and post-edge region using the ATHENA software included in an IFEFFIT package [15]. The spectra were then matched with those from MnO, MnO<sub>2</sub>, Mn<sub>2</sub>O<sub>3</sub>, Mn<sub>3</sub>O<sub>4</sub> as well as Mn standards in the fingerprinting method. Due to a large noise at high *k*-space, the Fourier transform (FT) of the *k*<sup>3</sup>-weighted normalized EXAFS data was performed with a maximum *k*-value of 10 Å<sup>-1</sup>. Maghemite (γ-Fe<sub>2</sub>O<sub>3</sub>), and hematite (α-Fe<sub>2</sub>O<sub>3</sub>) standards were also measured to use as references in comparison with spectra from the FPW.

### III. Results and discussion

Different particle sizes and shapes in cross-sectional SEM micrographs of ceramics revealed different firing temperatures in their manufacturing process [16,17]. As exemplified in Fig. 2a, sample KOM\_001 consists of grains of several μm in size suggesting that this sample was prepared at low temperature conditions. SEM micrographs of other samples in Figs. 2b-2f exhibit more homogeneous textures as a result of high temperatures of firing. Welded grains and clay plates were evidently formed. Aligned pores seen only in sample PT7\_014 according to Fig. 2d, may be attributed to the coiling of pottery [18]. Distribution of bloating pores in this sample reveals that it was fired in multi-step technology at relatively high temperature [19,20]. Such morphological variations might come from different requirements by consumers, as previously suggested in literature [9].

EDS spectra signify sources of clay with the presence of Si, Al, Fe, K, Ti, Mg, Ca and Na. The percentages of elemental composition are averaged from measurements at three spots in each sample and listed

in Table 1. These results are consistent with the published XRF results in literature [10] on the same set of samples that suggested the compositions of the samples from KTC and KOM are comparable. The percentages of K and P in sample SL5\_003 are much higher than those for other samples. Potassium compounds disassemble as fluxes during firing, encouraging the initial sintering and extensive vitrification [21]. A high amount of oxygen is expected since the FPW were manufactured by firing in an oxidizing atmosphere. Carbon element observed for all samples can be due to depositing soot particles or pyrolytic decomposition of hydrocarbons in the kiln atmosphere on the surface of the hot vessel. This can explain its larger variation than any other elements. Interestingly, two samples from PT7 exhibit conspicuously high compositions of C with small amounts of Cl, which is not detected in other samples. The measurements were then repeated on these samples (PT7\_014 and PT7\_020) for 4–6 times and the amount of C listed

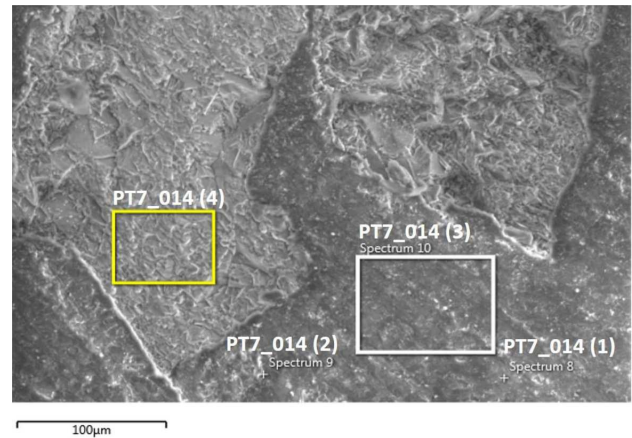


Figure 3. SEM micrograph identifying four spots on the sample PT7\_014 with high carbon contents in the EDS measurement

Table 1. Elemental composition of FPW shreds from EDS measurements

Sample	Elemental composition [%]											
	O	Si	Al	C	Fe	K	Ti	Mg	Ca	Na	P	Cl
KTC_004	47.63 ±0.90	23.93 ±1.33	12.63 ±0.51	9.40 ±2.55	2.86 ±0.55	1.30 ±0.06	0.53 ±0.06	0.30 ±0.00	0.76 ±0.21	0.20 ±0.00	0.40 ±0.17	-
KOM_001	47.83 ±1.00	24.00 ±1.32	13.53 ±0.64	7.50 ±2.88	4.20 ±0.10	1.40 ±0.10	0.66 ±0.06	0.43 ±0.06	0.20 ±0.00	0.23 ±0.06	-	-
SL5_003	52.30 ±0.79	17.20 ±0.84	12.60 ±0.61	7.63 ±2.00	3.06 ±0.06	2.20 ±0.10	0.56 ±0.06	0.20 ±0.00	-	0.23 ±0.06	3.87 ±0.15	-
PT7_019	48.13 ±0.32	19.06 ±1.35	16.93 ±0.12	8.70 ±1.41	3.70 ±0.26	1.76 ±0.06	0.63 ±0.06	0.23 ±0.06	0.40 ±0.00	0.10 ±0.00	0.23 ±0.06	-
PT7_014 (1)	20.0	1.1	0.3	76.1	0.1	0.2	-	-	0.1	0.2	-	2.0
PT7_014 (2)	16.7	2.8	0.9	75.2	0.4	0.6	-	0.1	-	0.2	-	3.1
PT7_014 (3)	22.3	1.7	0.4	73.4	0.1	0.3	-	0.2	-	0.2	-	1.3
PT7_014 (4)	31.2	11.9	2.1	51.2	0.1	2.5	-	0.1	-	0.3	-	0.6
PT7_014 (5)	42.5	14.0	9.2	27.7	2.6	1.8	0.5	0.2	0.8	-	0.4	0.2
PT7_014 (6)	51.6	19.7	13.4	8.6	2.8	2.0	0.4	0.2	0.7	0.2	0.2	-
PT7_020 (1)	30.3	5.3	5.7	55.5	1.3	0.6	0.2	0.1	0.2	0.1	-	0.6
PT7_020 (2)	43.7	13.2	15.0	21.8	3.0	1.4	0.6	0.3	0.4	0.2	0.3	0.1
PT7_020 (3)	45.3	15.3	15.0	15.8	4.8	1.9	0.6	0.3	0.4	0.2	0.4	0.1
PT7_020 (4)	47.4	17.3	16.2	10.5	4.6	2.0	0.5	0.3	0.5	0.2	0.5	-

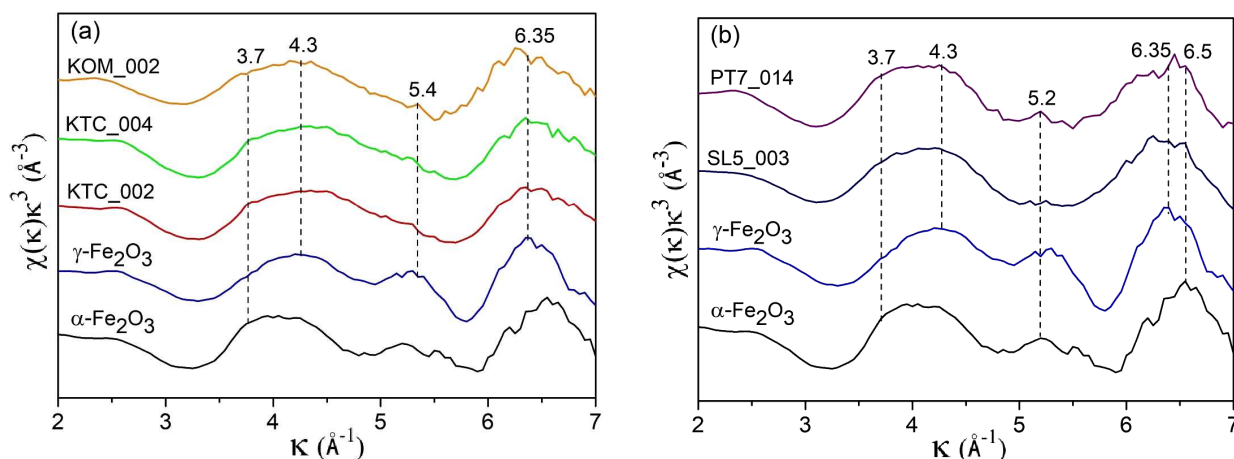


Figure 4. Comparison of FT Fe K-edge EXAFS spectra of Fe and its oxide standards with the FPW shreds from: a) Kota Cina (KTC) and Kok Moh (KOM), b) Phra Mahathat temple (PT7) and Suan Lang temple (SL5)

in Table 1 exceeded 50% in some spots. Four measurement spots on the samples PT7\_014 with high compositions of C shown in Fig. 3 are more than 100  $\mu\text{m}$  apart. According to Table 1, the C contents at PT7\_014 (1), PT7\_014 (2), PT7\_014 (3) and PT7\_014 (4) are respectively 76.1, 75.2, 73.4 and 51.2 as the probe moved away from the edge. Since the samples were not coated in the production and imaging processes, this anomaly can be explained by the process called the ash glazing, originated in China during the Shang Dynasty around 1500 B.C. Incorporations of ash during either mixing or firing lead to the random glaze effect [22]. The exclusive observation of ash glazing in the samples from PT7 is another supportive evidence of FPW production sites in Nakhon Si Thammarat.

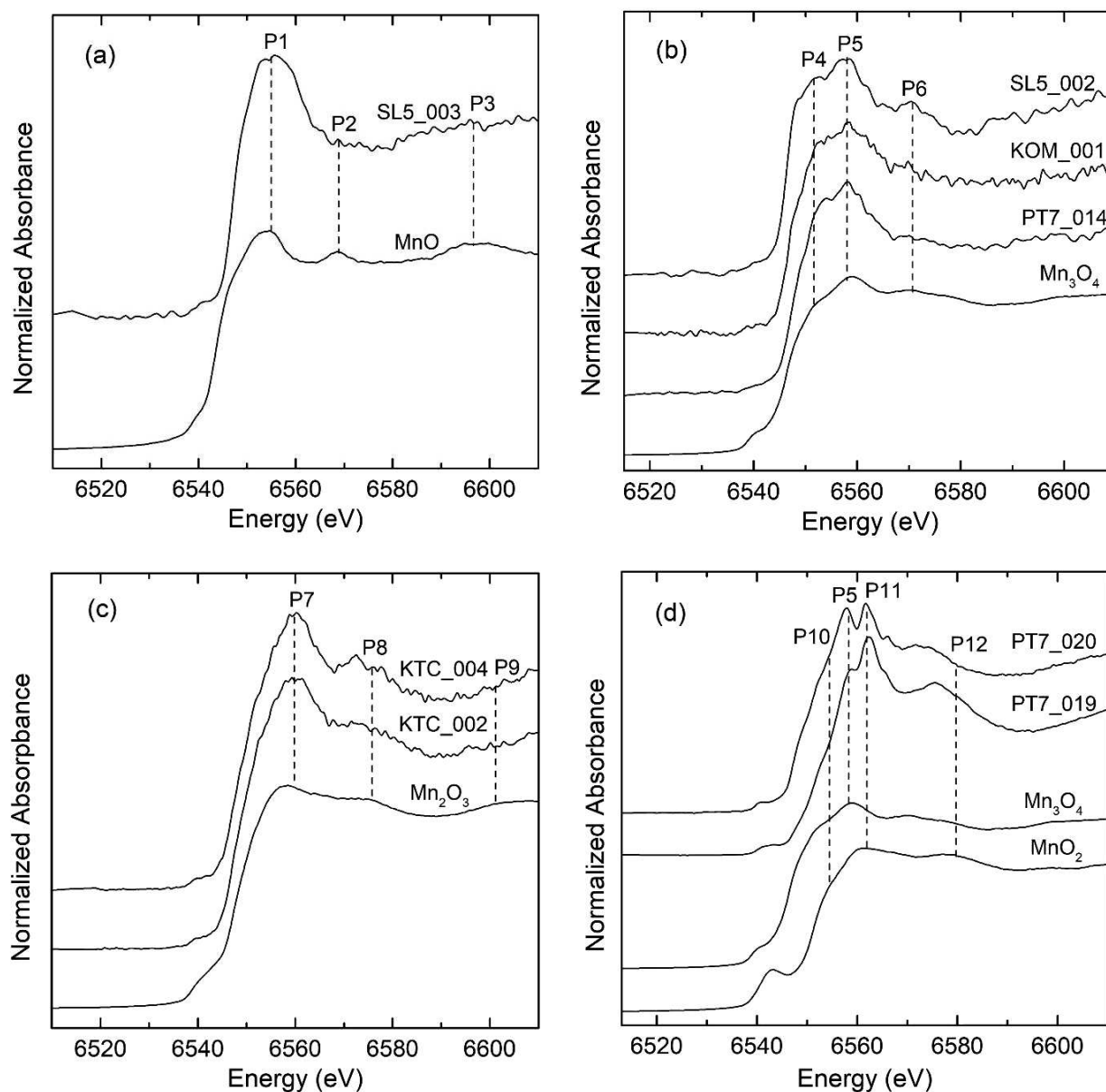
Since the published Fe K-edge XANES results indicated a large fraction of  $\alpha\text{-Fe}_2\text{O}_3$  phase in FPW from Nakhon Si Thammarat [10], the forms of iron oxides are further characterized in this work using FT-EXAFS shown in Fig. 4. Firstly,  $\gamma\text{-Fe}_2\text{O}_3$  and  $\alpha\text{-Fe}_2\text{O}_3$  standards have comparable spectra with varying peak positions. By comparing the main peaks, it can be concluded that all five samples contained both phases with different fractions. In Fig. 4a, the sample KOM\_002 and two samples from KTC exhibit the peaks at 4.30, 5.40 and 6.35  $\text{\AA}^{-1}$  signifying the dominance of  $\gamma\text{-Fe}_2\text{O}_3$  phase. On the other hand, two samples from Nakhon Si Thammarat (SL5\_003 and PT7\_014) exhibit peak shifts from 4.30 and 5.40  $\text{\AA}^{-1}$  to lower  $k$  values in Fig. 4b. Moreover, the peak at 6.35  $\text{\AA}^{-1}$  is displaced towards 6.50  $\text{\AA}^{-1}$  resembling the  $\alpha\text{-Fe}_2\text{O}_3$  characteristics. These spectra therefore add the phase identification to the reported el-

emental composition and confirm the unique clay source in FPW found in Nakhon Si Thammarat.

In addition to the major clay compositions, substantial information of FPW can be derived from trace elements in the shreds including Mn. In this work, synchrotron X-ray absorption is sensitive to Mn species even without the use of decoration layer. Normalized Mn K-edge XANES spectra of the samples are compared to five standard references with different oxidation states of Mn in Fig. 5. Standard spectra collected for calibrating peak position exhibit peaks at different positions of the absorption edge. The pre-edge peak, notable characteristic of  $\text{MnO}_2$ , is not clearly observed in all samples. The absorption edges ranking from the lowest to the highest energy are Mn, MnO,  $\text{Mn}_2\text{O}_3$ ,  $\text{Mn}_3\text{O}_4$  and  $\text{MnO}_2$  according to the oxidation state ranging from 0 to 4+. Spectra of the samples do not exactly resemble any single standard but it implies the combinations of two or more standards. Although the spectra could not be fitted with the linear combination of these standards due to the measurement noise, the samples can be quantitatively compared by locating the peak positions as shown in Fig. 5 and listed in Table 2. In Fig. 5a, the sample from SL5 (SL5\_003) exhibit the peaks at 6553, 6568 and 6595 eV, respectively matched with the MnO standard peaks denoted as P1, P2, P3. However, the characteristic for the sample SL5\_002 is completely different. In Fig 5b, the XANES spectrum for SL5\_002 is strikingly similar to those of KOM\_001 and PT7\_014 with a broad peak (P5) at around 6558 eV. The other peaks at 6550 eV (P4) and 6570 eV (P6) are also closely resembled the  $\text{Mn}_3\text{O}_4$  standard. As shown in Fig. 5c, both

Table 2. List of positions of Mn K-edge XANES peaks denoted as P1–P12 in Fig. 5

Standard references	Peak position [eV]											
	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12
MnO	6553	6568	6595									
$\text{Mn}_3\text{O}_4$				6550	6558	6570						
$\text{Mn}_2\text{O}_3$							6560	6575	6600			
$\text{MnO}_2$										6555	6563	6580



**Figure 5.** Comparison of normalized Mn K-edge XANES spectra of Mn and its oxide standards with the FPW shreds from: a) Suan Lang temple (SL5\_003), b) Suan Lang temple (SL5\_003), Kok Moh (KOM\_001), Phra Mahathat temple (PT7\_014), c) Kota Cina (KTC\_002, KTC\_004) and d) Phra Mahathat temple (PT7\_019, PT7\_020)

samples from Kota China have Mn K-edge XANES peaks comparable to those of  $\text{Mn}_2\text{O}_3$  standard (P7, P8, P9). In addition to P10 and P12 peaks of  $\text{MnO}_2$  standard, spectra for the samples PT7\_019 and PT7\_020 in Fig. 4d have distinct double peaks which are attributable to different phases in these two samples. By comparing them with standards, their peaks are matched with peaks of  $\text{MnO}_2$  and  $\text{Mn}_3\text{O}_4$  at approximately 6563 eV (P11) and 6558 eV (P5) respectively. Similar XANES spectra of the samples from different places and different spectra of the samples from the same places suggest that there were trade system of FPW within this region. In addition to FPW manufactured in Nakhon Si Thammarat, PT7 and SL5 may have been important temples which tend to have a collection of pottery from other manufacturing sites including KOM.

#### IV. Conclusions

Pottery shreds distributed in the 11<sup>th</sup>–14<sup>th</sup> century maritime Southeast Asia were studied. Different textures and elemental compositions for dissimilar samples from four archaeological sites revealed various sources of clay different production sites as well as process. Comparable compositions and phases in some samples from different sites suggested that there were exchanges and trading within this region. Moreover, unique amount of carbon and X-ray absorption spectra were supporting evidences that there were likely FPW production sites using ash glazing process in Nakhon Si Thammarat area.

**Acknowledgement:** The project is funded by Walailak University annual budget (WU61117). The authors

would like to thank Prof. John Miksic and Dr. Kaoru Ueda for supplying valuable FPW samples and their information. The facility supports by Synchrotron Light Research Institute (Public Organization), Nakhon Ratchasima, Thailand and the beamline manager (Dr. Pinit Kidkhunthod) are acknowledged. SEM imaging was carried out at Thaksin University.

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